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Dimethylammonium 3,5-bis(trifluoromethyl)pyrazolide

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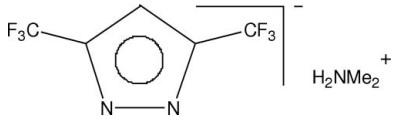
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.005 Å; R factor = 0.067; wR factor = 0.191; data-to-parameter ratio = 12.5.

The title compound, $C_2H_8N^+\cdot C_5HF_6N_2^-$, crystallizes with discrete anions and cations, which are linked by N—H···N hydrogen bonds to form centrosymmetric dimers. Geometric parameters are in the usual ranges.

Related literature

For related literature, see: Bieller *et al.* (2006); Graziani *et al.* (2002); Trofimenko (1993).



Experimental

Crystal data

$C_2H_8N^+\cdot C_5HF_6N_2^-$
M_r = 249.17
Monoclinic, $P2_1/c$
a = 5.9507 (11) Å
b = 15.4492 (19) Å
c = 11.8647 (18) Å
β = 90.362 (13)°
V = 1090.7 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.17 mm^{-1}
T = 173 (2) K
0.25 × 0.12 × 0.12 mm

Data collection

Stoe IPDSII two-circle diffractometer
Absorption correction: none
8093 measured reflections
1911 independent reflections
1287 reflections with $I > 2\sigma(I)$
R_{int} = 0.062

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.067
$wR(F^2)$ = 0.191
S = 1.02
1911 reflections
153 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3A···N1	0.90 (4)	1.94 (5)	2.834 (4)	175 (4)
N3—H3B···N2^{i}	1.02 (4)	1.81 (4)	2.826 (4)	175 (3)

Symmetry code: (i) $-x+1, -y+1, -z+1$.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2338).

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supplementary materials

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H. Vitze, H.-W. Lerner and M. Bolte

Comment

Tris(1-pyrazolyl)borates ("*scorpionates*") were invented by Trofimenko more than 30 years ago and are today well established as ligands in coordination chemistry (Trofimenko, 1993). The degree of steric crowding around the boron center appears to be an important factor influencing the stability of scorpionates. The results of earlier studies and investigations in our group have shown that the scorpinates *R*'B(3-Rpz)$_3^-$ (II) and *R*'B(3,5-R2pz)$_3^-$ (III) decompose much more easily when *R* and *R*' are bulky (Bieller *et al.* 2006, Graziani *et al.* 2002). We have now discovered that (I) was obtained as a side product of the reaction between K[3,5-(CF$_3$)$_2$Pz] and 3,5-(CF$_3$)$_2$PzH on the one side and $C_6F_5B(NMe_2)_2$ on the other side. Geometric parameters of the title compound (I) (Fig. 1) are in the usual ranges. In the crystal, anions and cations form centrosymmetric dimers linked by N—H···N hydrogen bonds (Fig.2).

Experimental

All experiments were carried out under dry argon or nitrogen using standard Schlenk techniques. 0.385 g (1.45 mmol) $(C_6F_5)B(NMe_2)_2$, 0.350 g (1.45 mmol) 3,5-(CF$_3$)$_2$PzH and 0.585 g (2.9 mmol) K[3,5-(CF$_3$)$_2$Pz] were solved in 9 ml THF. After stirring for 4 days at room temperature. The solvent was removed *in vacuo*. Thereby we obtained colourless crystals of the title compound (I) from the residual oil as a side product of the reaction of K[3,5-(CF$_3$)$_2$Pz] and 3,5-(CF$_3$)$_2$PzH with $C_6F_5B(NMe_2)_2$.

Refinement

H atoms bonded to C were refined using a riding model with fixed individual displacement parameters [U(H) = 1.2 U_{eq}(C) or U(H) = 1.5 U_{eq}(C_{methyl})] with C—H ranging from 0.95Å to 0.98 Å. The H atoms bonded to N were located in a difference map and refined freely.

Figures

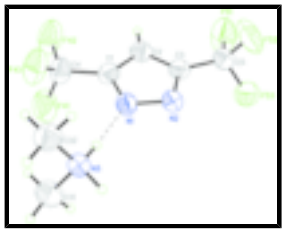
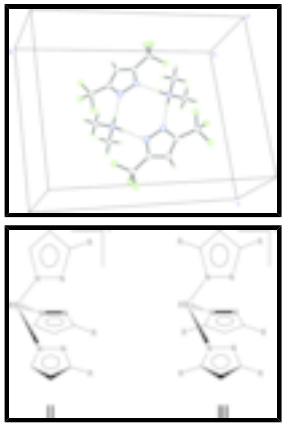


Fig. 1. Perspective view of the title compound (I) with the atom numbering scheme. Displacement ellipsoids are at the 50% probability level. H atoms are drawn as small spheres of arbitrary radii. Hydrogen bonds are shown as dashed lines.
Fig. 2. Partial packing diagram of the title compound (I); hydrogen bonds are shown as dashed lines.



Dimethylammonium 3,5-bis(trifluoromethyl)pyrazolide

Crystal data

$C_2H_8N^+ \cdot C_5HF_6N_2^-$	F_{000} = 504
M_r = 249.17	D_x = 1.517 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 5427 reflections
a = 5.9507 (11) Å	θ = 3.5–25.1º
b = 15.4492 (19) Å	μ = 0.17 mm^{-1}
c = 11.8647 (18) Å	T = 173 (2) K
β = 90.362 (13)º	Needle, colourless
V = 1090.7 (3) Å^3	0.25 × 0.12 × 0.12 mm
Z = 4	

Data collection

Stoe IPDSII two-circle diffractometer	1287 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.062
Monochromator: graphite	θ_{max} = 25.0º
T = 173(2) K	θ_{min} = 3.4º
ω scans	h = −7→7
Absorption correction: none	k = −18→17
8093 measured reflections	l = −13→14
1911 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.067	H atoms treated by a mixture of independent and constrained refinement

$wR(F^2) = 0.191$	$w = 1/[\sigma^2(F_o^2) + (0.1128P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.02$	$(\Delta/\sigma)_{max} < 0.001$
1911 reflections	$\Delta\rho_{max} = 0.30$ e Å^{-3}
153 parameters	$\Delta\rho_{min} = -0.36$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > 2\text{sigma}(F^2)$ 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}
N1	0.2583 (4)	0.55360 (16)	0.3472 (2)	0.0434 (7)
N2	0.3744 (4)	0.48549 (17)	0.3036 (2)	0.0423 (6)
N3	0.3413 (4)	0.62168 (17)	0.5653 (3)	0.0411 (6)
H3A	0.322 (6)	0.598 (3)	0.497 (4)	0.060 (11)*
H3B	0.451 (7)	0.586 (3)	0.612 (3)	0.060 (10)*
C1	0.1235 (5)	0.5846 (2)	0.2647 (3)	0.0436 (8)
C2	0.1473 (6)	0.5373 (2)	0.1658 (3)	0.0473 (8)
H2	0.0727	0.5450	0.0954	0.057*
C3	0.3069 (5)	0.4762 (2)	0.1955 (3)	0.0434 (7)
C4	−0.0324 (6)	0.6564 (2)	0.2881 (3)	0.0549 (9)
C5	0.4053 (6)	0.4066 (2)	0.1254 (3)	0.0543 (9)
C31	0.1198 (6)	0.6163 (3)	0.6213 (4)	0.0584 (10)
H31A	0.0687	0.5560	0.6218	0.088*
H31B	0.0107	0.6519	0.5800	0.088*
H31C	0.1334	0.6374	0.6989	0.088*
C32	0.4315 (6)	0.7110 (2)	0.5599 (3)	0.0538 (9)
H32A	0.5774	0.7104	0.5221	0.081*
H32B	0.4500	0.7338	0.6365	0.081*
H32C	0.3267	0.7478	0.5176	0.081*
F41	−0.1977 (5)	0.63381 (18)	0.3586 (3)	0.0956 (10)
F42	−0.1288 (5)	0.68795 (18)	0.1954 (2)	0.0934 (10)
F43	0.0655 (5)	0.72381 (15)	0.3391 (2)	0.0815 (8)
F51	0.2993 (6)	0.3963 (2)	0.0282 (2)	0.1060 (12)
F52	0.6212 (5)	0.4206 (2)	0.0986 (3)	0.0970 (10)
F53	0.4094 (5)	0.32993 (14)	0.1768 (2)	0.0803 (8)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0449 (14)	0.0413 (14)	0.0442 (16)	0.0022 (11)	0.0044 (12)	−0.0043 (11)
N2	0.0438 (13)	0.0425 (14)	0.0409 (15)	0.0030 (11)	0.0046 (11)	−0.0013 (11)
N3	0.0421 (14)	0.0421 (14)	0.0391 (16)	0.0015 (11)	0.0029 (12)	−0.0019 (12)
C1	0.0451 (17)	0.0436 (17)	0.0422 (19)	0.0023 (13)	−0.0005 (14)	0.0047 (13)
C2	0.0528 (19)	0.0509 (18)	0.0380 (18)	0.0020 (15)	−0.0044 (14)	0.0033 (14)
C3	0.0498 (17)	0.0429 (17)	0.0375 (18)	0.0003 (13)	0.0026 (13)	0.0017 (13)
C4	0.056 (2)	0.052 (2)	0.057 (2)	0.0114 (16)	−0.0082 (17)	−0.0055 (17)
C5	0.068 (2)	0.055 (2)	0.040 (2)	0.0085 (17)	0.0063 (16)	−0.0007 (15)
C31	0.0456 (18)	0.066 (2)	0.064 (2)	−0.0067 (16)	0.0150 (16)	0.0052 (18)
C32	0.053 (2)	0.0518 (19)	0.057 (2)	−0.0080 (16)	0.0023 (17)	0.0020 (16)
F41	0.0723 (16)	0.0850 (18)	0.130 (3)	0.0210 (13)	0.0406 (17)	0.0022 (16)
F42	0.113 (2)	0.0882 (18)	0.0785 (19)	0.0518 (16)	−0.0357 (16)	−0.0122 (14)
F43	0.0949 (18)	0.0526 (13)	0.0967 (19)	0.0166 (12)	−0.0201 (14)	−0.0215 (12)
F51	0.145 (3)	0.114 (2)	0.0582 (17)	0.060 (2)	−0.0348 (16)	−0.0388 (15)
F52	0.0852 (18)	0.099 (2)	0.108 (2)	0.0051 (15)	0.0506 (16)	−0.0257 (16)
F53	0.127 (2)	0.0465 (12)	0.0674 (16)	0.0130 (13)	0.0239 (15)	−0.0008 (10)

Geometric parameters (Å, °)

N1—C1	1.350 (4)	C4—F42	1.330 (4)
N1—N2	1.363 (4)	C4—F43	1.336 (4)
N2—C3	1.349 (4)	C4—F41	1.342 (5)
N3—C31	1.482 (4)	C5—F51	1.321 (4)
N3—C32	1.482 (4)	C5—F53	1.332 (4)
N3—H3A	0.90 (4)	C5—F52	1.343 (4)
N3—H3B	1.02 (4)	C31—H31A	0.9800
C1—C2	1.391 (5)	C31—H31B	0.9800
C1—C4	1.474 (5)	C31—H31C	0.9800
C2—C3	1.383 (5)	C32—H32A	0.9800
C2—H2	0.9500	C32—H32B	0.9800
C3—C5	1.481 (5)	C32—H32C	0.9800
C1—N1—N2	107.4 (2)	F43—C4—C1	113.5 (3)
C3—N2—N1	107.2 (2)	F41—C4—C1	112.7 (3)
C31—N3—C32	113.3 (3)	F51—C5—F53	107.4 (3)
C31—N3—H3A	106 (2)	F51—C5—F52	105.4 (3)
C32—N3—H3A	112 (3)	F53—C5—F52	103.7 (3)
C31—N3—H3B	107 (2)	F51—C5—C3	112.9 (3)
C32—N3—H3B	107 (2)	F53—C5—C3	113.3 (3)
H3A—N3—H3B	110 (3)	F52—C5—C3	113.5 (3)
N1—C1—C2	111.3 (3)	N3—C31—H31A	109.5
N1—C1—C4	120.2 (3)	N3—C31—H31B	109.5
C2—C1—C4	128.5 (3)	H31A—C31—H31B	109.5
C3—C2—C1	102.5 (3)	N3—C31—H31C	109.5
C3—C2—H2	128.7	H31A—C31—H31C	109.5

C1—C2—H2	128.7	H31B—C31—H31C	109.5
N2—C3—C2	111.7 (3)	N3—C32—H32A	109.5
N2—C3—C5	119.6 (3)	N3—C32—H32B	109.5
C2—C3—C5	128.7 (3)	H32A—C32—H32B	109.5
F42—C4—F43	105.9 (3)	N3—C32—H32C	109.5
F42—C4—F41	107.3 (3)	H32A—C32—H32C	109.5
F43—C4—F41	103.9 (3)	H32B—C32—H32C	109.5
F42—C4—C1	112.9 (3)		
C1—N1—N2—C3	−0.3 (3)	N1—C1—C4—F43	50.2 (5)
N2—N1—C1—C2	0.4 (4)	C2—C1—C4—F43	−133.8 (4)
N2—N1—C1—C4	177.0 (3)	N1—C1—C4—F41	−67.5 (4)
N1—C1—C2—C3	−0.3 (4)	C2—C1—C4—F41	108.4 (4)
C4—C1—C2—C3	−176.5 (3)	N2—C3—C5—F51	169.9 (3)
N1—N2—C3—C2	0.1 (3)	C2—C3—C5—F51	−10.5 (5)
N1—N2—C3—C5	179.9 (3)	N2—C3—C5—F53	47.5 (5)
C1—C2—C3—N2	0.1 (4)	C2—C3—C5—F53	−132.8 (4)
C1—C2—C3—C5	−179.6 (3)	N2—C3—C5—F52	−70.3 (4)
N1—C1—C4—F42	170.7 (3)	C2—C3—C5—F52	109.3 (4)
C2—C1—C4—F42	−13.4 (6)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3A···N1	0.90 (4)	1.94 (5)	2.834 (4)	175 (4)
N3—H3B···N2^{i}	1.02 (4)	1.81 (4)	2.826 (4)	175 (3)

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1.

Fig. 1

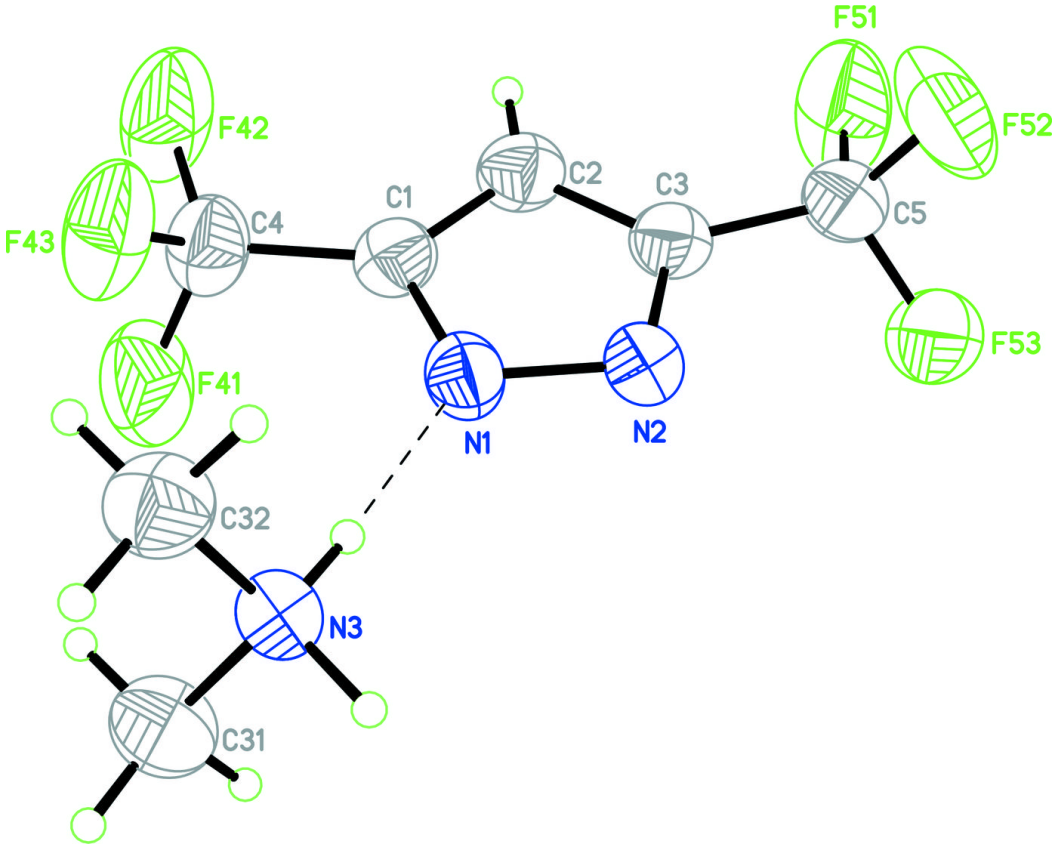


Fig. 2

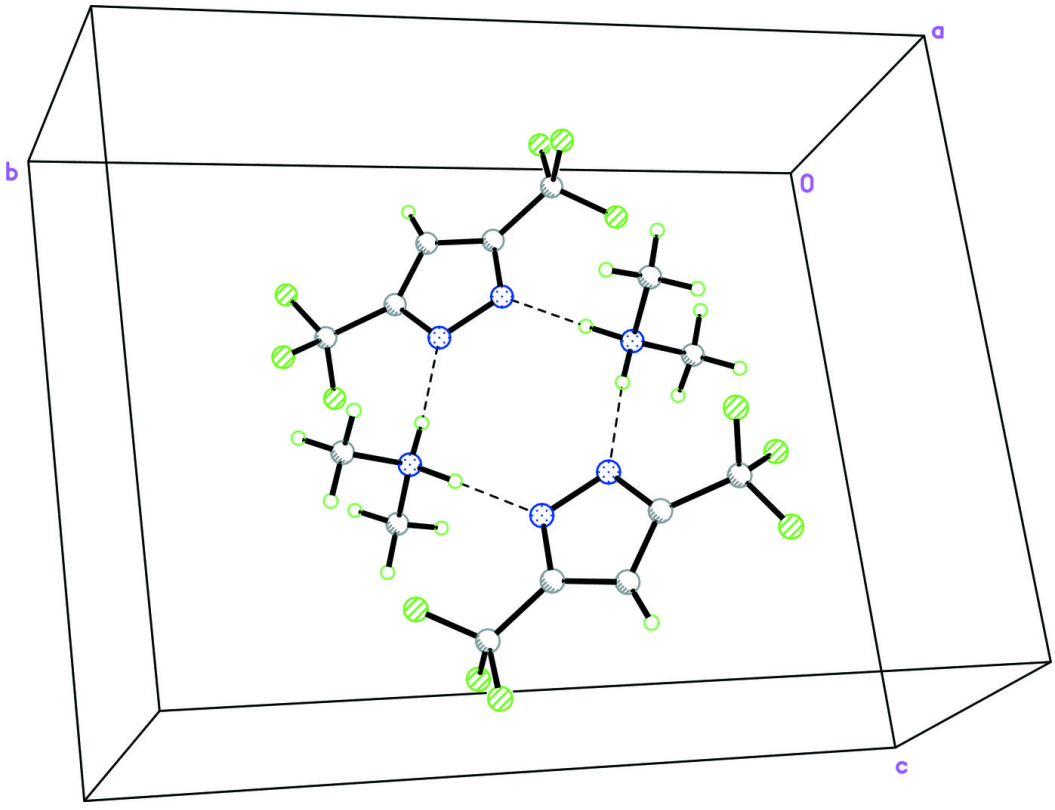
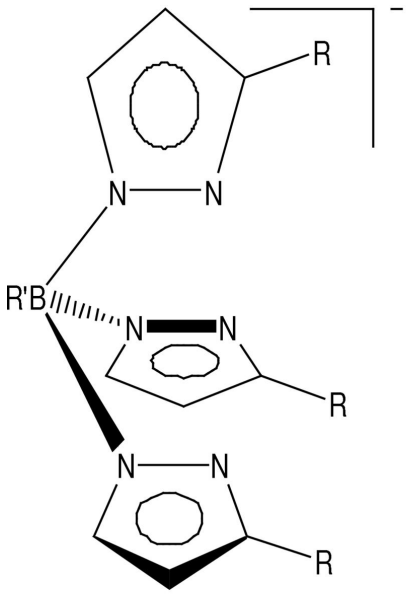


Fig. 3



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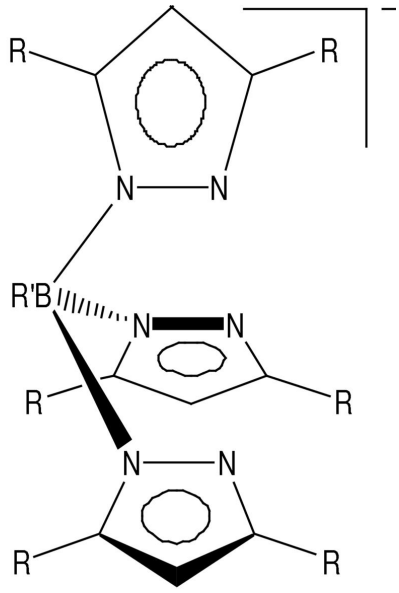

III