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bats@chemie.uni-frankfurt.de**Key indicators**Single-crystal X-ray study
 $T = 148\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.002\text{ \AA}$
 R factor = 0.049
 wR factor = 0.124
Data-to-parameter ratio = 23.5For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.**3-Phenylpyrazole, a pseudosymmetric structure with $Z' = 6$** The title compound, $\text{C}_9\text{H}_8\text{N}_2$, is triclinic (space group $P\bar{1}$ with $Z' = 6$). Six molecules are connected by $\text{N}-\text{H}\cdots\text{N}$ hydrogen bonds into centrosymmetric hexamers. The two independent hexamers are related by a pseudosymmetric glide plane.

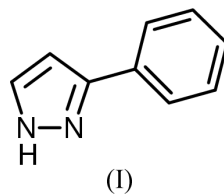
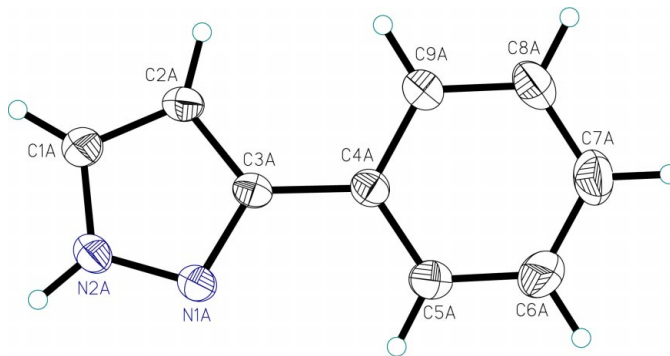
Received 4 November 2002

Accepted 8 November 2002

Online 15 November 2002

Comment

Tris(1-pyrazolyl)borates (scorpionates) were first produced more than 30 years ago and are today well established as ligands in coordination chemistry (Trofimenko, 1993). Scorpionates now find wide-ranging applications in chemistry, from modeling the active site of metal enzymes, through analytical chemistry and organic chemistry, to catalysis and material science (Edelmann, 2001). Given this background, we became interested in the synthesis of transition metal complexes of hydrotri(3-phenylpyrazol-1-yl)borate. Therefore, we prepared 3-phenylpyrazole, (I). The synthesis of (I) was confirmed by a crystal structure determination.

The asymmetric unit of (I) contains six independent molecules, labeled *A* to *F* (Fig. 1). The dimensions of the six molecules are very similar. The pyrazole and phenyl groups of each molecule are approximately planar. The angles between the planes of the pyrazole and phenyl groups are 9.21 (11), 8.10 (5), 9.38 (6), 27.62 (7), 6.21 (4) and 21.33 (4) $^\circ$ for mol-**Figure 1**The structure of molecule *A*, shown with 50% probability displacement ellipsoids. A similar numbering scheme is used for molecules *B*, *C*, *D*, *E* and *F*.

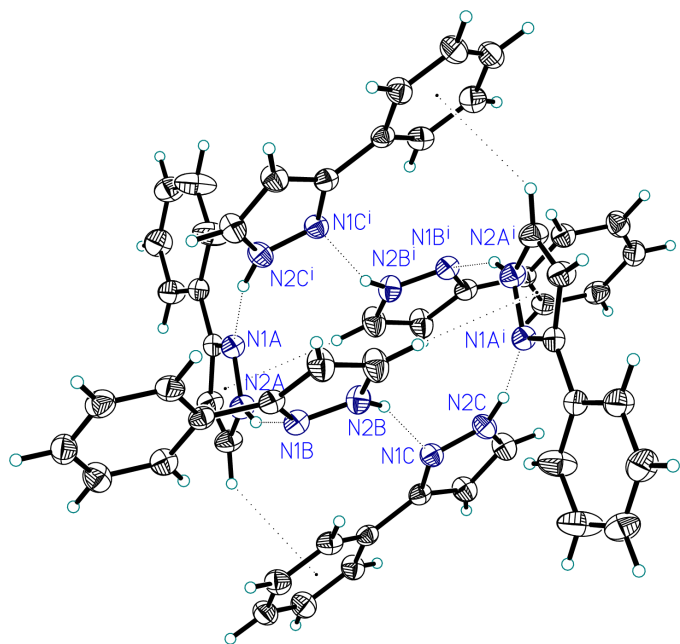


Figure 2
The hydrogen-bonded hexamer of molecules *A*, *B*, *C*, *A*^{*i*}, *B*^{*i*} and *C*^{*i*} [symmetry code: (i) $1 - x, 1 - y, 1 - z$].

ecules *A*–*F*, respectively. The crystal structure contains two independent clusters of six molecules connected by $\text{N} \cdots \text{H} \cdots \text{N}$ hydrogen bonds (Table 1). The six-membered clusters are centrosymmetric. One hexamer contains molecules *A*, *B* and *C*, while the other contains molecules *D*, *E* and *F*. Both hexamers have rather similar conformations (Fig. 2). Each is additionally stabilized by two symmetry-related $\text{C}_{\text{pyrazole}} - \text{H} \cdots \pi_{\text{pyrazole}}$ and $\text{C}_{\text{pyrazole}} - \text{H} \cdots \pi_{\text{phenyl}}$ interactions, with $\text{H} \cdots \pi$ distances between 2.64 and 2.83 Å, and $\text{C} - \text{H} \cdots \pi$ angles between 138 and 152° (π represents the centroid of the corresponding pyrazole or phenyl ring). Different hexamers are connected by seven additional intermolecular $\text{C} - \text{H} \cdots \pi_{\text{pyrazole}}$ or $\text{C} - \text{H} \cdots \pi_{\text{phenyl}}$ interactions, with $\text{H} \cdots \pi$ distances between 2.76 and 3.14 Å, and $\text{C} - \text{H} \cdots \pi$ angles between 123 and 170°. One interaction is found between each of the symmetry-related hexamers, while five interactions connect symmetry-independent hexamers. The hexamer containing molecules *A*, *B* and *C* mainly occupies the section of the unit cell defined by $\frac{1}{4} < z < \frac{3}{4}$ (Fig. 3). The hexamer containing molecules *D*, *E* and *F* is mainly confined to the section of the unit cell defined by $-\frac{1}{4} < z < \frac{1}{4}$. The crystal packing in both sections of the unit cell is very similar. The arrangement of the molecules in the latter section can be approximately obtained from the structure of the first section by interchanging the *a* and *b* axes. This shows the crystal structure of the title compound to be pseudosymmetric. The coordinates of the atoms in the two independent hexamers are related by the pseudosymmetry relations: $x_{\text{ABC}} \sim \frac{1}{2} - y_{\text{DEF}}$, $y_{\text{ABC}} \sim 1 - x_{\text{DEF}}$, $z_{\text{ABC}} \sim \frac{1}{2} - z_{\text{DEF}}$ and $x_{\text{ABC}} \sim \frac{1}{2} + y_{\text{D}^{\text{ii}}\text{E}^{\text{ii}}\text{F}^{\text{ii}}}$, $y_{\text{ABC}} \sim x_{\text{D}^{\text{ii}}\text{E}^{\text{ii}}\text{F}^{\text{ii}}}$, $z_{\text{ABC}} \sim \frac{1}{2} + z_{\text{D}^{\text{ii}}\text{E}^{\text{ii}}\text{F}^{\text{ii}}}$ [symmetry code: (ii) $1 - x, -y, -z$]. The latter pseudosymmetry relation corresponds to an approximate *c*-glide plane perpendicular to the *a*, *b* diag-

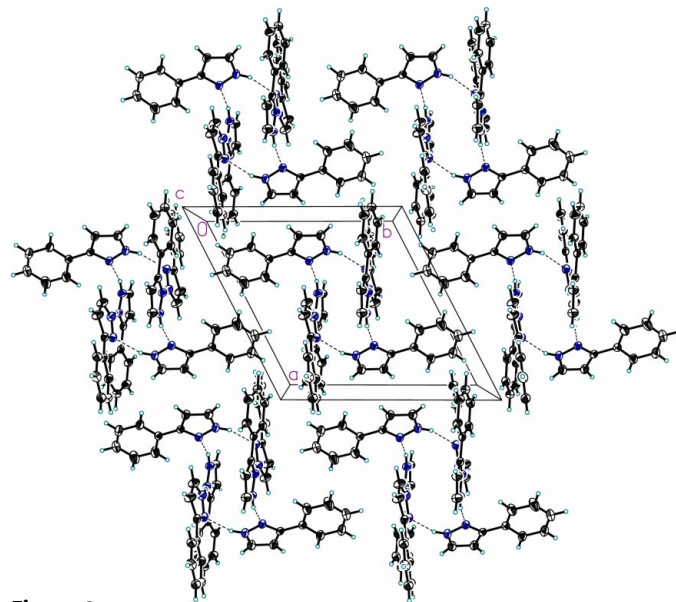


Figure 3
The crystal packing of (I), viewed down *c*. A section with $\frac{1}{4} < z < \frac{3}{4}$ containing only hexamers with molecules *A*, *B* and *C* is shown.

onal. A crystal structure with similar pseudosymmetric domains connected by $\text{C} - \text{H} \cdots \pi$ interactions has been reported by Bats *et al.* (1999).

Experimental

3-Phenylpyrazole was synthesized as reported by Buchner & Hachumian (1902). Single crystals suitable for data collection were obtained by recrystallization from chloroform at ambient temperature.

Crystal data

$\text{C}_9\text{H}_8\text{N}_2$	$Z = 12$
$M_r = 144.17$	$D_x = 1.262 \text{ Mg m}^{-3}$
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 12.1514 (10) \text{ \AA}$	Cell parameters from 324 reflections
$b = 12.3120 (11) \text{ \AA}$	$\theta = 3\text{--}26^\circ$
$c = 17.114 (2) \text{ \AA}$	$\mu = 0.08 \text{ mm}^{-1}$
$\alpha = 88.164 (11)^\circ$	$T = 148 (2) \text{ K}$
$\beta = 88.103 (12)^\circ$	Block, colorless
$\gamma = 62.816 (6)^\circ$	$0.50 \times 0.48 \times 0.40 \text{ mm}$
$V = 2275.9 (4) \text{ \AA}^3$	

Data collection

Siemens SMART CCD diffractometer	$R_{\text{int}} = 0.038$
ω scans	$\theta_{\text{max}} = 31.0^\circ$
Absorption correction: numerical (SHELXTL; Sheldrick, 1996)	$h = -17 \rightarrow 17$
$T_{\text{min}} = 0.959, T_{\text{max}} = 0.975$	$k = -17 \rightarrow 17$
40438 measured reflections	$l = -23 \rightarrow 24$
14026 independent reflections	557 standard reflections
10303 reflections with $I > 2\sigma(I)$	frequency: 600 min
	intensity decay: none

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.04P)^2 + 0.9P]$
$R[F^2 > 2\sigma(F^2)] = 0.049$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.124$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.05$	$\Delta\rho_{\text{max}} = 0.34 \text{ e \AA}^{-3}$
14026 reflections	$\Delta\rho_{\text{min}} = -0.27 \text{ e \AA}^{-3}$
596 parameters	Extinction correction: SHELXL97
H-atom parameters constrained	Extinction coefficient: 0.0011 (3)

Table 1
Hydrogen-bonding geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$N2A-H2NA \cdots N1B$	0.88	2.06	2.8905 (17)	158
$N2B-H2NB \cdots N1C$	0.88	2.12	2.9601 (16)	159
$N2C-H2NC \cdots N1A^i$	0.88	2.12	2.9540 (18)	157
$N2D-H2ND \cdots N1E$	0.88	2.06	2.8767 (18)	154
$N2E-H2NE \cdots N1F$	0.88	2.12	2.9587 (15)	159
$N2F-H2NF \cdots N1D^{ii}$	0.88	2.11	2.9157 (15)	152

Symmetry codes: (i) $1-x, 1-y, 1-z$; (ii) $1-x, -y, -z$.

A difference Fourier synthesis showed all six independent molecules to be protonated at the N atom labeled N2. The H atoms were positioned geometrically and were refined with fixed individual isotropic displacement parameters [$U_{iso}(H) = 1.2U_{eq}(C,N)$], using a riding model with fixed distances [$H-C = 0.95 \text{ \AA}$ and $H-N = 0.88 \text{ \AA}$]. The crystal was found not to be twinned, despite the observed pseudosymmetry and the almost equal values of the lattice parameters a and b and the angles α and β .

Data collection: *SMART* (Siemens, 1995); cell refinement: *SMART*; data reduction: *SAINT* (Siemens, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 1996); software used to prepare material for publication: *SHELXL97*.

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