The Layered Coordination Polymer Bi(SCN)3*½H2O

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Yellow crystals of $Bi(SCN)_3 \cdot ½H_2O$ are obtained by reacting $(BiO)_2CO_3$ and HSCN in aqueous solution. X-ray diffraction on a single-crystal revealed a triclinic lattice (space group $P\bar{1}$) with a = 843.6(2) pm, b = 920.4 (2) pm, c = 1210.7(2) pm, α = 109.16(3) °, β = 109.06(3) °, γ = 90.22(3) °, $V = 832.8(3) \cdot 10^6$ pm^3, and Z = 4. All thiocyanate anions bind with both ends to different cations. The coordination network expands in plane layers parallel (1 $\bar{1}$ 0). The water molecule coordinates one of the two independent Bi^{3+} cations. Dehydration sets in at 100 °C, followed by stepwise thermal decomposition to Bi_2S_3.

Introduction

The thiocyanate anion belongs to the so-called pseudo-halogenides. Salts with such monovalent bi- or triatomic anions show extensive chemical similarity to the corresponding halogenides. In the context of metal-rich compounds, however, the number of corresponding low-valent pseudo-halogenides is much smaller than would be expected. Most examples are found with thiocyanate or azide as terminal ligands of Nb_6Cl_{12} cluster cores [1]. In analogy to the chemistry of bismuth-rich halogenides [2], the synproportionation of Bi with $Bi(SCN)_3$ could be a potential synthetic approach to low-valent thiocyanates. Surprisingly, reliable information on $Bi(SCN)_3$, including its crystal structure, is not available.

The existence of bismuth thiocyanate has already been suggested in the 19th century by *Bender* [3]. In 1906, *Rosenheim* and *Vogelsang* mentioned a hydrate with the (unlikely) composition $Bi(SCN)_3 \cdot 14H_2O$ [4]. Later the structures of complex salts like $RbBi(SCN)_4$ [5] or $Cs_2Na[Bi(SCN)_6]$ [6] have been reported. In accordance to the HSAB principle the bismuth cation is coordinated by the "soft" sulfur atom, while the "hard" nitrogen atom binds preferably to the alkaline metal cation. The first structural information on $Bi(SCN)_3$, which was synthesized by the method of *Gallais* and *Familiades* [7], was provided by *Bensch* et al., who proposed a triclinic cell (space group $P\bar{1}$, a = 662.9(3) pm, b = 1178.4(6) pm, c = 1200.8(5) pm, α = 114.18(4) °, β = 102.33(5) °, γ = 98.59(5) °, $V = 805.9 \cdot 10^6$ pm^3) [8].

In view of this, we attempted to grow crystals of bismuth thiocyanate following diverse synthetic strategies. What we were able to characterize by single-crystal diffraction turned out to be the semi-hydrate $Bi(SCN)_3 \cdot ½H_2O$.

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Experimental Section

Synthesis: $Bi(SCN)_3 \cdot ½H_2O$ was synthesized from $(BiO)_2CO_3$ and HSCN according to [7]:

$$(BiO)_2CO_3 + 6HSCN \rightarrow 2Bi(SCN)_3 \cdot ½H_2O + CO_2 + 2H_2O.$$

In a three-necked flask 40 g $(NH_4)_2SO_4$ (Grüssing, 99.5 %) were dissolved in 10 mL H_2O. Two dropping funnels with (a) 75 g NH_4SCN (Grüssing, 99 %) dissolved in 300 mL H_2O and (b) 120 mL of an aqueous solution of 100 g H_2SO_4 (Biesterfeld, 95 – 97 %) were mounted. 30 g H_2SO_4 (95 – 97 %) were added to the $(NH_4)_2SO_4$ and the flask was heated to 60 °C. Simultaneously, both solutions were added dropwise. The freshly produced HSCN was distilled at reduced pressure (20 – 50 Pa) and condensed into an ice-cooled receiver. Addition of $(BiO)_2CO_3$ (Schering) to the distillate resulted in the evolution of gas and the solution turned orange. After 1 h of stirring, the solution was separated from the excess of $(BiO)_2CO_3$.
Fractions of the orange solution were used for probing several crystallization methods. Evaporation at ambient conditions yielded yellow crystals, the use of a rotary evaporator resulted in yellow powder. Yellow crystals and orange powder were obtained by extraction with MeCN, DMF, THF, Et_2O, or ethyl acetate and subsequent evaporation of the solvent. Tests with DMSO, toluene, or hexane were not successful.
Alternatively, the metathesis reaction of bismuth halogenides with thiocyanates of the alkaline metals [9] in the eutectic mixture n(NaSCN) : n(KSCN) = 7 : 3 (melting point 122 °C) [10], following the reaction

$$BiX_3 + 3MSCN \rightarrow Bi(SCN)_3 + 3MX,$$

as well as the corresponding Finkelstein reaction in dry acetone were tested. In both cases, the precipitation of *MX* was observed, but suitable crystals of bismuth thiocyanate were not obtained.

Vibrational Spectroscopy: The infrared spectrum of a pressed pellet of $Bi(SCN)_3 \cdot ½H_2O$ with KBr was recorded with a Nicolet Magna-IR 550 Series II spectrometer. The spectrum of a pure KBr sample was subtracted. The Raman spectrum of a $Bi(SCN)_3 \cdot ½H_2O$ crystal was measured using a Raman microscope Renishaw RM 2000 equipped with a CCD detector and an 800 L/mm lattice

monochromator. The wavelength of the laser was 632.9 nm, the power of the beam on the sample was 0.3 mW.

Thermal Analysis: $Bi(SCN)_3 \cdot ½H_2O$ was heated to 700 °C (rate 5 K/min) in an open Al_2O_3 crucible inside a thermobalance (Netzsch STA 409 PC Luxx). The atmosphere was a stream (100 mL/min) of Ar (99.999 %).

X-ray Crystallography: Intensity data of a single-crystal (yellow flat needle) were collected at 293(2) K on an imaging plate diffraction system IPDS-II (Stoe) using graphite-monochromated Mo-$K\alpha$ radiation. The raw-data were corrected for background, polarization and Lorentz factor. The microscopic description of the crystal shape, which was later used in the numerical absorption corrections (μ(Mo-$K\alpha$) = 21.9 mm^{-1}; max./min. transmission: 0.864/0.161) [11], was optimized using sets of reflections that are equivalent in the triclinic Laue class [12]. The structure was solved with direct methods [13] and subsequent difference Fourier syntheses in the space group $P\bar{1}$ (no. 2). Even the hydrogen atoms could be localized, although with high standard deviations of their parameters. For the hydrogen atoms a common isotropic displacement parameter could be refined, while anisotropic displacement parameters were determined for the other atoms. Atomic parameters are listed in Table 1, selected interatomic distances in Table 2. Data in brief: 15033 reflections measured, 5406 unique; $-12 \le h \le 12$, $-13 \le k \le 13$, $-17 \le l \le 17$, $2\theta_{max} = 64°$; $R_{int} = 0.043$, $R_\sigma = 0.076$; extinction parameter: $145(6) \cdot 10^{-5}$; 198 parameters; $R_1 = 0.033$ for 3444 $F_o > 4\sigma(F_o)$, $R_1 = 0.066$ for all F_o, $wR_2 = 0.045$; residual electron density 1.5 to –1.9 e/(10^6 pm^3). The experimental powder diffractogram, which was measured with a Stadi-P powder diffractometer (Stoe) using Cu-$K\alpha_1$ radiation, nicely matches the calculated pattern. Further data, in the form of a CIF, have been deposited with the Fachinformationszentrum Karlsruhe, D-76344 Eggenstein-Leopoldshafen, Germany (E-mail address: crysdata@fiz-karlsruhe.de), as supplementary material no. CSD-421612, and can be obtained by contacting the FIZ quoting the article details and the CSD number. Graphic representations of the crystal structure were developed with the program Diamond [14].

Table 1. Coordinates and equivalent isotropic displacement parameters of the atoms in $Bi(SCN)_3 \cdot ½H_2O$ at 293(2) K. U_{eq} (in pm^2) is defined as one third of the trace of the orthogonalized tensor U_{ij}.

Atom	*x*	*y*	*z*	U_{eq}/U_{iso}
Bi1	0.30158(3)	0.26911(2)	0.07828(2)	281.7(7)
Bi2	0.23536(3)	0.19707(2)	0.44276(2)	309.4(7)
S1	–0.0248(2)	0.2613(2)	–0.0659(2)	394(4)
S2	0.3864(2)	0.0909(2)	–0.1091(1)	388(3)
S3	0.6741(2)	0.3516(2)	0.1469(2)	385(4)
S4	0.5234(2)	0.2272(2)	0.3392(1)	315(3)
S5	0.4844(2)	0.1659(2)	0.6417(2)	429(4)
S6	0.0470(3)	0.3175(2)	0.5810(2)	498(5)
N1	–0.1971(7)	–0.0102(6)	–0.0843(6)	434(14)
N2	0.0704(8)	–0.0653(7)	–0.2804(5)	517(15)
N3	0.6991(8)	0.5413(7)	0.0139(6)	468(15)
N4	0.6647(7)	0.5370(6)	0.4637(5)	378(12)
N5	0.7020(9)	0.4406(7)	0.7361(6)	544(17)
N6	–0.1674(8)	0.0551(6)	0.5349(6)	447(14)
C1	–0.1220(8)	0.0992(7)	–0.0729(6)	320(14)
C2	0.1978(9)	0.0001(7)	–0.2081(6)	346(14)
C3	0.6862(8)	0.4642(7)	0.0669(6)	305(13)
C4	0.6080(8)	0.4092(7)	0.4133(5)	269(12)
C5	0.6118(9)	0.3276(7)	0.6958(6)	383(15)
C6	–0.0806(9)	0.1628(7)	0.5526(6)	359(15)
O	0.1044(7)	0.3237(6)	0.2731(5)	484(13)
H1	0.111(9)	0.416(8)	0.249(6)	470(150)
H2	0.005(11)	0.306(9)	0.235(7)	U_{iso}(H1)

Table 2. Selected interatomic distances (in pm) for $Bi(SCN)_3 \cdot ½H_2O$. Symmetry operators: (i): *x*, *y*, *z*; (ii): 1–*x*, 1–*y*, –*z*; (iii): –*x*, –*y*, –*z*; (iv): 1–*x*, 1–*y*, 1–*z*; (v): –*x*, –*y*, 1–*z*.

Bi1–N3^{ii}	235.9(5)	Bi2–N4^{iv}	235.1(5)
N1^{iii}	256.9(5)	N6^{v}	250.8(5)
S2^{i}	264.1(2)	O^{i}	262.6(5)
S1^{i}	272.2(2)	S6^{i}	266.2(2)
N5^{iv}	288.0(7)	N2^{iii}	266.3(6)
S3^{i}	300.1(2)	S5^{i}	273.0(2)
S4^{i}	325.2(2)	S4^{i}	312.4(2)
S–C	164.0(7) – 166.3(6)	N–C	112.8(7) – 114.8(8)

Results and Discussion

Yellow transparent crystals of bismuth trithiocyanate semi-hydrate, $Bi(SCN)_3 \cdot ½H_2O$, were crystallized following the route of *Gallais* and *Familiades* [7]. Single-crystal X-ray diffraction revealed a triclinic lattice (space group $P\bar{1}$) with a = 843.6(2) pm, b = 920.4 (2) pm, c = 1210.7(2) pm, α = 109.16(3) °, β = 109.06(3) °, γ = 90.22(3) °, V = $832.8(3) \cdot 10^6$ pm^3, and Z = 4. The powder pattern of the yellow crystals, which matches the results of the single-crystal structure determination, deviates significantly from the pattern of the microcrystalline orange by-product, which was described by *Bensch* et al. [8]. The unit cell volumes of the probably water-free orange powder and the yellow semihydrate differ by $26.9 \cdot 10^6$ pm^3, which corresponds to 8.1 $cm^3/mol(H_2O)$. *Biltz* has listed volume increments between 8 and 11 $cm^3/mol(H_2O)$ [15].

The crystal structure of $Bi(SCN)_3 \cdot ½H_2O$ consists of two-dimensional coordination networks that form plane layers parallel ($1\bar{1}0$). The full width of one layer is 623 pm, which includes an estimated van der Waals gap of about 237 pm. Two-dimensional coordination networks were also found for $M(SCN)_2$ with M = Zn [16], Sn [17], and Hg [18], while $M(SCN)_2$ with M = Eu, Sr, Ba [9], Ni [19], Cd [20], and Pb [21] prefer three-dimensional connectivity.

The thiocyanate anions and the water molecules constitute the surface of the layer, while the Bi^{3+} cations are situated inside (Figure 1). The axes of thiocyanate anions are inclined to the plane with the N atoms pointing to the interior. All thiocyanate anions coordinate with both ends to different cations. Except for S4, which is μ_2-bridging, all S and N atoms are non-bridging. The bond angle in the thiocyanate anions differs by 1.2(7) ° to 4.7(6) ° from 180 °. The S–C bond lengths range from 164.0(7) to 166.3(6) pm, the N–C bond lengths from 112.8(7) to 114.8(8) pm. Considering the given accuracy, these distances and angles agree with literature data [9].

Figure 1. Layered coordination network parallel (1 $\bar{1}$ 0) in the crystal structure of $Bi(SCN)_3 \cdot ½H_2O$. The S atoms of the thiocyanate anions and the water molecules constitute the layer surface.

For the orientation of the water molecule, the bonding of the O atom to the Bi2 cation seems to be the determining factor; the secondary bond to Bi1 is almost 60 pm longer (Figure 2). The hydrogen atoms, which were (approximately) localized by X-ray diffraction, are situated on the reverse site of the donating lone-pairs of the oxygen atom and point in the opposite direction of the Bi2–O bond. Thus the potential hydrogen bridge O–H1···N5, which is strongly bend (O···N5 276 pm, H1···N5 ~200 pm, angle at H1 < 135°), is most likely not essential for the structure.

Figure 2. Coordination of the Bi^{3+} cations in $Bi(SCN)_3 \cdot ½H_2O$. Ellipsoids indicate 70 % probability. Distances are given in pm.

The coordination number of both symmetry independent Bi^{3+} cations is seven. Bi1 is coordinated by four S and three N atoms. Bi2 binds to three S, and three N, and one O atom. Applying the bond-length bond-strength concept [22] in the version of *Brese* and *O'Keeffe* [23] and bond valence parameters R_{BiS} = 255 pm, R_{BiN} = 224 pm, and R_{BiO} = 209 pm, results in bond valence sums of 3.17(3) for Bi1 and 3.34(3) for Bi2. Even in view of the error limits of the concept, the formula, and the parameters, the calculated valence sum of Bi2 is too large (the given standard deviation is based only on the standard deviations of the distances). It can be argued that the dative bond of the O atom, which contributes about 0.23 to the sum, is not properly handled. In particular, the H atoms do not bind to any other atom and thus, in strict application of the principle, the O atom of the water molecule should have no remaining valence.

Figure 3. IR (top) and Raman spectra (below) of $Bi(SCN)_3 \cdot ½H_2O$.

The vibrational spectra of $Bi(SCN)_3 \cdot ½H_2O$ (Figure 3) can be interpreted in analogy to the detailed analysis of the $Ba(SCN)_2$ spectra by *Wickleder* [9]. The low-frequency Raman bands below 250 cm^{-1} are attributed to modes that include Bi atoms. The bands at higher frequencies in both spectra can be assigned to internal vibrations of the thiocyanate anion: δ(SCN) between 432 cm^{-1} and 460 cm^{-1}; ν(CS) between 719 cm^{-1} and 787 cm^{-1}; 2δ(SCN) between 889 cm^{-1} and 997 cm^{-1}; ν(CN) between 2003 cm^{-1} and 2144 cm^{-1}. The IR absorption at 1620 cm^{-1} originates from the bending mode of the H_2O molecule [24].

Figure 4. Thermogravimetry of $Bi(SCN)_3 \cdot ½H_2O$ in a flow of Ar.

In contrast to the water-free thiocyanates $M(SCN)_2$ with *M* = Eu, Sr, Ba [9], the semi-hydrate $Bi(SCN)_3 \cdot ½H_2O$ does not melt congruently, but decomposes in several steps in the course of heating. In a dynamic thermogravimetric heating experiment (Figure 4) the sample lost 1.9 wt.-% above 70 °C, which is assigned to adherent moisture. The following weight loss of again 1.9 wt.-% is probably caused by the coordination water (calc. 2.3 wt.-%). The product of the dehydration step, which is completed at 190 °C, proved to be amorphous in the X-ray diffraction experiment. Above 260 °C, the thiocyanate anions decompose successively into sulfide anions and gas phase species, probably CS_2, $(CN)_2$, and N_2 [25]. For the decay of one of the six crystallographically distinct thiocyanate anions a weight loss of 5.35 wt.-% is anticipated. Starting at 260 °C, 310 °C, and 610 °C, one, another three, and the last two groups undergo decomposition. The product after heating to 700 °C and cooling to room temperature is Bi_2S_3, which was identified by powder diffraction. The integral weight loss matches quite nicely.

Conclusions

$Bi(SCN)_3 \cdot ½H_2O$ has been characterized as one product of the aqueous synthesis of bismuth thiocyanate. Thermal dehydration is possible, but degrades the crystals to amorphous powder. Nevertheless this material can be used for further synthesis, although not in reactions above 260 °C. Mild solvothermal methods or the use of ionic liquids [26] are recommended.

Acknowledgement

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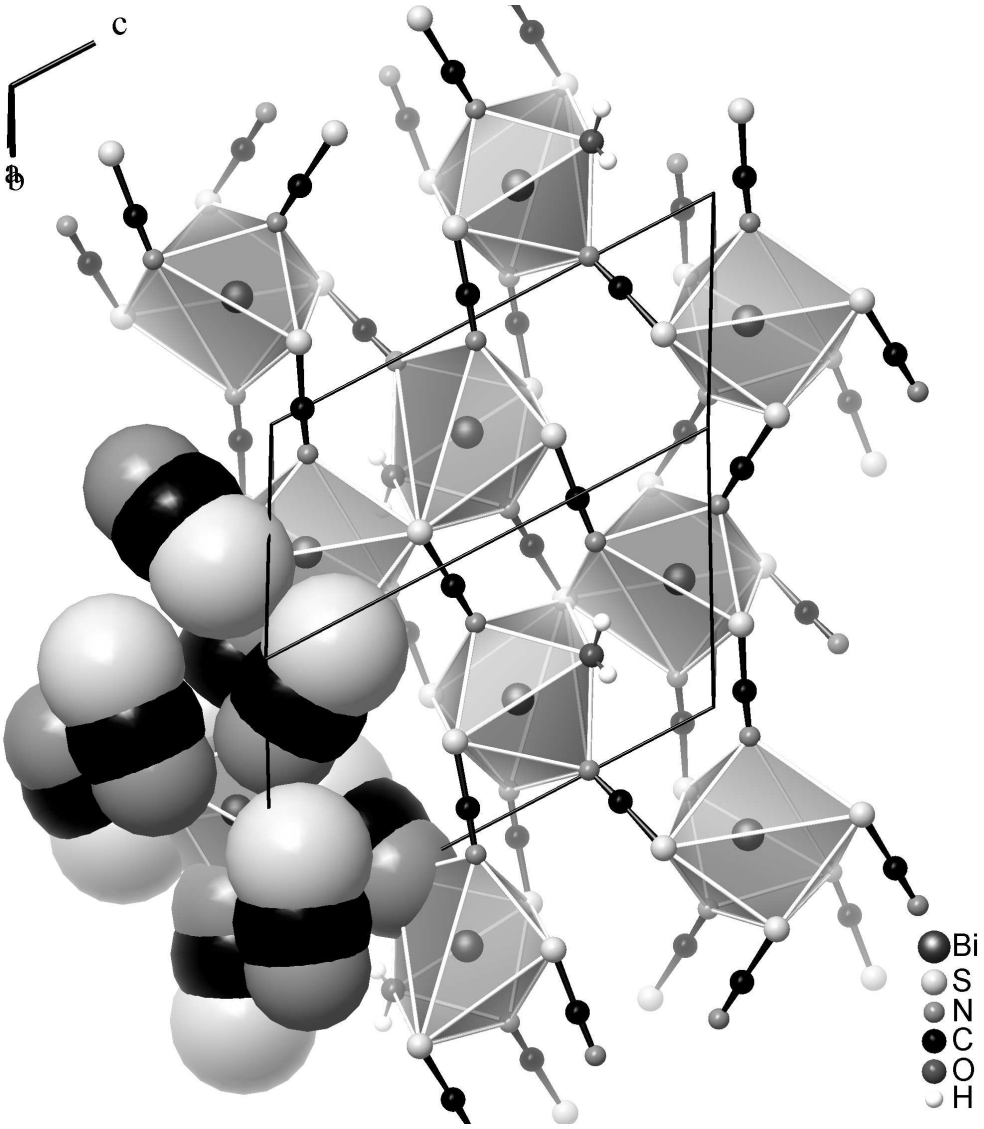

Layered coordination network parallel (1 -1 0) in the crystal structure of $Bi(SCN)_3 \cdot \frac{1}{2}H_2O$. The S atoms of the thiocyanate anions and the water molecules constitute the layer surface.
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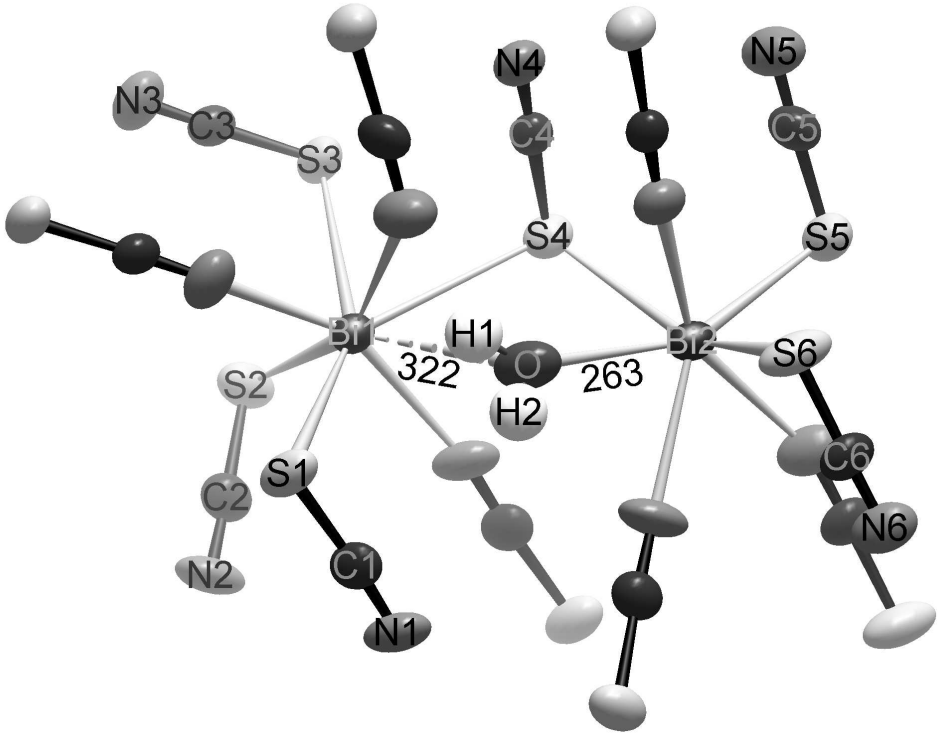

Coordination of the Bi^{3+} cations in $Bi(SCN)_3 \cdot ½H_2O$. Ellipsoids indicate 70 % probability. Distances are given in pm.
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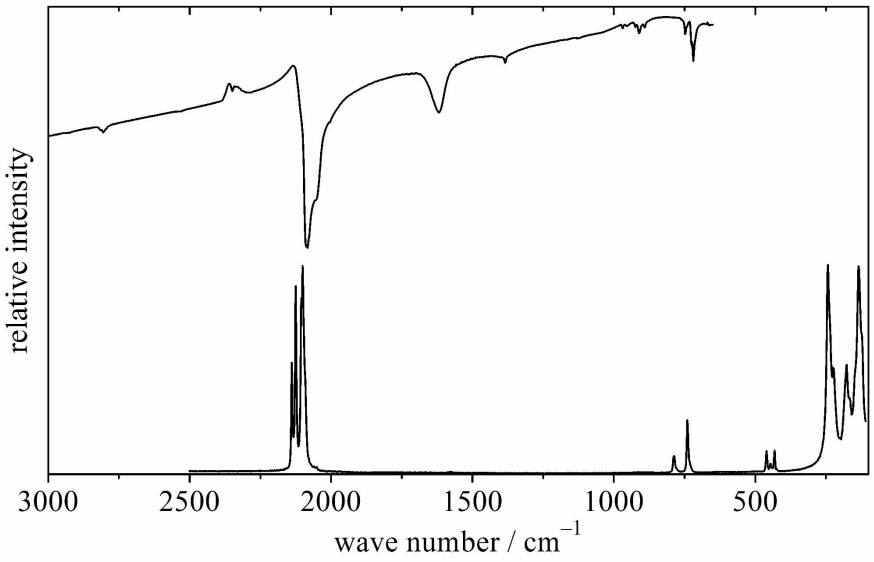

IR (top) and Raman spectra (below) of $Bi(SCN)_3 \cdot ½H_2O$.
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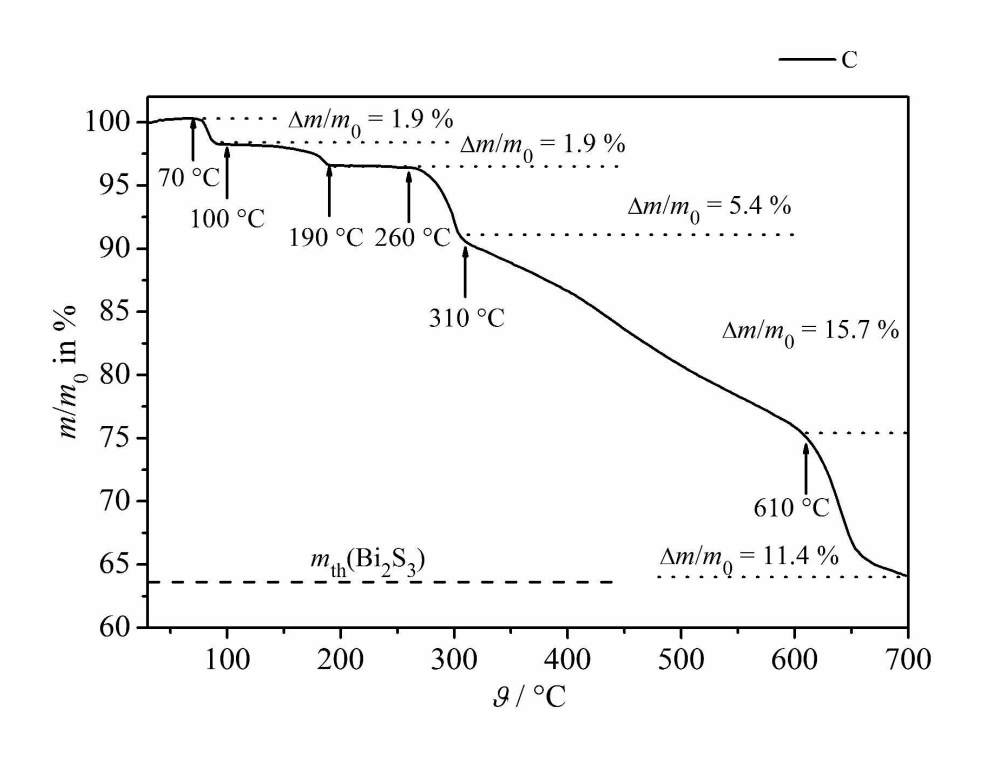

Thermogravimetry of $Bi(SCN)_3 \cdot ½H_2O$ in a flow of Ar.
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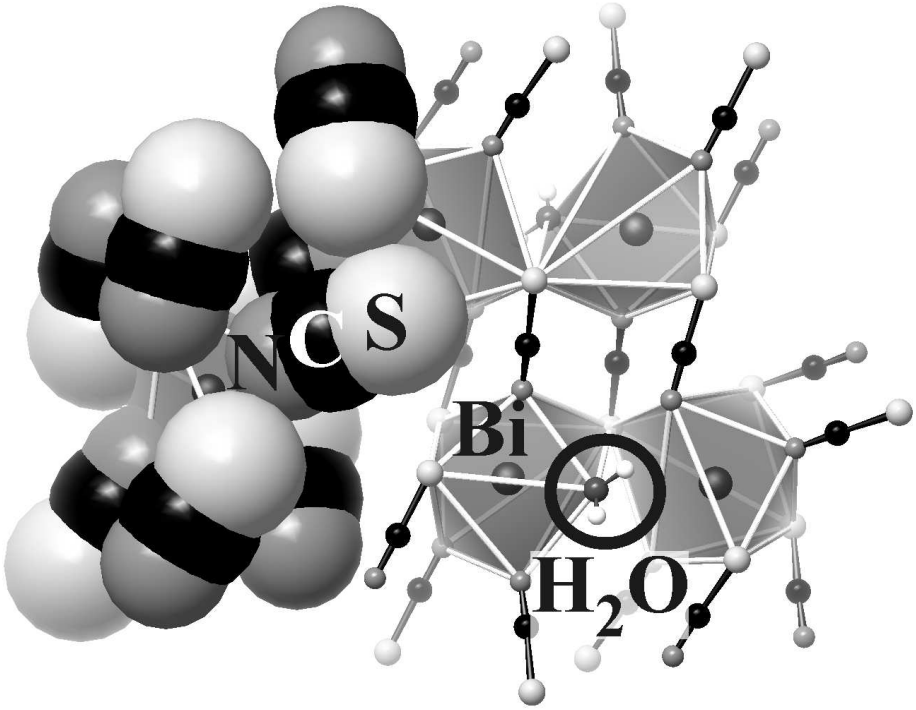
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