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Complexes of heteroscorpionate trispyrazolylborate ligands. Part XI. Weak CH/π interactions in crystals of hydrotris(3-phenylpyrazolyl) boratothallium(I) and hydrobis(5-methyl-3-phenylpyrazolyl)(3,5-dimethylpyrazol-yl)boratothallium(I) studied by X-ray crystallography

Zbigniew Ciunik[a], Tomasz Ruman[b], Marta Lukasiewicz[a], Stanislaw Wolowiec[a,b,*]

[a]*Faculty of Chemistry, University of Wroclaw, 14 Joliot-Curie St., 50-383 Wroclaw, Poland*
[b]*Faculty of Chemistry, Rzeszów University of Technology, 6 Powstańców Warszawy Ave., 35-959 Rzeszów, Poland*

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Abstract

Thallium(I) complexes of heteroscorpionate hydrobis(3-phenyl-5-methylpyrazolyl)(3,5-dimethylpyrazol-yl)borate and hydrotris(3-phenylpyrazol-1-yl)borate were studied crystallographically. Both ligands were coordinated in κ^3 fashion via N_2 atoms of pyrazol-1-yl moieties. Both compounds crystallize as centrosymmetric dimers in which weak CH/π intra- and interdimer interactions are responsible for arrangement in crystal structure.

Keywords: Trispyrazolylboratothallium complexes; Structures; CH/π interaction

1. Introduction

Tris(pyrazolyl)borates (Tp′) are convenient anionic ligands (scorpionates) to construct metal ion complexes of valuable properties [1]. The steric hindrance of those tripodal ligands can be tuned by choice of the appropriate alkyl and/or aryl substituents at 3–5 positions of pyrazolyl moieties. Thus, the properties of transition metal ion complexes of those ligands, of the general formula Tp′M(XYZ) (where M—transition metal ion, X, Y, Z—neutral or anionic exchangeable, monodentate ligands) can be influenced by steric demands of Tp's resulting in additional, metal ion centered reactivity of the complexes, including catalytic activity [2–14]. Our systematic studies of transition metal ion complexes of Tp′ ligands [15–22] are focused on construction of low-symmetry Tp′ ligands of desired spatial anisotropy. Tp′ ligands can be characterized by boron-centered bite angle or metal-ion centered bite angle in their complexes. Due to readily crystallization, Tp′Tl(I) complexes are particularly useful species used both for purification of Tp′ ligands and for their structural characterization [23–39]. Recently the subject of thallium(I) complexes with Cp and structurally related Tp ligands was extensively reviewed [40].

Here we report on the crystal and molecular structure of heteroscorpionate hydrobis(3-phenyl-5-methylpyrazolyl)(3,5-dimethylpyrazol-yl)boratothallium(I) and homoscorpionate hydrotris(3-phenylpyrazol-1-yl)boratothallium(I) complexes, for which the structural details were unavailable till now.

2. Experimental

2.1. Syntheses

3-Phenylpyrazole and potassium hydrotris(3-phenylpyrazolyl)borate KTp^{Ph} were synthesized according to known procedure [41]. Hydrotris(3-phenylpyrazolyl)boratothallium(I) ($Tp^{Ph}Tl(I)$) was obtained according to slightly modified published method [40], i.e. by addition of 120 mg of KTp^{Ph} (0.25 mmol) dissolved in 25 cm^3 tetrahydrofuran

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* Corresponding author. Address: Faculty of Chemistry, Rzeszów University of Technology, 6 Powstańców Warszawy Ave., 35-959 Rzeszów, Poland. Tel.: +48-17-8651657; fax: +48-17-8543655.
E-mail address: sw@prz.rzeszow.pl (S. Wolowiec).

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into the stock water solution of thallium(I) nitrate (50 cm^3, 0.1 M). The reaction mixture was stirred 2 h and the product was extracted with 50 cm^3 methylene dichloride. The organic phase was washed twice with 100 cm^3 water followed by removal of methylene dichloride. The crude product was dissolved in hot toluene and left for crystallization from toluene/heptane at 4 °C (130 mg, 0.20 mmol, 80% yield). The same crystals were also obtained by crystallization from chloroform. In both cases the crystals were examined by X-ray diffraction method.

Anal. Calcd. for $C_{27}H_{22}N_6BTl$: C, 50.22; H, 3.43; N, 13.02. Found: C, 50.51; H, 3.60; N, 12.84. Sodium hydrobis(3-phenyl-5-methylpyrazolyl)(3,5-dimethylpyrazol-yl)borate (Na[HB(3-Ph,5-Mepz)$_2$(3,5-diMepz)]) has been obtained as before [20]. The thallium complex [HB(3-Ph,5-Mepz)$_2$(3,5-diMepz)]Tl(I) has been synthesized in the same way as Tp^{Ph}Tl(I). The crystals suitable for crystallographic studies were grown from THF/heptane.

Anal. Calcd. for $C_{25}H_{26}BN_6Tl$: C, 47.99; H, 4.19; N, 13.43. Found: C, 48.31; H, 4.31; N, 13.16. ^{1}H NMR (CDCl$_3$, 298 K): 7.9–11.5 (15H, overlapping multiplets, *o*-, *m*-, and *p*- H of 5-Ph and 3-Ph); 5.93 (1H, s, 4-H(3,5-diMepz); 6.35 (2H, s, 4-H(3-Ph,5-Mepz); 4.72 (1H, B-H, broad s); 2.17 (6H, two overlapping s, 3- and 5-CH$_3$(3,5-diMepz), assigned by NOESY experiment); 2.03 (3H, 5-CH$_3$(3-Ph,5-Mepz)).

2.2. X-ray crystallography

Crystal data are given in Table 1, together with refinement details. All measurements of crystals were performed on a Kuma KM4CCD κ-axis diffractometer with graphite-monochromated Mo Kα radiation. Both crystals were positioned at 65 mm from the KM4CCD camera. 612 frames were measured at 0.75° intervals with a counting time of 15 s. The data were corrected for Lorentz and polarization effects. Absorption correction based on least-squares fitted against to $|F_c| - |F_o|$ differences were also applied [42]. Data reduction and analysis were carried out with the Kuma Diffraction (Wroclaw) programs. The structures were solved by direct methods (program SHELXS97 [43]) and refined by the full-matrix least-squares method on all F^2 data using the SHELXL97 [44] programs. Non-hydrogen atoms were refined with anisotropic thermal parameters; hydrogen atoms were included from geometry of molecules but were not refined.

Table 1
Crystal data and structure refinement

Compound	**1**	**2**
Empirical formula	$C_{27}H_{22}BN_6Tl$	$C_{25}H_{26}BN_6Tl$
Formula weight	645.69	625.70
T (K)	100(2)	100(2)
λ (Å)	0.71073	0.71073
Crystal system	Triclinic	Monoclinic
Space group	$P\bar{1}$	$C2/c$
a (Å)	10.609(2)	20.139(2)
b (Å)	10.968(2)	11.8827(10)
c (Å)	12.098(2)	24.976(2)
α (°)	66.40(3)	90
β (°)	87.58(3)	111.080(15)
γ (°)	72.32(3)	90
V (Å^3)	1224.0(4)	5576.9(9)
Z	2	8
D_c (Mg m^{-3})	1.752	1.490
μ (mm^{-1})	6.626	5.814
F(000)	624	2432
Crystal size/mm	0.12 × 0.10 × 0.10	0.12 × 0.12 × 0.10
θ range for data collection (°)	3.43-28.86	3.50-28.44
h, k, l ranges	$-12 \rightarrow 14$, $-14 \rightarrow 14$, $-16 \rightarrow 15$	$-26 \rightarrow 23$, $-15 \rightarrow 15$, $-33 \rightarrow 33$
Reflections collected	8741	18808
Independent reflections (R_{int})	5585 (0.0742)	6529 (0.1344)
Data/parameters	5585/316	6529/302
Transmission coefficients min./max.	0.5036/0.5571	0.5421/0.5940
Goodness-of-fit (F^2)	1.059	1.087
Final R_1/wR_2 indices ($I > 2\sigma_I$)	0.0406/0.1092	0.0767/0.2055
Largest diff. peak/hole (e Å^{-3})	2.951/−3.520	3.314/−3.149

3. Results and discussion

Two scorpionate trispyrazolylboratethallium(I) complexes have been studied structurally here: the homoscorpionate hydrotris(3-phenyl)pyrazolyl)boratothallium(I) (**1**) and the heteroscorpionate hydrobis(3-phenyl-5-methylpyrazolyl)(3,5-dimethylpyrazolyl)borato thallium(I) (**2**). Both complexes crystallized as centrosymmetric dimers, in which Tp′ ligands are coordinated in κ^3 fashion via N$_2$ atoms of pyrazol-1-yl moieties (Fig. 1). Selected bond distances and angles are given in the caption of Fig. 1. The Tl–N bond lengths for both **1** and **2** (2.513(8)–2.620(4) Å) and N–Tl–N bond angles ranging from 69.39(14) to 79.56(13)° fall into the region typical for all Tp′Tl complexes based on 3-Rpyrazolyl moieties (where R—aromatic substituent, entries 1–6 in Table 2). Longer Tl–N distances were observed in Tp′Tl complexes with large steric hindrance around metal center (hydrotris(3-(9-triptycyl)pyrazolyl)boratothallium(I)—2.76 Å [23] and hydrotris(7-*t*-butylindazol-2-yl)boratothallium(I)—2.66 Å [24] and in those cases where additional donor atoms were involved: (hydrotris(3-(pyrrolidinylcarbonyl)pyrazol-1-yl)boratothallium(I)—2.71 Å [25] and hydrotris(3-(2-pyridyl)pyrazolyl)boratothallium(I)—2.67 Å [26] as well as in Tp$^{(CF_3)}$$_2$ Tl complex (2.71 Å), in the latter possibly due to electronic reason [27].

There are weak edge-to-face CH/π interactions present in the lattice of both **1** and **2**, which are responsible for crystal organization. This kind of interaction has been recognized as important factor responsible for crystal organization in many solids [45]. The crystal packing of **1** with three edge-to-face contacts is presented at Fig. 2A.

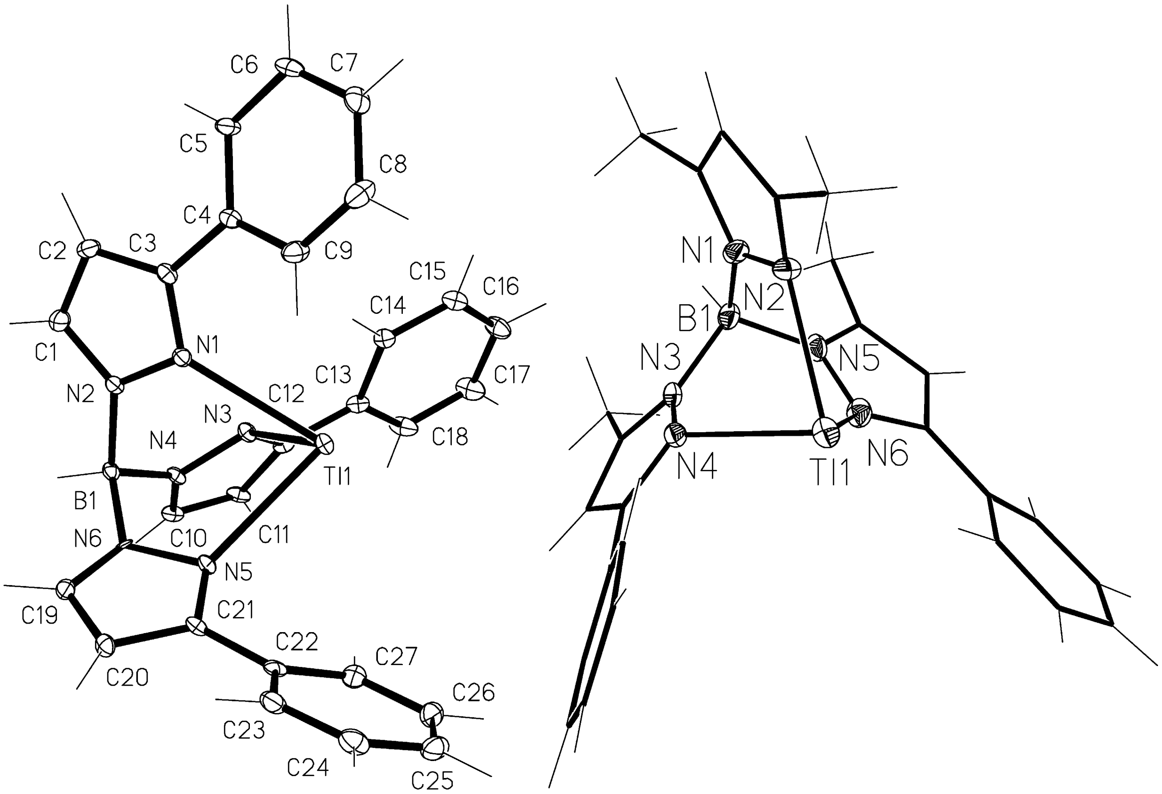

Fig. 1. Molecular structures of **1** (left) and **2** (right) with crystallographic numbering. Selected bond lengths (Å) and angles (°), **1**: Tl(1)–N(1), 2.598(4); Tl(1)–N(3), 2.596(4); Tl(1)–N(5), 2.620(4); N(3)–Tl(1)–N(1), 69.39(14); N(3)–Tl(1)–N(5), 79.56(13); N(1)–Tl(1)–N(5), 76.46(14), **2**:Tl(1)–N(2), 2.513(8); Tl(1)–N(4), 2.519(7); Tl(1)–N(6), 2.609(8); N(2)–Tl(1)–N(4), 75.2(3); N(2)–Tl(1)–N(6), 78.2(3); N(4)–Tl(1)–N(6), 75.9(2).

The 3-phenyl moieties within $Tp^{Ph}Tl–TlTp^{Ph}$ dimers are arranged in specific way; there are three (Ph)C–H Ph(centroid) intermolecular, intradimer contacts of edge-to-face type, with two H_{meta} Ph and one H_{ortho} Ph distances equal 3.03, 2.57, and 3.26 Å, respectively, and additional interdimer (Ph)C–H Ph(centroid) contact with H Ph distance of 2.99 Å (Table 3). These result in formation of polymeric chains composed of $Tp^{Ph}Tl$ $TlTp^{Ph}$ dimers with aromatic hydrophobic surface rendering the complex totally insoluble in common organic solvents. Similar interactions were observed in other cases. In dimeric $Tp^{p\text{-}Tol}Tl$ complex the R-phenyl rings of two Tp′ ligands intermesh and their aromatic rings are stacked with a distance longer than 4.0 Å without interdimer contacts [28]. The dimeric $Tp^{(2,4(OMe)_2\text{-}}$ Ph)Tl shows both intra- and interdimer C–H···Ph contacts, similarly as those in **1** [29]. Thus, there are three edge-to-face intradimer C(Ph)H_{meta} Ph contacts with 3.01 Å distance and three interdimer *o*-CH_3 Ph contacts with 2.91 Å distance (calculated for idealized 0.96 Å C–H bond length) in trigonal crystal.

Moreover, the intermolecular CH(fused benzene ring in dihydrobis(indazol-1-yl)boratothallium(I))/π interaction was found to be responsible for crystal packing [46], and chiral supramolecular organization in crystals of tris(indazol-1-yl)boratothallium(I) ($Tp^{Bo}Tl$) [47] as well as in bis-ligand $Tp^{Bo}_2M(II)$ (M = Fe, Co, Ni, Cu, and Zn) complexes [48].

The intradimer Tl···Tl contact in **1** is equal to 3.85 Å. Such interaction has been found previously in hydrotris(3-(*p*-tolyl)pyrazolyl)boratothallium(I) (3.86 Å) [28] and in hydrotris(3-(2,4-dimethoxyphenyl)pyrazolyl)boratothallium(I) (3.99 Å) [29] but it was absent in hydrotris(3,5-di-(*p*-*t*-butyl)phenylpyrazolyl)boratothallium(I) [30] due to

Table 2
Geometry of the C–H···Ph(centroid) hydrogen bonds for **1** and **2**

C–H···Ph	H···Ph	∠C–H···Ph
Compound 1		
C(8)–H(8)···Ph1$^{(i)}$	3.03	134
C(9)–H(9)···Ph1$^{(i)}$	3.26	123
C(15)–H(15)···Ph2$^{(i)}$	2.57	154
C(23)–H(23)···Ph3$^{(ii)}$	2.99	154
Compound 2		
C(23)–H(23A)···Ph1$^{(iii)}$.	2.83	137
C(18)–H(18A)···pz$^{(iv)}$.	2.63	148

Symmetry transformations used to generate equivalent atoms: (none) *x*, *y*, *z*; (i) 1 − *x*, −*y*, −*z*; (ii) 2 − *x*, −*y*, −*z*; (iii) *x*, 1 + *y*, *z*; (iv) −*x*, 1 − *y*, −*z*.

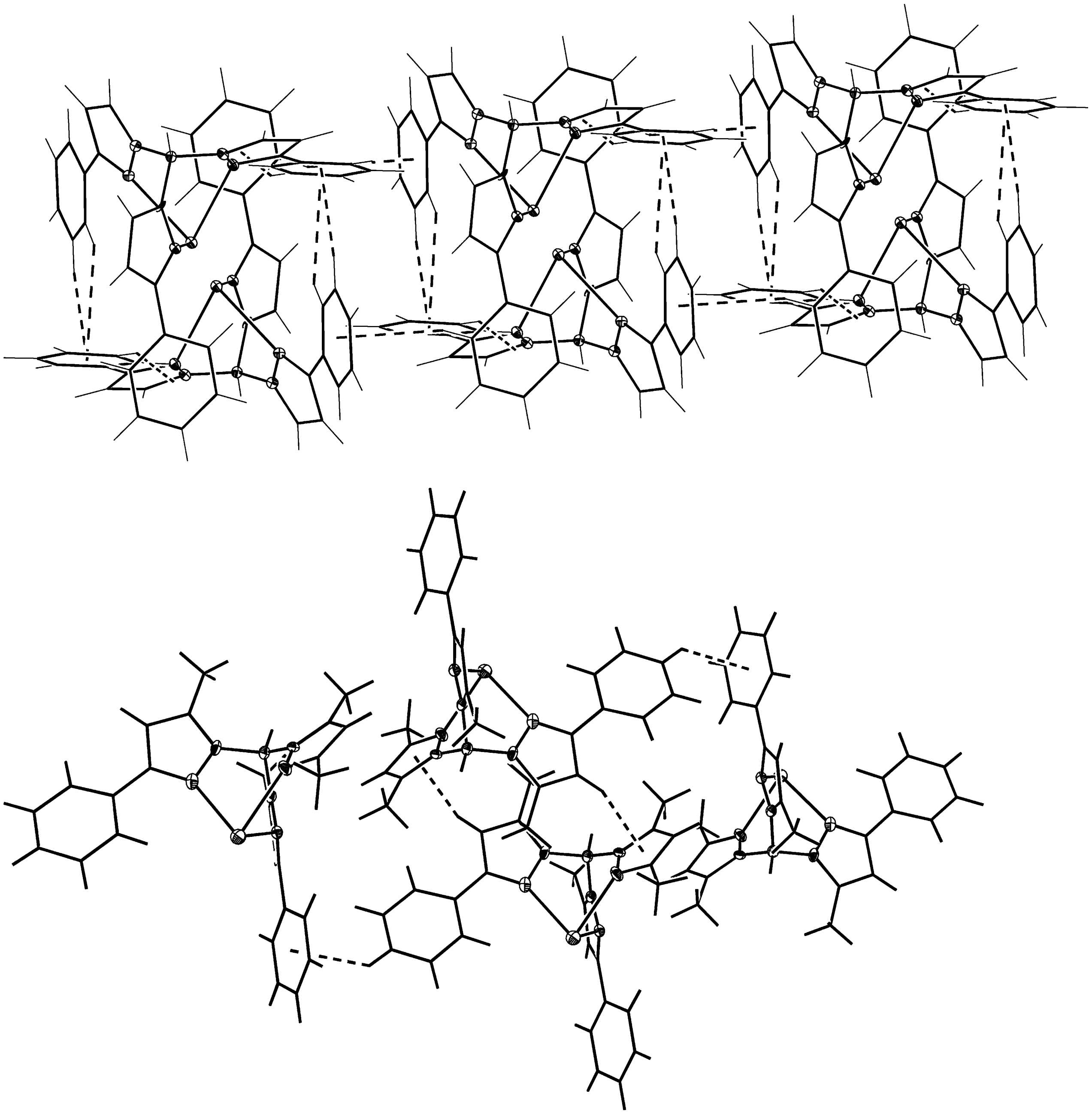

Fig. 2. Crystal packing diagram with CH/π contacts in the crystal structure of **1** (top) and **2** (bottom).

considerable steric hindrance from *t* Bu substituents. Neither it was found in hydrotris(3-(2-methoxyphenyl)pyrazol-1-yl)boratothallium(I) [31] due to additional involvement of two methoxy group oxygens engaged in coordination.

Unlike in **1**, two intradimer CH/π interactions occur between 4-H(3-Phpz) and 3,5-dimethylpyrazolyl π acceptor in **2**. Two thallium metal ions point away from the center of dimer (Tl···Tl distance is 10.70 Å). Additionally, there are two CH/π (3-PH) interdimer interactions, which are responsible for formation of 1D chains composed of dimers.

The thallium-centered (*α*) and boron-centered (*β*) bite angles for Tp′Tl complexes (Table 3) reflect the steric hindrance of Tp′ ligand depending on 3- and/or 5-substituents. The boron-centered bite angle has been introduced by us in order to compare the metal ion-independent ligand steric demands [20]. Thus, this parameter is attributed to ligand. The value of *β* for heteroscorpionate $[HB(3\text{-}Ph,5\text{-}Mepz)(3,5\text{-}diMepz)]^-$ varies depending on coordination mode of the ligand and coordination number of central metal ion and is equal: 90.0° (bidentate in $Tp'Rh(CO)_2$, [49]) 86.9° (terdentate in Tp′Tl, **2**), 80.4° (terdentate in Tp’Co(NCS)(THF), [20]).

The comparison made for Tp’Tl(I) complexes, in which Tp′ ligands are κ^3 coordinated via three N_2 pyrazol-1-yl

Table 3
Values of thallium-centered (α), boron-centered (β) bite angles and averaged Tl–N(pz) bond lengths in Tp'Th(I) complexes. The fourth column shows the Tl–Tl distance in Tp′Tl TlTp′ dimers

Entry	Compound	α (°)[a]	β (°)[a]	Tl–N (Å)	Tl–Tl (Å)
1	Hydrotris(3-phenylpyrazolyl)boratothallium(I) ($Tp^{Ph}Tl$)[h]	167.1	84.0	2.61	3.85
2	Ferrocenyltris(3-phenylpyrazolyl)boratothallium(I) [50]	167.1	85.8	2.56	
3	Hydrotris(3-(*p*-tolyl)pyrazolyl)boratothallium(I) ($Tp^{p\ MePh}Tl$) [28]	161.0	79.1	2.59	3.86
4	Hydrotris(3-(2-methoxyphenyl)pyrazolyl)boratothallium(I) [31]	151.4	77.1	2.59[f]	
5	Hydrotris(3-(2,4-dimethoxyphenyl)pyrazolyl)boratothallium(I) ($Tp^{2,4\text{-}diOMePh}Tl$) [29]	164.2	80.6	2.58	3.99
6	Hydrotris(3,5-di-(*p*-*t*-butyl)phenylpyrazolyl)boratothallium(I) ($Tp_2^{(p\text{-}t\ BuPh)}Tl$) [30]	166.9[b]	87.1[b]	2.60	
7	Hydrotris(3-(2-pyridyl)pyrazolyl)boratothallium(I) [26]	164.2	73.3	2.67[e]	
8	Hydrotrispyrazolylboratothallium(I) (TpTl) [32]	243.5	120.2	2.63	
9	Hydrotris(3,5-dimethylpyrazolyl)boratothallium(I) ($Tp_2^{Me}Tl$) [33]	190.8	97.4	2.52	
10	Hydrotris(3-*t*-butylpyrazolyl)boratothallium(I) ($Tp^{t\ Bu}Tl$) [34]	162.5	81.8	2.58	
11	Hydrotris(3-*t*-butyl-5-methylpyrazolyl)boratothallium(I) ($Tp^{t\ Bu,Me}Tl$) [35]	172.4	88.2	2.50	
12	Hydrobis(3-*t*-butylpyrazolyl)(pyrazolyl)boratothallium(I) [36]	217.8	107.7	2.62	
13	Hydrotris(3,5-di-trifluoromethylpyrazolyl)boratothallium(I) [27]	172.4[c]	76.2[c]	2.71	
		199.9[d]	97.7[d]		
14	Hydrotris(3-trifluoromethyl,5-(2-thienyl)pyrazolyl)boratothallium(I) [37]	172.4[c]	81.2[c]	2.62	
		196.6[d]	99.0[d]		
15	Hydrotris(3-(9-triptycyl)pyrazolyl)boratothallium(I) ($Tp^{Triptycyl}Tl$) [23]	136.0	73.8	2.76	
16	Hydrotris(7-*t*-butylindazol-2-yl)boratothallium(I) [24]	150.1	75.6	2.66	
17	(Hydrotris(1,4-dihydroindeno(1,2-c)pyrazol-1-yl)borato)thallium(I) ($Tp^{inden}Tl$) [38]	164.1	89.8	2.53	
18	Hydrotris(7(*R*)-*t*-butyl-4(*R*)-Me-4,5,6,7-tetrahydro-2-indazolyl)hydroboratothallium(I) [39]	167.7	83.9	2.56	
19	Hydrotris(7(*R*)-*i*-propyl-4(*R*)-Me-4,5,6,7-tetrahydro-2-indazolyl)hydroboratothallium(I) [39]	171.2	81.4	2.55	
20	Hydrotris(3-(pyrrolidinylcarbonyl)pyrazol-1-yl)boratothallium(I) [25]	164.5	78.7	2.71 [g]	
21	Hydrobis(3-phenyl-5-methylpyrazolyl)(3,5-dimethylpyrazolyl)-boratothallium(I)[h]	189.2	86.9	2.55	

a, measured for the atom of 3-R substituent (non-hydrogen atom) closest to metal ion, unless otherwise stated; b, measured for *o*-C(Ph); c, measured to fluorine atom of CF_3; d, measured to carbon atom of CF_3; e, Tl-N(py); mean contact distance 3.18 Å; f, two methoxy group oxygen atoms are in close contact with Tl(I) (3.20 Å); g, three carbonyl oxygen atoms are at the mean distance of 3.00 Å from Tl atom; h, this work.

atoms indicates clearly that unsubstituted Tp and 3,5-diMe substituted ligands possess the smallest steric hindrance, whereas the 3-(9-triptycyl) substituent produces the most sterically demanding homoscorpionate Tp′ ligand. Considering the boron-centered bite angle, the Tp′ ligands can be put in the following order of increasing steric hindrance of 3,5 substituents:

$$Tp < Tp^{Bo} < Tp_2^{Me} < Tp^{inden} < Tp^{t\,Bu,Me} < [HB(3\text{-}Ph,5\text{-}Mepz)_2(3,5\text{-}diMepz)]^- < Tp_2^{(p\text{-}t\,BuPh)} < Tp^{Ph} < Tp^{t\,Bu} < Tp^{2,4\text{-}diOMePh} < Tp^{pMePh} < Tp^{Triptycyl}$$

According to the values of β (see above) the steric hindrance from 3-aryl substituents is comparable to that from 3-*t*Bu one. Moreover, the presence of 5-R substituents different from hydrogen atom results in increase of bite angle which can be seen when one compare the values of β for $Tp^{t\,Bu}$ (81.8°) and $Tp^{t\,Bu,Me}$ (88.2°) or Tp^{pMePh} (79.1°) and $Tp_2^{(p\text{-}t\,BuPh)}$ (87.1°). Similarly, the replacement of ferrocenyl residue for boron-attached hydrogen in Tp^{Ph} [50] resulted in increase of β from 84.0 to 85.8°. These steric effects can be assigned to the fact, that both of those structural modifications of $Tp^{3\text{-}R}$ cause the severe twist of coordinated pyrazolyl rings in respect to the B-Tl axis, or more precisely the deviation of $B-N_1-N_2-Tl$ dihedral angle from 0°. These effects demonstrate the flexibility of Tp′ ligands in teracoordinate Tp′Tl complexes. As one can anticipate, the bite angles for heteroscorpionate **2** (entry 21 in Table 3) are intermediate between $Tp^{Ph}Tl$ and Tp_2^{Me} although the measured β for **2** (86.9°) is apparently larger from the averaged value for those two (84.7°).

In conclusion, the structural data are obtained from X-ray analysis of crystals of $Tp^{Ph}Tl(I)$ and [HB(3-Ph,5-Mepz)$_2$(3,5-diMepz)]Tl(I) obtained from various organic solvents as dimers stabilized by numerous weak edge-to-face C(Ph)H···Ph interactions. These weak interactions are responsible for arrangement of dimers rather than thallium-thallium interaction.

4. Supplementary data

Crystallographic data for the structures reported in this paper have been deposited with the Cambridge

Crystallographic Data Centre, CCDC no. 208716 for compound 1, and CCDC no. 208715 for 2. Copies of this information may be obtained free of charge from the Director, CCDC, 12 Union Road, Cambridge 1EZ. UK (fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk).

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