

Syntheses, Characterization and X-ray Structure of Potassium Hydrotris(2-Mercaptothiazolyl)Borate, KMt, and Potassium Hydrotris(Methimazole)Borate, KTm

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Os compostos hidrottris(2-mercaptotiazolil)borato de potássio, KMt, (**1**), e hidrottris(2-metimidazolil)borato de potássio, KTm, (**2**), foram preparados ao se reagir a amina tiol correspondente com KBH_4 . Foram caracterizados por métodos espectroscópicos e análise elementar. As estruturas no estado sólido de $\text{KMt}\cdot 4\text{H}_2\text{O}$ e do composto **2** foram determinadas por análises de difração de raios-X. A maior maciez do ânion em $\text{KMt}\cdot 4\text{H}_2\text{O}$, em comparação com **2**, é indicada pela ausência de interação entre ele e o cátion, que possui moléculas de água em sua esfera de coordenação. Em $\text{KMt}\cdot 4\text{H}_2\text{O}$ a geometria em torno do íon potássio é octaédrica distorcida e em **2**, pirâmide de base quadrada distorcida. Em **2** o íon potássio está coordenado a dois átomos de enxofre dos anéis de um mesmo ânion e a três outros átomos de enxofre pertencentes a unidades KTm vizinhas. Ambos os compostos possuem uma estrutura polimérica.

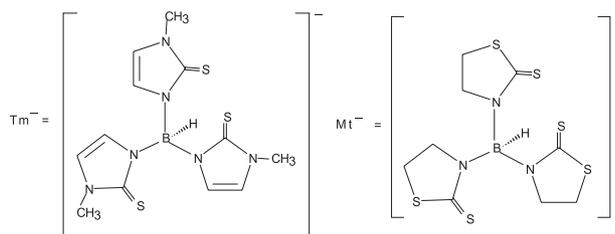
Potassium hydrotris(2-mercaptotiazolyl)borate, KMt, (**1**) and potassium hydrotris(2-methimidazole), KTm, (**2**), were prepared by reacting the appropriate amine thiol with KBH_4 . They were characterized by spectroscopic methods and elemental analysis. Solid state structures of $\text{KMt}\cdot 4\text{H}_2\text{O}$ and compound **2** were determined by X-ray diffraction analyses. The softer nature of the anion in the former as compared to **2** is indicated by the lack of interaction of the anion with the cation, that has water molecules in its coordination sphere. In $\text{KMt}\cdot 4\text{H}_2\text{O}$ the geometry around the potassium cation is distorted octahedral and in **2**, distorted square pyramidal. In **2** the potassium ion is coordinated to two sulfur atoms of one hydrotris(methimidazole)borate unit and to other three sulfur atoms belonging to neighboring KTm units. Both compounds have a polymeric structure.

Keywords: hydroborate S,N ligands potassium salts, X-ray structure

Introduction

The cyclopentadienyl anion, C_5H_5^- , Cp^- , is a useful supporting ligand and has been used as such with almost all transition metals. However, in some cases, the synthesis of the CpM starting material is rather difficult, especially when M = early transition metal, mainly due to the lack of stability of the intermediates that originate the metallocenes.¹ The hydrotris(pyrazolyl)borate ligand, $\text{HB}(\text{pz})_3^-$, Tp^- , belongs to a novel class of potentially tridentate proligands, which are isolobal with Cp^- . For many applications it is easier to handle, more stable and cheaper, than Cp^- .² The pyrazolyl nitrogen atoms are hard donors and analogues of this proligand bearing softer donor atoms,

such as sulfur, are of interest. For example, by following Trofimenko's procedure for the preparation of the Tp^- proligand,² Reglinski *et al.* demonstrated that reaction of methimazole (2-mercapto-1-methylimidazol) with NaBH_4 yields hydrotris(methimazole)borate, Tm^- .³ A similar but softer proligand, hydrotris(2-mercaptotiazolyl)borate, Mt^- , derived from 2-mercaptotiazoline, has also been prepared as the sodium salt.⁴



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In this paper we wish to describe the synthesis and characterization of the potassium salt of Mt^- and the solid-state structures of the potassium salts of Mt^- and Tm^- , determined by X-ray diffraction analyses.

Experimental

General considerations

KBH_4 and 2-mercapto-1-methylimidazole were purchased from Fluka, and 2-mercaptothiazoline, from Aldrich. All reagents were used as received. Potassium hydrotris(methimazole)borate, KTm (**2**), was prepared as described previously.⁵ Elemental analyses (C, H, N) were performed with a Perkin Elmer mod. PE-2400 CHN instrument. IR spectra were recorded on a Perkin Elmer 283B instrument. 1H and ^{13}C spectra were recorded on a Bruker Avance DPX200 spectrometer operating at room temperature, using tetramethylsilane as an internal reference. ^{11}B NMR spectrum was recorded on a Bruker Avance DPX400 spectrometer operating at room temperature, using $BF_3 \cdot Et_2O$ as an internal reference.

Preparation of potassium hydrotris(2-mercaptothiazolyl)borate, KMt (**1**)

Finely divided KBH_4 (0.24 g, 4.45 mmol) and 2-mercaptothiazoline, (2.12 g, 17.80 mmol) were placed together with a magnetic stirring bar into a Schlenk flask. The flask was connected to a volumetric device and placed into a bath resting on a heating and stirring plate. The mixture was kept to 80 °C where upon evolution of H_2 commences. The temperature was controlled at 120 °C until 13.4 mmol of H_2 had been evolved. Then the mixture was allowed to cool down to room temperature and 20 mL of CH_2Cl_2 was added. This mixture was stirred and allowed to rest for 15 min. after which a white solid settled in the bottom of the flask. The solid and the solution were separated and this process was repeated three times. The remaining white solid was dried under vacuum. Yield 75%. 1H NMR (200 MHz, D_2O): δ 3.24 (t, 6H, SCH_2 , J_{H-H} 7.6 Hz), 4.06 (t, 6H, NCH_2 , J_{H-H} 7.6 Hz). $^{13}C\{^1H\}$ NMR (50 MHz, D_2O): δ 31.31 (s, SCH_2), 59.65 (s, NCH_2), 197.19 (C=S). ^{11}B NMR (128.32 MHz, $BF_3 \cdot Et_2O$): δ -5.15. IR (CsI, nujol mulls) ν_{max}/cm^{-1} : 2440 (B-H), 2240 (C-N), 1290 (C=S). Anal. for $C_9H_{13}BN_3KS_6$ calcd (found): C, 27.06 (26.66); H, 3.24 (3.24); N, 10.24 (10.36)%. mp (d): 220 °C.

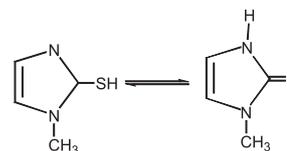
A sample of compound **1** was dissolved in ethanol and left at room temperature for 37 days after which time crystals of $KMt \cdot 4H_2O$ suitable for X-ray analysis were obtained. Anal. for $C_9H_{21}BKN_3O_4S_6$ calcd (found): C, 22.63 (23.34); H, 4.44 (4.30); N, 8.80 (9.09%).

X-ray structure analysis of compounds $KMt \cdot 4H_2O$ (**1**) and KTm (**2**)

Good quality crystals of KTm were obtained from an aqueous solution that was left at room temperature for several days. All measurements were made at 120 K on an Enraf-Nonius Kappa-CCD diffractometer (95 mm CCD camera on κ -goniostat) with graphite monochromated $Mo K\alpha$ ($\lambda = 0.71073 \text{ \AA}$) radiation. The temperature was controlled using an Oxford Cryosystem low temperature device. Data collections (φ scans and ω scans with κ offsets) were carried out using the *COLLECT* program.⁶ Integration and scaling of the reflections were performed with the *HKL Denzo-Scalepack* system of programs.⁷ The final unit cell parameters were based on all reflections using *HKL Scalepack*.⁷ Absorption corrections were carried out using the multi-scan method.⁸ The structures were solved using Patterson methods with *SHELXS-97*.⁹ The models were refined by full-matrix least-squares procedures on F^2 using *SHELXL-97*.¹⁰ The *WINGX* program was used to analyze and prepare the data for publication.¹¹ Crystal data, data collection procedures, structure determination methods and refinement results are summarized in Table 1. The ORTEP's were prepared using the ORTEP-3 for Windows.¹²

Results and Discussion

Amine thiols such as methimazole exist as tautomers of the thiol and thione forms:



As pointed out by Reglinski and co-workers³ and Lobia and co-workers,¹³ in these systems the thione form predominates, the acidic hydrogen is bound to the nitrogen atom, thus allowing the preparation of the sodium salt of Tm^- . Our preparation is similar to that of Lobia and co-workers,¹³ with some modifications in temperature and solvent washes, which led to better yields of Tm^- , as the potassium salt.⁵ The NMR data of the two compounds indicated that the three rings were equivalent, in agreement with the literature,³ and confirmed by the solid state structures determined by X-ray diffraction studies, discussed below.

Solid State Structural data of $KMt \cdot 4H_2O$ (**1**) and KTm (**2**)

Although the preparation of compound **1** has been previously described in the literature, its crystal structure

Table 1. Crystal data and structure refinement for compounds $\text{KMt.4H}_2\text{O}$ (1) and KTm (2)

Empirical formula	$\text{C}_9\text{H}_{21}\text{BKN}_3\text{O}_4\text{S}_6$	$\text{C}_{12}\text{H}_{16}\text{BKN}_6\text{S}_3$
Formula weight	477.56	390.40
Crystal system	Monoclinic	Triclinic
Space group	$P2_1/n$	$\bar{1}$
Unit cell dimensions	$a = 14.0090(2) \text{ \AA}$ $b = 5.8960(1) \text{ \AA}$ $c = 24.7920(3) \text{ \AA}$ $\alpha = 90^\circ$ $\beta = 93.70^\circ$ $\gamma = 90^\circ$	$a = 7.9210(3) \text{ \AA}$ $b = 10.2870(4) \text{ \AA}$ $c = 11.7970(6) \text{ \AA}$ $\alpha = 82.241(2)^\circ$ $\beta = 83.221(2)^\circ$ $\gamma = 70.992(2)^\circ$
Volume	$2043.47(5) \text{ \AA}^3$	$897.70(7) \text{ \AA}^3$
Z	4	2
Density (calculated)	1.552 Mg/m^3	1.444 Mg/m^3
Absorption coefficient	0.891 mm^{-1}	0.650 mm^{-1}
$F(000)$	992	404
Crystal size	$0.20 \times 0.05 \times 0.05 \text{ mm}^3$	$0.06 \times 0.04 \times 0.02 \text{ mm}^3$
Theta maximum	1.63 to 25.00°	2.59 to 25.00°
Reflections collected	6778	5914
Independent reflections	3583 [$R(\text{int}) = 0.0168$]	3151 [$R(\text{int}) = 0.0651$]
Data	3583	3151
parameters	217	211
Goodness-of-fit on F^2	1.149	1.129
Final R indices [$I > 2\sigma(I)$]	$R1 = 0.0316$, $wR2 = 0.1018$	$R1 = 0.0454$, $wR2 = 0.1205$
R indices (all data)	$R1 = 0.0426$, $wR2 = 0.1313$	$R1 = 0.0575$, $wR2 = 0.1554$
Largest diff. peak and hole	0.843 and $-0.758 \text{ e. \AA}^{-3}$	0.357 and $-0.544 \text{ e. \AA}^{-3}$

has not been reported so far.⁴ The geometry around the boron atom in $\text{KMt.4H}_2\text{O}$ is almost a regular tetrahedron. The anion has an approximate C_3 symmetry (average N-B-N = $108.6(3)^\circ$) with the pseudo-symmetry C_3 axis parallel to the crystallographic b axis, Figure 1.

The rings are bound to the boron through the nitrogen atoms. The B-H bond is located along the C_3 pseudo-

symmetry axis. Each ring is twisted around the B-N bond and the average angle between the three planes formed by them is $85(3)^\circ$. In each unit the potassium cation is coordinated to six water molecules. Two of them are symmetrically dependent of other neighboring $\text{K}(\text{H}_2\text{O})_6$ units. A view focusing on the potassium solvate sphere is shown in Figure 2. The soft nature of the Mt anion is

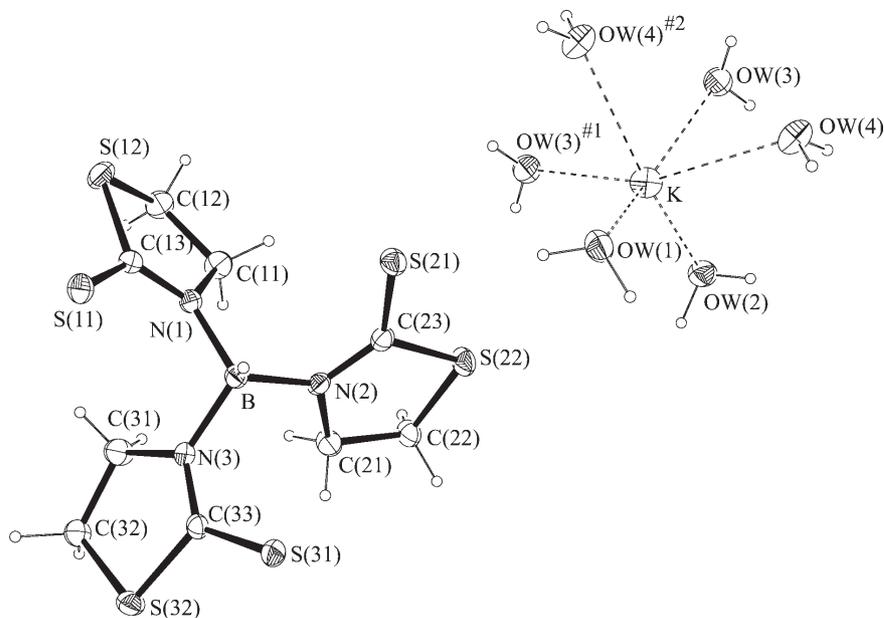


Figure 1. ORTEP3 view of the anion and $\text{K}(\text{H}_2\text{O})_6$ units of compound $\text{KMt.4H}_2\text{O}$ (1) showing the atom-labelling scheme. Displacement ellipsoids drawn at the 50% probability. H atoms are represented by circles of arbitrary size. (Symmetry Codes: #1 $-x+1, -y, -z+1$; #2 $-x+1, -y+1, -z+1$).

indicated by the lack of interaction between its donor atoms and the potassium ion.

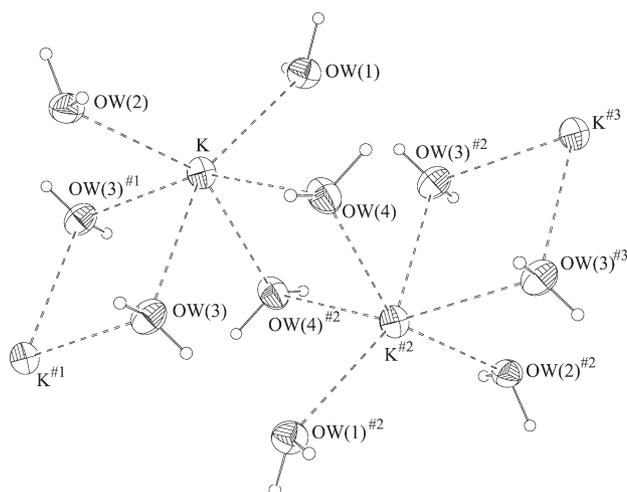


Figure 2. ORTEP3 view of potassium coordination environment in $\text{KMt.4H}_2\text{O}$ (**1**). Displacement ellipsoids drawn at the 50% probability. (Symmetry Codes: $\#1$ $-x+1, -y, -z+1$; $\#2$ $-x+1, -y+1, -z+1$; $\#3$ $x, y+1, z$).

The geometry around the potassium cation can be described as a very distorted octahedron, better as a pentagonal pyramid, as observed before for other hydrotris borates.¹⁴ The largest deviation from the 90° degree octahedral geometry takes place in $\text{OW}(1)\text{-K-OW}(2)$ and $\text{OW}(4)\text{-K-OW}(3)$ angles, with values of $120.8(1)^\circ$ and $71.80(6)^\circ$, respectively, Table 2. The $\text{K-OW}(1)$ bond distance is the shortest one, $2.676(2)\text{\AA}$, and is shorter than the sum of the $\text{K}^+ + \text{O}^{2-}$ ionic radii of 2.78\AA .¹⁵ Compound **1** has a polymeric structure, with an infinite chain consisting of $\text{K}(\text{H}_2\text{O})_n$ units that grows along the b axis, inside an approximate hexagonal cavity that is formed by the packing of the anions, that has an average diameter of $10(1)\text{\AA}$, Figure 3. The equivalent bond distances in the three rings were found to have similar values and all of them agree with the expected values for the bonds involved.

The bond distances and angles found for compound **1**, are similar to the ones recently described in the literature for the TlTbz , $\text{Tbz} = 2\text{-mercaptobenzothiazoline, complex}$.⁴ In this compound the Tl cations are coordinated to the sulfur atoms and there are infinite one-dimensional polymeric chains, that exhibit a zigzag array of Tl and S atoms that are formed by the association of the monomeric units through the thione sulfur atoms.

The structure of compound **2**, described here and shown in Figure 4, exhibits similarities to the X-ray structure previously reported for $\text{NaTm.4.5H}_2\text{O}$,³ such as bond distances and angles, Table 3. Crystallographic parameters and data collection and refinement details are summarized in Table 1.

Table 2. Selected bond distances (\AA) and angles ($^\circ$) for atoms and the potassium ion for $\text{KMt.4H}_2\text{O}$ (**1**) according to X-ray diffraction analysis

C(11)-C(12)	1.521(4)	K-OW(3)	2.784(2)
N(1)-C(13)	1.316(3)	K-S(21)	4.945(2)
N(1)-C(11)	1.484(3)	K-S(22)	4.323(2)
N(2)-C(21)	1.477(4)	K-K ^{#2}	3.859(1)
N(2)-C(23)	1.318(3)	K-K ^{#1}	4.077(1)
N(3)-C(31)	1.475(3)	N(1)-C(13)-S(12)	113.7(2)
N(3)-C(33)	1.313(4)	S(11)-C(13)-S(12)	120.1(2)
S(11)-C(13)	1.697(3)	C(11)-N(1)-B	121.0(2)
S(12)-C(13)	1.750(3)	C(13)-N(1)-B	123.6(2)
S(12)-C(12)	1.813(3)	N-B-N (mean value)	108.6(3)
S(21)-C(23)	1.687(3)	N-B-H (mean value)	110(2)
S(22)-C(22)	1.809(3)	OW(1)-K-OW(3) ^{#1}	111.5(1)
S(22)-C(23)	1.756(3)	OW(1)-K-OW(2)	120.8(1)
S(31)-C(33)	1.699(3)	OW(4)-K-OW(3)	71.80(6)
S(32)-C(32)	1.810(3)	OW(2)-K-OW(3)	75.84(6)
S(32)-C(33)	1.747(3)	OW(3) ^{#1} -K-OW(4) ^{#2}	78.43(6)
B-H	0.96	OW(4) ^{#2} -K-OW(3)	80.88(6)
N-B (mean value)	1.564(4)	OW(3) ^{#1} -K-OW(2)	83.67(6)
K-OW(1)	2.676(2)	OW(3) ^{#1} -K-OW(3)	83.94(6)
K-OW(3) ^{#1}	2.698(2)	OW(1)-K-OW(4) ^{#2}	86.06(7)
K-OW(2)	2.723(2)	OW(1)-K-OW(4)	89.99(6)
K-OW(4) ^{#2}	2.730(2)	OW(4) ^{#2} -K-OW(4)	90.36(6)
K-OW(4)	2.745(2)	OW(2)-K-OW(4)	96.97(6)

Symmetry Codes: $\#1$ $-x+1, -y, -z+1$; $\#2$ $-x+1, -y+1, -z+1$.

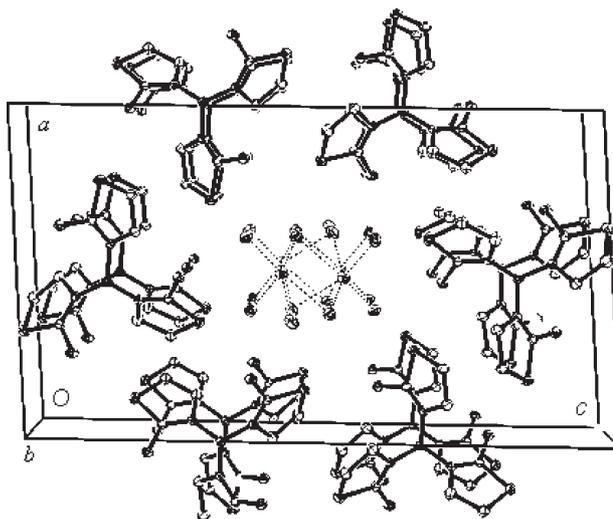


Figure 3. ORTEP3 view of $\text{KMt.4H}_2\text{O}$ (**1**) showing the approximate hexagonal cavity that is formed by the packing of the anions along the b and the infinite chain of $\text{K}(\text{H}_2\text{O})_n$ units.

The counter-ion K^+ of **2** was found to be coordinated to two sulfur atoms of the hydrotris(methimazole)borate unit and to other three sulfur atoms, symmetrically dependent, belonging to neighboring KTm units, which is indicative of the soft nature of the ligand, and resulting in a pentacoordination environment around the cation. A better view of the environment around the potassium cation is shown in Figure 5.

The geometry around K^+ can be described as distorted square pyramidal. The $\text{K-S}(1)$ bond, which is the longest one in the structure, forms the apex of the pyramid. The $\text{K-S}(2)^{\#3}$ is the shortest K-S bond in the structure and

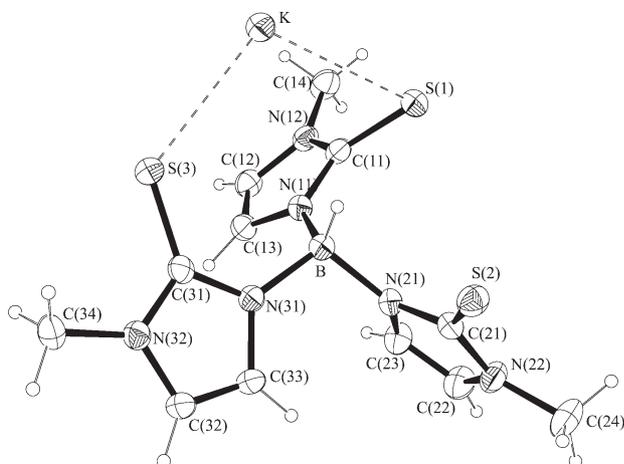


Figure 4. ORTEP3 view of compound **2** showing the atom-labeling scheme. Displacement ellipsoids drawn at the 50% probability. H atoms are represented by circles of arbitrary size.

Table 3. Selected bond distances (Å) and angles (°) for compound **2** according to X-ray diffraction analysis

N(11)-C(11)	1.362(4)	C(13)-N(11)-B	127.3(3)
N(11)-C(13)	1.394(4)	C(11)-N(11)-C(13)	108.3(3)
N(12)-C(11)	1.358(4)	C(11)-N(12)-C(12)	109.5(3)
N(12)-C(12)	1.378(4)	C(11)-N(12)-C(14)	125.7(3)
N(12)-C(14)	1.456(4)	C(12)-C(13)-N(11)	107.8(3)
S(1)-C(11)	1.709(3)	C(12)-N(12)-C(14)	124.7(3)
C(12)-C(13)	1.347(5)	C(13)-C(12)-N(12)	107.4(3)
H-B (mean value)	1.550(5)	N(11)-C(11)-S(1)	127.1(3)
N-H	0.96	N(11)-C(11)-N(12)	107.0(3)
S(3)-K ^{#2}	3.270(1)	N(12)-C(11)-S(1)	126.0(2)
S(2)-K ^{#3}	3.152(1)	K ^{#2} -K-K ^{#3}	132.27(3)
S(1)-K ^{#3}	3.292(1)	K ^{#3} -S(1)-K	74.54(3)
K-S(1)	3.301(1)	K-S(3)-K ^{#2}	92.86(3)
K-S(1) ^{#3}	3.292(1)	S(2) ^{#3} -K-S(1)	110.83(3)
K-S(2) ^{#3}	3.152(1)	S(2) ^{#3} -K-S(1) ^{#3}	97.87(3)
K-S(3)	3.167(1)	S(2) ^{#3} -K-S(3)	140.39(3)
K-S(3) ^{#2}	3.270(1)	S(2) ^{#3} -K-S(3) ^{#2}	80.07(3)
K-K ^{#3}	3.992(2)	S(3) ^{#2} -K-S(1)	99.05(3)
K-K ^{#2}	4.664(2)	S(3) ^{#2} -K-S(1) ^{#3}	154.34(3)
N-B-N (mean value)	109(2)	S(3)-K-S(1) ^{#3}	78.23(3)
H-B-N (mean value)	110.4(3)	S(3)-K-S(3) ^{#2}	87.14(3)
C(11)-N(11)-B	124.3(3)	S(1) ^{#3} -K-S(1)	105.46(3)

Symmetry codes: ^{#1} $x+1, y, z$; ^{#2} $-x, -y, -z$; ^{#3} $-x+1, -y, -z$.

together with the K-S(3) are shorter than the sum of the K^+ + S^{2-} ionic radius of 3.22Å.¹⁵ In this compound there are infinite dimeric chains, along the *a* axes, that are held together by the potassium atoms, Figure 6. The structure of the sodium salt of Tm, NaTm.4.5H₂O, was shown to consist of discrete Tm⁻ anions having a distorted one-dimensional chains of hydrated sodium cations.¹

Conclusions

The potassium salts of the anions hydrotris(2-mercaptothiazolyl)borate, M^{t-}, and hydrotris

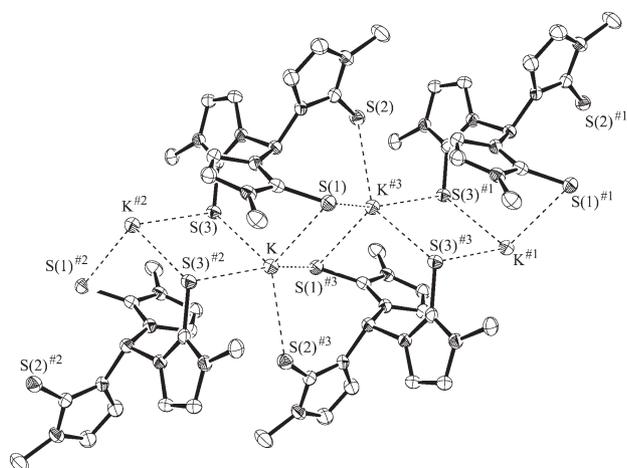


Figure 5. ORTEP3 view of the compound **2** showing in details the potassium coordination environment. Displacement ellipsoids drawn at the 50% probability. (Symmetry Codes: ^{#1} $x+1, y, z$; ^{#2} $-x, -y, -z$; ^{#3} $-x+1, -y, -z$).

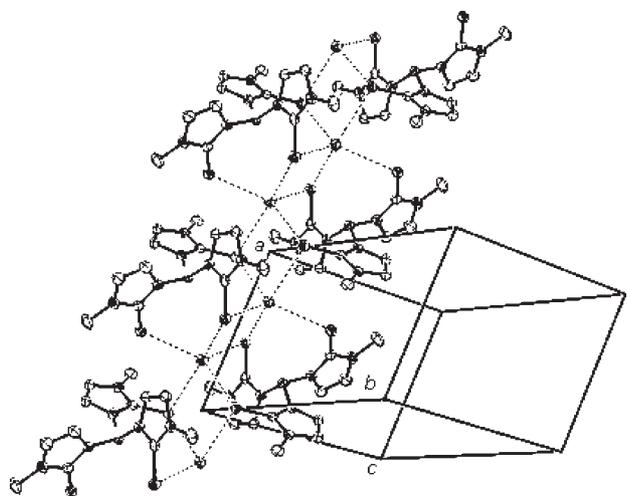


Figure 6. Packing of compound **2** showing the infinite dimeric chains along the *a* axes. Displacement ellipsoids drawn at the 50% probability.

(methimazolyl)borate, Tm⁻, were prepared in better yields than previously described in the literature^{3,4} and were characterized in the solid state and in solution. The solution of their structures confirms the softer nature of the anion in **1**, as compared to **2**, indicated by the lack of interaction of the anion with the cation, in **1**, that is surrounded by six water molecules. The X-ray diffraction analyses also determined that the salts have an extended structure.

Supplementary Material

Supplementary Crystallographic data have been deposit with the Cambridge Crystallographic Data centre

as supplementary publication no. CCDC 181571 (K_{Mt}.4H₂O) and 181572 (K_{Tm}). Copies of available material can be obtained free of charge, on application to the Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (fax: +44123-336-033; e-mail: deposit@ccdc.cam.ac.uk or <http://www.ccdc.ac.uk>).

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Received: December 4, 2003

Published on the web: August 24, 2004

FAPESP helped in meeting the publication costs of this article.