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Hexaamidostannates(IV) of the alkali metals potassium, rubidium and caesium, $M_2[Sn(NH_2)_6]$ (M = K, Rb, Cs), synthesis and crystal structures

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Abstract

Hexaamidostannates(IV) of the alkali metals potassium, rubidium and caesium were prepared by the reaction of tetraphenyl tin with alkali amides in an autoclave made of stainless steel at 385 K with ammonia as solvent. Single crystals were obtained in the form of colourless, transparent hexagonal plates.

Their structures were solved by X-ray methods including all hydrogen positions for $K_2[Sn(NH_2)_6]$ in the space group $R\bar{3}$ with $Z = 3$ (hexagonal setting), and for $Rb_2[Sn(NH_2)_6]$ and $Cs_2[Sn(NH_2)_6]$ in the space group $P\bar{3}$ with $Z = 1$.

$K_2[Sn(NH_2)_6]$ $a = 6.337(4)$ Å, $c = 19.769(5)$ Å, $F_0^2 \geqslant 3\sigma(F_0^2) = 869$, $N(\text{var.}) = 22$, $R/R_w = 0.019/0.022$

$Rb_2[Sn(NH_2)_6]$ $a = 6.669(2)$ Å, $c = 5.771(1)$ Å, $F_0^2 \geqslant 3\sigma(F_0^2) = 245$, $N(\text{var.}) = 23$, $R/R_w = 0.022/0.029$

$Cs_2[Sn(NH_2)_6]$ $a = 6.879(1)$ Å, $c = 5.850(1)$ Å, $F_0^2 \geqslant 3\sigma(F_2^0) = 734$, $N(\text{var.}) = 23$, $R/R_w = 0.018/0.018$

Both structure types contain isolated octahedra units $[Sn(NH_2)_6]^{2-}$. Potassium is coordinated trigonal anti-prismatically by 6 amide ions, rubidium and caesium anti-cuboctahedrally by 12 amide ions. The orientation of the hydrogen atoms is related to the size of the alkali metal ions.

Keywords: Hexaamidostannates(IV) of potassium; Rubidium; Caesium; Synthesis; Crystal structure

1. Introduction

There is only little knowledge about Sn(IV)/N-bonding in the solid state chemistry of amides, imides and nitrides. One of the first investigations concerning this aspect was published by Fitzgerald [1] in 1907. In 1964, Schmitz-DuMont et al. [2] studied the behaviour of organo-Sn(IV)-compounds in liquid ammonia based on the results of Fitzgerald. They treated Ph_4Sn ($Ph = C_6H_5$) with KNH_2 in liquid ammonia at ambient temperature and obtained a white precipitate. The molar ratio of K:Sn:N:H was determined to be 2:1:6:12. They interpreted their results as $K_2[Sn(NH_2)_6]$ and formulated the following equation:

$$Ph_4Sn + 2KNH_2 + 4NH_3 \rightarrow K_2[Sn(NH_2)_6] + 4C_6H_6 \quad (1)$$

The X-ray powder patterns could not be indexed [3].

In 1973 Rouxel et al. [4] synthesized hexaamidostannates(IV) of sodium, potassium, rubidium and caesium and investigated the compounds by X-ray and IR spectroscopical methods. Three years later, Rouxel et al. [5] published structure determinations of $K_2[Sn(NH_2)_6]$ based on powder diffraction data and for $Rb_2[Sn(NH_2)_6]$ determined by X-ray single crystal diffraction data. They reported the space groups $R\bar{3}m$ for $K_2[Sn(NH_2)_6]$ and $P\bar{3}$ for $Rb_2[Sn(NH_2)_6]$. The positions of the hydrogen atoms, however, could not be determined.

In our studies we were able to isolate single crystals of $K_2[Sn(NH_2)_6]$, $Rb_2[Sn(NH_2)_6]$ and $Cs_2[Sn(NH_2)_6]$. The structures were determined by X-ray single-crystal diffraction data including all hydrogen positions (redetermined in the case of $Rb_2[Sn(NH_2)_6]$).

2. Experimental details

Hexaamidostannates(IV) of the alkali metals potassium (J.T. Baker, Chem. Co., Phillipsburg, "purified"), rubidium (Koch-Light, Labs Ltd, Colnbrook, UK, 99.98%) and caesium (E. Merck, Darmstadt, 99.98%) are obtained by reaction of tetraphenyl tin (Alfa

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Table 1
Cell parameters based on powder data (Debye–Scherrer method) and volume of one formular unit

	$K_2[Sn(NH_2)_6]$	$Rb_2[Sn(NH_2)_6]$	$Cs_2[Sn(NH_2)_6]$
Cell parameters (Å)	$a = 6.369(4)$	$a = 6.662(3)$	$a = 6.873(3)$
	$c = 19.757(9)$	$c = 5.770(2)$	$c = 5.829(3)$
Volume of one formular unit (Å^3)	231.4	221.8	238.5

products, Karlsruhe, "crystallized") with alkali amides in liquid ammonia (Messer Griesheim, Duisburg, 99.999%). The required alkali metal amides were synthesized as described by Jacobs and Schmidt [6] and Peters [7]. Most of the compounds show great sensitivity for oxygen and humidity, so all manipulations have to be carried out in a glove-box filled with argon (Messer Griesheim, Krefeld, 99.998%) [6]. The starting materials were filled into an autoclave made of stainless steel (material no. 1.4571) [8] in stoichiometric ratio [2]. The autoclave is filled with liquid ammonia using a Hüttig-Tensieudiometer [9]. The reaction components were treated at 385 K for 7 days and $p(NH_3) \approx 80$ bar.

Debye–Scherrer powder patterns (CuKα) of the products could be indexed completely and showed pure alkali metal hexaamidostannates(IV). The cell parameters are given in Table 1. Colourless and transparent single crystals with the shape of hexagonal plates were obtained in the colder part of the autoclave after 14 days using a temperature gradient of 385 K to 325 K.

Crystals were isolated in sealed glass capillaries. Suitable crystals were tested by precession photographs with MoKα radiation. Intensity data were collected on a four circle diffractometer CAD4 (Enraf-Nonius, Delft, Netherlands).

The structures were determined by direct methods in the case of $K_2[Sn(NH_2)_6]$ and interpretation of Patterson maps in case of $Rb_2[Sn(NH_2)_6]$ and $Cs_2[Sn(NH_2)_6]$. Subsequent calculations were carried out by using the SDP-system of programs [10] and the SHELXTL [11] software package.

Hydrogen positions were determined by Fourier difference maps after the refinement of the anisotropic thermal displacement parameters of the alkali metal, tin and nitrogen atoms. Only one overall *B*-value for crystallographic different hydrogen atoms was refined.

Experimental details and positional and thermal parameters are given in Tables 2–5.

3. Discussion

3.1. $K_2[Sn(NH_2)_6]$

The unit cell of $K_2[Sn(NH_2)_6]$ is depicted in Fig. 1. It shows that $K_2[Sn(NH_2)_6]$ is built of isolated $[Sn(NH_2)_6]^{2-}$ octahedra connected by potassium. The structure is closely related to the CaF_2-structure type with a cubic close packing of the octahedra centres and all tetrahedral voids filled by potassium (Fig. 2). Potassium forms slightly puckered 6^3-nets between the layers of octahedra. The complex anions $[Sn(NH_2)_6]^{2-}$ form nearly regular octahedra with equidistant bonds

Table 2
Data characterizing X-ray structure determination on $K_2[Sn(NH_2)_6]$, $Rb_2[Sn(NH_2)_6]$ and $Cs_2[Sn(NH_2)_6]$

	$K_2[Sn(NH_2)_6]$	$Rb_2[Sn(NH_2)_6]$	$Cs_2[Sn(NH_2)_6]$
Radiation	AgKα	MoKα	AgKα
Monochromator	graphite	graphite	graphite
Crystal dimension (mm)	$0.2 \times 0.2 \times 0.2$	$0.05 \times 0.1 \times 0.1$	$0.1 \times 0.2 \times 0.25$
$1/\mu$ (mm) Transmission	0.532	0.073	0.187
Measuring range (degree)	$3 \leq \theta \leq 30$	$3 \leq \theta \leq 30$	$3 \leq \theta \leq 30$
h, k, l	$0-11, \pm 11, \pm 35$	$0-9, \pm 9, \pm 8$	$\pm 12, \pm 12, -10+7$
R_{int_6}	0.027	0.035	0.018
a (Å)	6.377(4)	6.669(2)	6.879(1)
c (Å)	19.769(5)	5.771(1)	5.850(1)
Volume (Å^3)	696.2	222.3	239.7
ρ_x (g cm^{-3})	2.097	2.882	3.329
Space group	R $\bar{3}$ (no. 148)	P $\bar{3}$ (no. 147)	P $\bar{3}$ (no. 147)
Z	3	1	1
Absorption correction	empirical (Psi-Scan)	empirical (Psi-Scan)	empirical (Psi-Scan)
F_0 (asymm. unit)	923	442	952
$f_0^2 \geq 3\sigma(F_0^2)$	869	245	734
$N_{var.}$	22	23	23
R/R_w ($w = 1$)	0.019/0.022	0.022/0.029	0.018/0.018
Residual electron density (e Å^{-3})	0.47	1.33	2.21
R incl. unobs reflections	0.021	0.070	0.024

Table 3(a)
Atomic coordinates and isotropic displacement parameters of $K_2[Sn(NH_2)_6]$ (standard deviation)

Site	Atom	x	y	z	B (Å^2)
3a	Sn	0	0	0	1.699(2)
6c	K	0	0	0.18066(3)	2.626(7)
18f	N	0.2687(3)	0.2782(3)	0.06595(7)	2.47(3)
18f	H(1)	0.216(5)	0.384(5)	0.074(1)	2.5(5)[a]
18f'	H(2)	0.400(5)	0.362(5)	0.043(1)	2.5(5)[a]

[a] Restricted.

Table 3(b)
Anisotropic thermal displacement parameters U_{ij}($\times10^3$Å^2) of $K_2[Sn(NH_2)_6]$ (standard deviation)

Atom	U_{11}	Y_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Sn	18.83(7)	U_{11}	19.5(1)	$\frac{1}{2}U_{11}$	0	0
K	29.18(6)	U_{11}	57.3(2)	$\frac{1}{2}U_{11}$	0	0
N	26.9(6)	28.3(6)	30.3(7)	13.2(4)	−5.8(5)	−2.7(6)

Table 4(a)
Atomic coordinates and isotropic displacement parameters of $Rb_2[Sn(NH_2)_6]$ (standard deviation)

Site	Atom	x	y	z	B (Å^2)
2d	Rb	1/3	2/3	0.2083(3)	3.05(2)
1a	Sn	0	0	0	1.43(1)
6g	N	0.3052(9)	0.2047(8)	0.213(1)	2.0(1)
6g	H(1)	0.40(1)	0.16(1)	0.23(1)	0(1)[a]
6g	H(2)	0.27(1)	0.20(1)	0.39(1)	0(1)[a]

[a] Restricted.

Table 4(b)
Anisotropic thermal displacement parameters U_{ij} ($\times10^3$Å^2) of $Rb_2[Sn(NH_2)_6]$ (standard deviation)

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Rb	23.6(3)	U_{11}	68.7(9)	$\frac{1}{2}U_{11}$	0	0
Sn	15.8(2)	U_{22}	22.9(4)	$\frac{1}{2}U_{11}$	0	0
N	22(2)	21(2)	34(3)	11(1)	−7(2)	−2(2)

Table 5(a)
Atomic coordinates and isotropic displacement parameters of $Cs_2[Sn(NH_2)_6]$ (standard deviation)

Site	Atom	x	y	z	B (Å^2)
2d	Cs	1/3	2/3	0.27918(6)	2.991(4)
1a	Sn	0	0	0	1.505(3)
6g	N	0.2969(3)	0.1882(3)	0.2119(3)	2.27(3)
6g	H(1)	0.389(5)	0.147(5)	0.176(5)	1.1(4)[a]
6g	H(2)	0.269(5)	0.153(5)	0.349(5)	1.1(4)[a]

[a] Restricted.

Table 5(b)
Anisotropic thermal displacement parameters U_{ij} ($\times10^3$Å^2) of $Cs_2[Sn(NH_2)_6]$ (standard deviation)

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cs	28.19(6)	U_{11}	57.3(2)	$\frac{1}{2}U_{11}$	0	0
Sn	18.83(7)	U_{11}	19.5(1)	$\frac{1}{2}U_{11}$	0	0
N	26.9(6)	28.3(6)	30.3(7)	13.2(4)	−5.8(5)	−2.7(6)

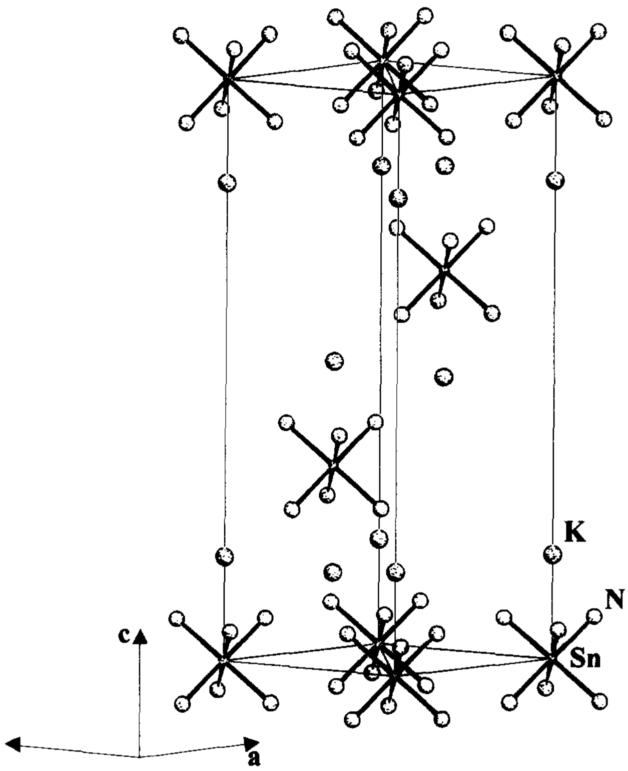

Fig. 1. Unit cell of $K_2[Sn(NH_2)_6]$ without H sites.

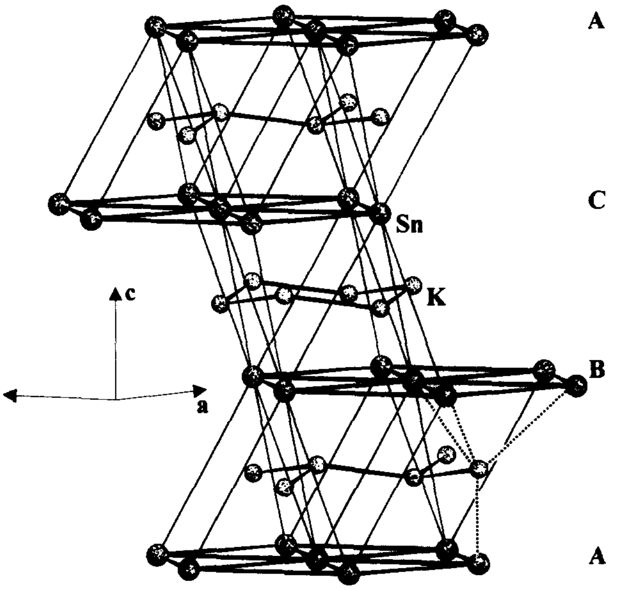

Fig. 2. c.c.p. model of the centre of octahedra $[Sn(NH_2)_6]^{2-}$ (tin atoms) with potassium atoms in tetrahedral voids.

d(Sn–N) = 2.178 Å and bond angles ∠(N–Sn–N) of 87.8° and 92.2° (Fig. 3; Table 6). The amide ion shows nearly equal bond distances d(N–H) and bond angles ∠(H(1)–N–H(2)), ∠(H–N–Sn) near the ideal tetrahedral angle (Table 6). This supports the assumption of sp^3-hybridized nitrogen.

Potassium is located in the tetrahedral voids of the $[Sn(NH_2)_6]^{2-}$ package and is coordinated trigonal

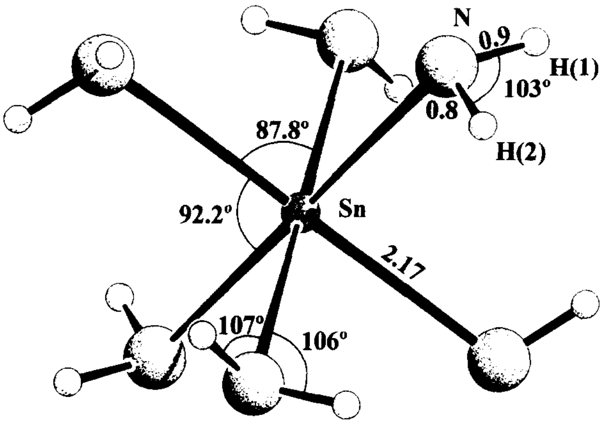

Fig. 3. Octahedron $[Sn(NH_2)_6]^{2-}$ in $K_2[Sn(NH_2)_6]$ with distances in Å and angles in degrees.

Table 6
Some selected distances in Å and angles in degrees with standard deviations in parentheses for $K_2[Sn(NH_2)_6]$, $Rb_2[Sn(NH_2)_6]$ and $Cs_2[Sn(NH_2)_6]$ (M = alkali metal)

	$K_2[Sn(NH_2)_6]$	$Rb_2[Sn(NH_2)_6]$	$Cs_2[Sn(NH_2)_6]$
Tin			
Distances			
Sn–N	6*2.178(1)	6*2.177(4)	6*2.178(1)
Sn–H	⩾2.57(1)	⩾2.55(1)	⩾2.67(1)
Sn–M	⩾3.574(6)	⩾4.034(4)	⩾4.294(1)
Angles			
N–Sn–N	3*180	3*180	3*180
N–Sn–N	6*92.20(5)	6*88.7(1)	6*89.22(6)
N–Sn–N	6*87.80(5)	6*91.2(1)	6*90.87(6)
Nitrogen			
Distances			
N–H(1)	0.90(1)	0.793(8)	0.842(7)
N–H(2)	0.86(2)	1.05(1)	0.83(1)
N–N	2*3.138(1)	2*3.048(1)	2*3.059(2)
	2*3.020(1)	2*3.111(6)	2*3.100(1)
	2*3.020	2*3.111(6)	2*3.100(1)
N–N	⩾3.893(1)	⩾3.767(7)	⩾3.817(2)
Angles			
H(1)–N–H(2)	103(1)	95(1)	104.1(9)
H(1)–N–Sn	106(1)	120.6(9)	107.3(7)
H(2)–N–Sn	107(2)	113.0(5)	111.4(6)
Alkali metal			
Distances			
M–N	3*2.862(1)	3*2.991(4)	3*3.198(1)
	3*2.934(1)	3*3.224(5)	3*3.631(1)
		3*3.953(5)	3*3.714(1)
		3*3.686(4)	3*3.740(1)
M–H	⩾2.99(2)	⩾2.98(1)	⩾3.15(2)
Angles			
Sn–N–M	89.27(4)	101.4(1)	104.44(4)
	136.78(5)	94.6(1)	91.87(4)
		152.9(2)	158.51(6)
		82.6(1)	88.99(5)
N–M–N	3*63.70(3)		
	3*89.26(4)		

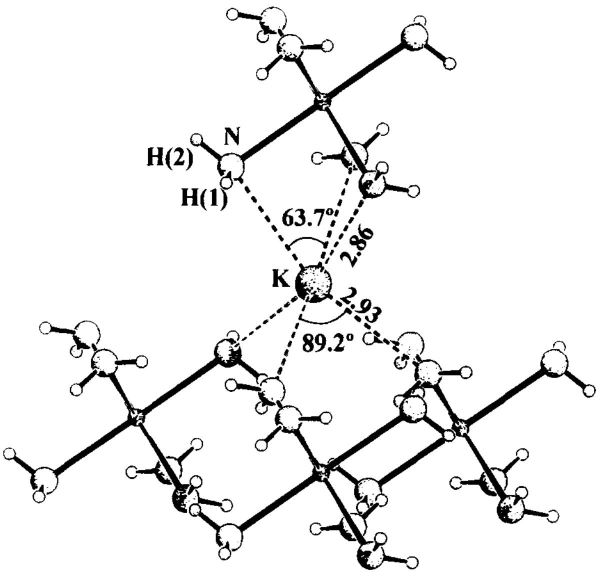

Fig. 4. Anti-prismatical coordination of potassium by NH_2 in $K_2[Sn(NH_2)_6]$ with distances in Å and angles in degrees.

anti-prismatically by amide ions of the complex anions (Fig. 4). One triangular face of the prism results from amide ions of one anion $[Sn(NH_2)_6]^{2-}$. The other consists of amide ions of three different complex anions. The distances d(K–N) and the angles ∠(N–K–N) towards the plane belonging to one complex anion are smaller than those towards the other (Table 6). The orientation of hydrogen belonging to the amide ions of the coordination polyhedron is of further interest. No hydrogen is located either nearby or in the coordination polyhedron, nor even orientated towards potassium.

The coordination of an amide ion shows one tin and two potassium atoms in the first coordination sphere (Fig. 5). The positions of K suggest the coordination of the free electron pair provided nitrogen is sp^3-hybridized. This would explain the slight distortion of the angle ∠(N–Sn–N) = 87.8° in the octahedron of $[Sn(NH_2)_6]^{2-}$ and reflects bonding interactions between potassium and the three amide ions of the octahedron forming one triangular plane in the coordination sphere of K^+ (Fig. 4).

3.2. $Rb_2[Sn(NH_2)_6]$ and $Cs_2[Sn(NH_2)_6]$

The structures of $Rb_2[Sn(NH_2)_6]$ and $Cs_2[Sn(NH_2)_6]$ are isotypic. They can be described as a variant of the $BaNiO_3$-structure type (hex. perovskite) with alkali metal cations and amide ions in a common distorted hexagonal close packing (Fig. 6). In contrast to $BaNiO_3$ the hexaamidostannates(IV) of rubidium and caesium contain no strings of occupied face-sharing octahedra. According to the composition, half of the Ni-positions of the hexagonal perovskite

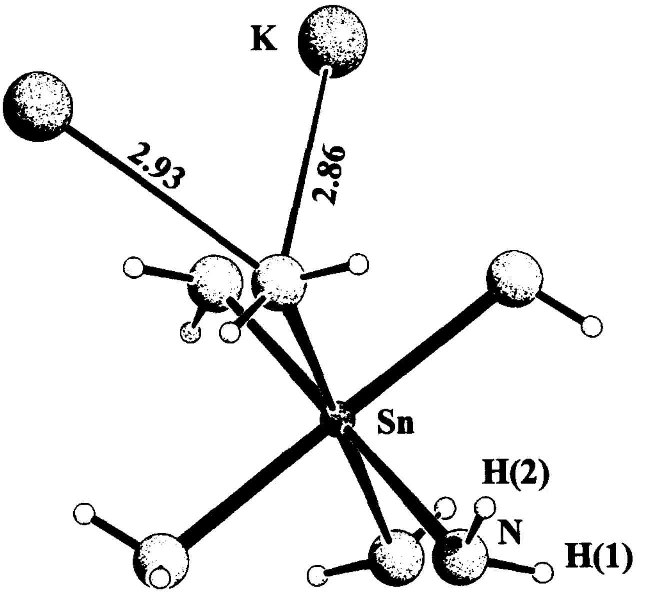

Fig. 5. Coordination of amide ions in $K_2[Sn(NH_2)_6]$ with distances in Å.

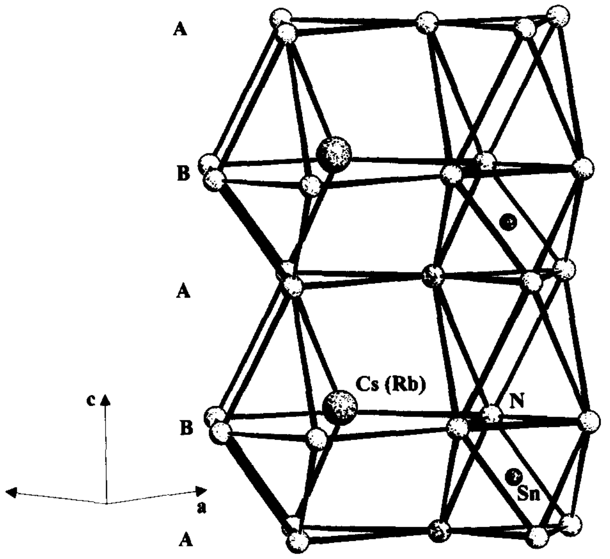

Fig. 6. Arrangement of atoms in $M_2[Sn(NH_2)_6]$ (M = Rb, Cs).

are not occupied. Therefore isolated octahedra $[Sn(NH_2)_6]^{2-}$ occur, as is known from $K_2[Sn(NH_2)_6]$ (Fig. 7).

In Fig. 8 the octahedral coordination of tin by amide ions is depicted. The complex anions $[Sn(NH_2)_6]^{2-}$ show equal bond lengths d(Sn–N) in both compounds (Table 6). The angle ∠(N–Sn–N) is slightly distorted compared with an ideal octahedron. This distortion can be described as a compression along the z-axis.

The amide ions in both compounds differ slightly in bond distances d(N–H) and bond angles ∠(H(1)–N–H(2)) and ∠(H–N–Sn), although they have comparable orientations (Fig. 8; Table 6). The angles ∠(H(1)–N–H(2)) and ∠(H–N–Sn) are nearly ideal tetrahedral.

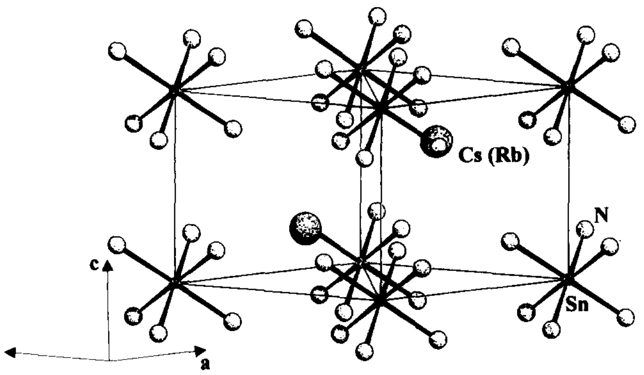

Fig. 7. Unit cell of $M_2[Sn(NH_2)_6]$ (M = Rb, Cs) without H sites.

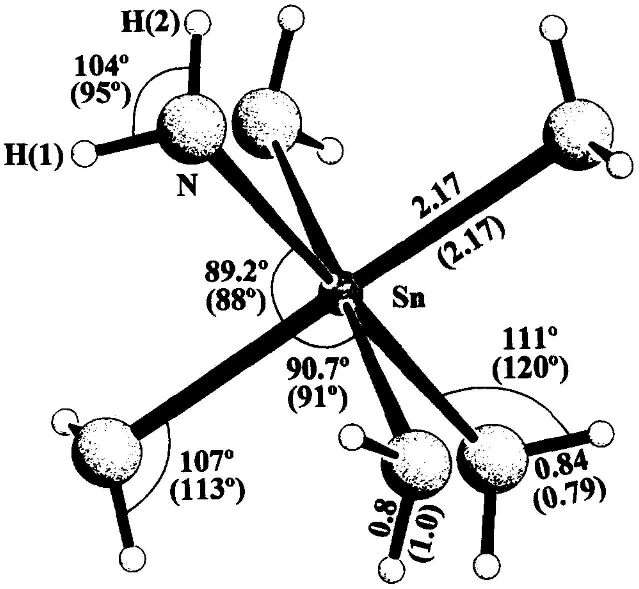

Fig. 8. Octahedron $[Sn(NH_2)_6]^{2-}$ in $M_2[Sn(NH_2)_6]$ (M = Rb, Cs) with distances in Å, angles in degrees and values for the Rb compound in parentheses.

Therefore a sp^3-hybridization of nitrogen can be assumed again.

The alkali metals are coordinated anti-cuboctahedrally by amide ions. Fig. 9 shows this coordination including hydrogen positions. Each anti-cuboctahedron shares faces with three different complex anions, and three amide ions are from three different octahedra of $[Sn(NH_2)_6]^{2-}$ units. In both compounds the alkali metal is located on a 3-fold axis and has two different distances d(M–N) in the equatorial plane, one to the top and one to the bottom of the coordination polyhedron. It is obvious that the positions of rubidium and caesium are not the same. Caesium is positioned above and rubidium nearly in the equatorial plane of the anti-cuboctahedron. A closer inspection of H positions shows that some hydrogen atoms are slightly oriented into the coordination sphere. A comparison of hydrogen orientation at the bottom of the anti-cuboctahedra in Figs. 9(a) and (b) illustrates

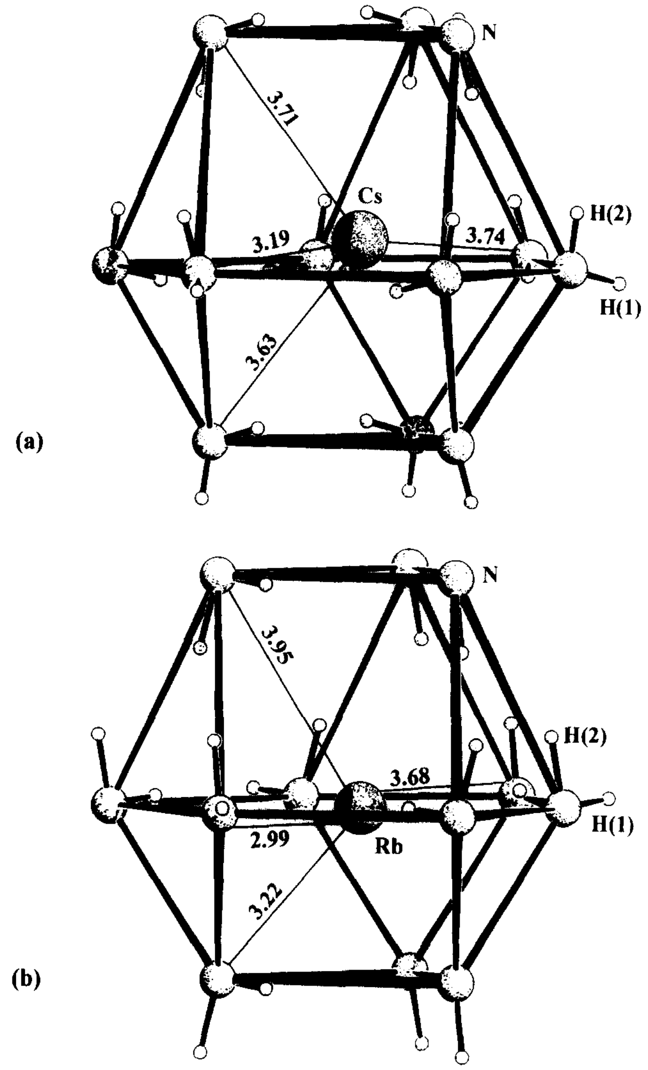

Fig. 9. Anti-cuboctahedral coordination of caesium in $Cs_2[Sn(NH_2)_6]$ (a) and of rubidium in $Rb_2[Sn(NH_2)_6]$ (b), with distances in Å.

the relationship between the hydrogen atom positions and those of alkali metals. In the caesium compound the atom H(1) is slightly oriented into the coordination polyhedron. In contrast, the corresponding H(1) atom of $Rb_2[Sn(NH_2)_6]$ does not point into it. This suggests that H(1) avoids rubidium with respect to an electrostatic point of view and this leads to a slightly different orientation of the amide ion. In both compounds every second H(1) atom of the equatorial plane is slightly orientated into the anti-cuboctahedron, too. The other hydrogen atoms are located on faces or point into other unoccupied polyhedra.

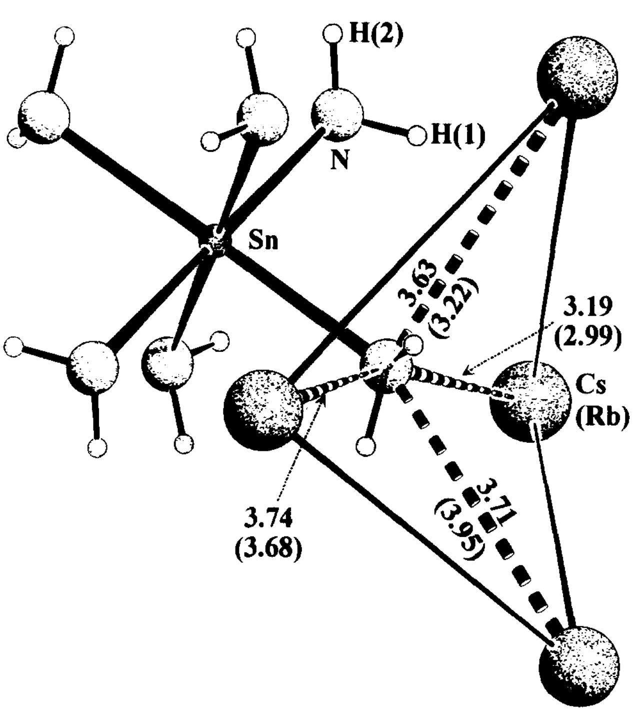

Fig. 10. Coordination of amide anions in $M_2[Sn(NH_2)_6]$ (M = Rb, Cs) with distances in Å and values for the Rb compound in parentheses.

The coordination sphere, which has not been discussed yet, is the coordination of amide ions (Fig. 10). They are coordinated by one tin and four alkali metal ions. The polyhedron can be described as an extremely distorted tetragonal pyramid with an amide ion at the apex. This coordination sphere includes all four distances *d*(M–N) discussed above. It is conspicuous, though the *d*(M–N) differ between the two compounds, that the shortest distances are compatible with the orientation of a free electron pair of N, provided by an sp^3-hybridized nitrogen atom.

4. Results

The three hexaamidostannates(IV) of potassium, rubidium and caesium crystallize in two different structure types. $Rb_2[Sn(NH_2)_6]$ is almost isotypic to $Cs_2[Sn(NH_2)_6]$. Both types show isolated complex anions $[Sn(NH_2)_6]^{2-}$ in the form of almost ideal octahedra. The angles and distances found at the amide ion itself and the coordination of amide ions by tin and alkali metal ions suggest an sp^3-hybridization of nitrogen, although the hydrogen positions in $K_2[Sn(NH_2)_6]$, $Rb_2[Sn(NH_2)_6]$ and $Cs_2[Sn(NH_2)_6]$ are quiet different. The various hydrogen orientations can be explained by the position and size of the alkali metal ions. The two different structure types illustrate the change of alkali metal coordination by amide ions from coordination number 6 for $K_2[Sn(NH_2)_6]$ to coordination number 12 for $Rb_2[Sn(NH_2)_6]$ and $Cs_2[Sn(NH_2)_6]$.

This change effects the molecular volume of the compounds (Table 1, volume of one formular unit). A comparison shows a minimum for $Rb_2[Sn(NH_2)_6]$.

This may be related to the different orientations of hydrogen atoms and a maximum of thermodynamic stability for this compound. This may also explain why $Rb_2[Sn(NH_2)_6]$ crystallizes significantly better than the other two compounds.

Acknowledgements

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