Bis(n-pentanethiolato)mercury(II), Hg(SC₅H₅)₂ – Preparation, Characterization, Crystal and Molecular Structure; Reactivity towards Organic Thiols

Gerhard G. Hoffmann a,*, Ingeborg Steinfatt a, Wolfgang Brockner b, and Volker Kaiser c

a Institut für Erdöl- und Erdgasforschung, Walther-Nernst-Str. 7, D-38678 Clausthal-Zellerfeld, Germany
b Institut für Anorganische und Analytische Chemie, Technische Universität Clausthal, Paul-Ernst-Str. 4, D-38678 Clausthal-Zellerfeld, Germany
c Institut für Kristallographie, RWTH Aachen, Jägerstr. 17-19, D-52056 Aachen, Germany

* Reprint requests to Dr. G. G. Hoffmann. E-mail: Gerhard.Hoffmann@ife-clausthal.de

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Crystal structure, IR Data, Raman Data, Mass Spectrum, Bis(n-pentanethiolato)mercury(II)

The preparation of bis(n-pentanethiolato)mercury(II), Hg(SC₅H₅)₂, and n-pentanethiolato-mercury(II) iodide, Hg(SC₅H₅)I, starting from HgI₂ and n-pentanethiol, is presented; the X-ray crystal structure determination of Hg(SC₅H₅)₂ reveals a layered structure of the bis(thiolate) molecule. With 178.69(1.02)° the S-Hg-S moiety is nearly linear. The Hg-S bond length is shorter than the sum of the covalent radii of sulphur and mercury atoms indicating a primary co-ordination number of two, which is supported by the IR and Raman spectra. Assignments of νₛₐₘ(Hg-S) and νₐₙₚ(Hg-S) are given. The mass spectrum reveals the title compound to be monomeric in the gas phase. Melting and thermal decomposition have been investigated by DTA methods. The behaviour towards organic thiols is discussed. In addition, IR and Raman spectroscopic data of HgSC₅H₅I₁ are presented and discussed.

Introduction

The chemistry of mercury has attracted considerable interest, not only due to the high toxicity of mercury and its compounds to living systems [1 - 7]. Moreover, the reactivity of mercury compounds towards the thiolate function is of particular importance owing to the great affinity of thiols to mercury which has been known for a long time [8 - 12]. Especially this affinity of thiols is generally used to remove mercury from the body when poisoned by mercury and its compounds [13 - 15]. Furthermore, it plays an important role in the biological chemistry of mercury [16 - 19].

Previously, some of us reported the reaction of mercury with diorganyl disulphides, such as (CH₃)₂S₂ and (C₆H₅)₂S₂, to result in the formation of the corresponding bis(thiolato)mercury(II) derivatives [20, 21]. These reactions may occur in natural gas reservoirs. Generally, they play a pivotal role in natural systems because of their ability to mobilize and transport mercury into the environment [1 - 7].

Homoleptic thiolate complexes of mercury show a considerable variation in their co-ordination geometry [17, 19, 22 - 36], but the factors determining these geometries are not yet clear [37]. In this connection we report here the preparation and characterization of bis(n-pentanethiolato)mercury(II), as well as its thermochemical behaviour and its reactivity towards organic thiols.

Results and Discussion

Synthesis

An ethanolic solution of one equivalent of HgI₂ readily reacts at RT (20 - 25 °C) with two equivalents of n-pentanethiol to form a voluminous colourless precipitate. By adding excess triethylamine the precipitate becomes microcrystalline, and the title compound can be obtained in good yield, according to eq. (1):

\[
\text{HgI}_2 + 2 \text{C}_5 \text{H}_5 \text{SH} + 2 \text{N(C}_2 \text{H}_3)_3 \rightarrow \text{Hg(SC}_5 \text{H}_5)_2 + 2 [\text{N(C}_2 \text{H}_3)_3 \text{H}] I
\]

If the starting compounds are employed in a 1:1:1

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molar ratio a pale yellow solution results from which IHgSC_{5}H_{11} can be precipitated in high yield as a colourless solid by addition of water, according to eq. (2):

\[
\text{HgI}_2 + C_{5}H_{11}SH + N(C_2H_5)_3 \xrightarrow{1.\text{C}_2\text{H}_5\text{OH}} \text{IHgSC}_{5}\text{H}_{11} + [N(C_2H_5)_3]\text{H}\text{I}
\]  

(2)

To obtain high yields of IHgSC_{5}H_{11} it was again found necessary to use an auxiliary base, such as triethylamine.

**Crystal and molecular structure of Hg(SC_{5}H_{11})_{2}**

The results of this structure determination confirm the models postulated by Wells [22]. Bis(n-pentanethiolato)mercury(II) has a layer structure (Figs. 1a - c).

As with related structures of other mercury thiolates [22, 34, 38] the aliphatic chains of the ligands are cis-orientated. Because of this cis-orientation a close packing of the molecules becomes possible (see Fig. 1 c). The structural parameters could also explain the shape and the quality of the different crystal specimen as well as their reactivity. All these factors together with the well-known disorder of paraffinic crystals can serve as an explanation for the poor Rf values. The decomposition of the compound leading to di-n-pentyl disulphide and mercury seems to be preconditioned by both the cis-orientation and the weak interactions between the molecules. The habitus of the very thin crystal plates of different bis(n-alkanethiolato)mercury(II) moieties is also clearly determined by the cis-orientation and the packing as shown in Fig. 1. The Hg-S bond length is in good agreement with literature data (cf. summary in [35]).

**IR and Raman spectra**

IR and Raman spectra of Hg(SC_{5}H_{11})_{2} and IHgSC_{5}H_{11} are shown in Figs. 2 and 3. In previous studies Hg-S stretching frequencies \(\nu_{\text{sym}}(\text{Hg-S})\) and \(\nu_{\text{asym}}(\text{Hg-S})\) were reported as well as other stretching modes of interest, such as \(\nu(\text{C-S})\). Bendings, such as \(\delta(\text{C-C-C})\), \(\delta(\text{C-C-S})\), \(\delta(\text{C-S-Hg})\), and \(\delta(\text{Hg-S})\), as well as rockings, such as \(\rho(\text{CH}_{2})\) occur in the same region as the stretching frequencies (800 - 50 cm\(^{-1}\)) and may overlap each other [35, 39 - 44].

![Fig. 2. Infrared (upper) and FT-Raman spectra (lower) of bis(n-pentanethiolato)mercury(II) at room temperature in the region 850 - 50 cm\(^{-1}\).](image)
Hg-S stretching frequencies strongly depend upon the co-ordination number of mercury [35]. Increasing co-ordination from two to four corresponds with a decrease and with splittings of \( \nu (\text{Hg-S}) \) frequencies and modes, respectively. However, only atoms which are strongly bonded to mercury (average bond length below ca. 2.8 Å) are of influence on the relevant vibrational modes [35, 41].

Because of the almost linear S-Hg-S skeleton with a primary co-ordination of two for mercury in bis(n-pentanethiolato)mercury(II) two Hg-S stretching modes can be expected in the vibrational spectra. These modes can be assigned to the intense IR active band at 377 cm\(^{-1} \) [\( \nu _{\text{asym}} (\text{Hg-S}) \)] and the strong Raman active mode at 350 cm\(^{-1} \) [\( \nu _{\text{sym}} (\text{Hg-S}) \)] with a very weak IR counterpart, respectively. The C-S-Hg bending modes have been suggested to occur in the range of 180 - 120 cm\(^{-1} \) for related compounds [35, 39]. A strong Raman line at 131 cm\(^{-1} \) can be tentatively ascribed to that mode, but S-Hg-S bending modes are found in the same region. We propose the Raman frequency at 113 cm\(^{-1} \) to be caused by the \( \delta (\text{S-Hg-S}) \) bending. The bands observed in the region 730 - 700 cm\(^{-1} \) can be assigned to CH\(_2\) rocking (IR: 724 cm\(^{-1} \)) and C-S stretching (IR: 729 cm\(^{-1} \); Raman: 723 cm\(^{-1} \)) vibrations [39, 45].

Only very few vibrational data have been published concerning thiolatomercury(II) iodide derivatives, RSHgl [43, 44], and no crystal structures of such compounds are available. The Raman spectrum of IHgSC\(_5\)H\(_{11}\) shows only two strong bands in the region 400 - 50 cm\(^{-1} \) at 293 and 119 cm\(^{-1} \). We suggest the mode at 293 cm\(^{-1} \) to be caused by the symmetric Hg-S stretching, whereas the band at 119 cm\(^{-1} \) could be assigned to the symmetric Hg-I stretching. The corresponding asymmetric modes in the IR spectrum, which for its part shows far more absorptions in this region, can be assigned to the bands at 330 [\( \nu (\text{Hg-S}) \)] and 142 cm\(^{-1} \) [\( \nu (\text{Hg-I}) \)], respectively. The lowering of the Hg-S stretching modes indicates a higher primary co-ordination on the mercury atom. According to literature data a three-fold co-ordination could be considered [35]. The low \( \nu (\text{Hg-I}) \) frequency indicates an iodine-bridged dimeric structure for IHgSC\(_5\)H\(_{11}\). Hg-I stretching frequencies of similar bridged mercury-iodine complexes were observed at similar wavenumbers [50]. A strong IR absorption at 210 cm\(^{-1} \) could be assigned to another \( \nu (\text{Hg-S}) \) stretching mode, caused by a higher co-ordination of mercury or by a (Hg-I)\(_2\) vibration. However, the existence of more than two Hg-S stretching modes suggests an oligomeric or polymeric structure with alternating bridging functions of sulphur and iodine, in addition.

### Mass spectrometry

The mass spectrum of the title compound shows the molecular ion \( M^+ \) at \( m/z = 408 \) with the characteristic isotope splitting pattern and an intensity of about 3% of the base peak which appears at \( m/z = 71 \) (C\(_3\)H\(_{11}\)). There are no peaks at higher masses, indicating the compound to be monomeric in the gas phase. Other characteristic peaks for fragment ions containing mercury (most intense peaks) are found at \( m/z = 338 \) (C\(_5\)H\(_{11}\)SHgSH), \( m/z = 305 \) (C\(_5\)H\(_{11}\)SHg), \( m/z = 268 \), (Hg(SH)\(_2\)), and \( m/z = 202 \) (Hg). The latter peak together with \( m/z = 206 \) (C\(_5\)H\(_{11}\)SSC\(_5\)H\(_{11}\)) implies that bis(alkanethiolato)mercury(II) compounds decompose into mercury and the corresponding dialkyl disulphides. Fragment ions characteristic for this decomposition pattern are also present at \( m/z = 136 \) (C\(_5\)H\(_{11}\)SSH) and \( m/z = 103 \) [45]. The mass spec-
trum and the observed isotope pattern for Hg (inset) are shown in Fig. 4.

**Thermal decomposition**

Only scarce information is available concerning the thermochemical behaviour of mercury thiolate derivatives [46-52]. For the decomposition reaction of bis(ethanethiolato)mercury(II) in ethanol Otto could show the formation of mercury and diethyl disulphide [47]. Bis(n-pentanethiolato)mercury(II) decomposes in ethanolic solution into mercury and di-n-pentyl disulphide exclusively, and the reaction is not running to completion as shown by gas chromatographic investigations. This behaviour of the mercury thiolate indicates an equilibrium reaction in solution as suggested by Lecher [48]. The decomposition is accelerated in the presence of iron or iron sulphides.

Without solvent, small amounts of mercury sulphide (HgS) together with di-n-pentyl sulphide are also formed. TG measurements performed under an argon atmosphere support these results. The decomposition of the title compound starts at 110 °C. The reaction shows a maximum peak at 171.2 °C and is complete at 190 °C. The degradation proceeds in one step. At 225 °C a residue of 4.5% of the initial sample weight indicates that ca. 7% of the mercury dithiolate decompose to form HgS.

DSC investigations show the melting point at 68.6 °C. On cooling the melt it recrystallizes again at 57.6 °C.

**Reactivity towards organic thiols**

Ligand exchange reactions of mercury thiolates are of particular interest with respect to biological systems and have been subject to investigations concerning mainly methylmercury species [53, 54]. Bis(n-pentanethiolato)mercury(II) and ethanethiol exchange the functional thiolate groups easily already at ambient temperature. This reaction proceeds in solvents (e.g. CH₂Cl₂ or C₂H₅OH) as well as solventless. The corresponding mercury thiolates together with n-pentanethiol are formed, according to eq. (3):

\[
\text{Hg(SC}_5\text{H}_{11})_2 + 2 \text{C}_2\text{H}_5\text{SH} \leftrightarrow \text{Hg(SC}_2\text{H}_5)_2 + 2 \text{C}_5\text{H}_{11}\text{SH} \tag{3}
\]

Gas chromatographic investigations show the reaction to be an equilibrium which can be shifted to bis(ethanethiolato)mercury(II) if ethanethiol is employed in large excess. However, species containing the two different thiolate groups in one molecule (C₅H₁₁SHgSC₂H₅) could not be observed. Furthermore, binary mixtures of stoichiometric amounts of the two corresponding mercury bis-thiolates in CH₂Cl₂ solution do not react to give mixed compounds either. Gas chromatographic results show that at higher temperature decomposition of the bis(n-alkanethiolato)mercury(II) compounds ensues. Mercury and all possible disulphides are formed as shown in eq. (4):

\[
3 \text{Hg(SC}_5\text{H}_{11})_2 + 3 \text{Hg(SC}_2\text{H}_5)_2 \xrightarrow{\text{high } T} 6 \text{Hg} + 2 (\text{C}_2\text{H}_5\text{S})_2 + 2 (\text{C}_5\text{H}_{11}\text{S})_2 + 2 \text{C}_2\text{H}_5\text{SC}_5\text{H}_{11} \tag{4}
\]
In separate experiments it could be demonstrated that an exchange of the disulphide ligands of different disulphides is strongly accelerated in the presence of mercury thiolates, according to eq. (5):

$$\text{RSSR} + \text{R'SSR}' \rightarrow \text{Hg(SCS\textsubscript{R}H\textsubscript{R}')_2} \rightarrow 2 \text{RSSR'}$$  \hspace{1cm} (5)

$$\text{R, R' = alkyl}$$

**Conclusion**

Hg(S\textsubscript{C\textsubscript{5}H\textsubscript{n}}\textsubscript{2})\textsubscript{2} and IHg(SC\textsubscript{5}H\textsubscript{11}) have been prepared from HgI\textsubscript{2} and \textit{n}-pentanethiol using N(C\textsubscript{2}H\textsubscript{5})\textsubscript{3} as an auxiliary base. IR and Raman data indicate the title compound to be monomeric. This finding is consistent with the evidence from the crystal structure which shows that the compound occupies a highly ordered layered structure consisting of monomers with almost linear S-Hg-S units, and the mass spectrum showed the compound to be also monomeric in the gas phase. Thermoanalytical investigations revealed that the title compound has a reproducible melting point and decomposes at higher temperatures to give mercury, mercury sulphide and the corresponding di-\textit{n}-pentyl disulphide and sulphide. In the presence of thiols an exchange of the thiolate functional groups easily occurs in solution already at ambient temperature.

**Experimental**

All solvents and chemicals were reagent grade and used as received. FT-Raman spectra were recorded with a Raman module FRA 106 (Nd: YAG laser: 1064 nm, < 200 mW) attached to a Bruker IFS 66v interferometer. FT-IR spectra were received from KBr and CsI pellets, and/or Nujol grindings between PE plates with the interferometer. FT-IR spectra were recorded with a Perkin Elmer Paragon 1000 FT-IR spectrometer (in the range 4000 - 200 cm\(^{-1}\)) and a Perkin Elmer Paragon Nujol spectrometer equipped with an electron impact ion source (70 eV) at a resolution of m/Δm = 1000, using a direct probe-inlet system.

Gas chromatographic investigations were performed using a Hewlett Packard Model 5890 Series II instrument, equipped with a 30 m DB 5 capillary column (i.d.: 0.25 mm) and a Hewlett Packard Flame Photometric Detector (Initial temperature 35 °C and ramping to 320 °C at 5 °C/min). The injector temperature and the detector temperature were set to 280 °C, the helium carrier-gas flow was 1 ml/min.

Elemental analyses (C, H) were performed with a Heraeus Elemental Analyser, and sulphur was analysed according to the method of Schöniger [55].

DSC and TG measurements were obtained using a 910 Differential Scanning Calorimeter, a 951 Thermogravimetric Analyser, and a Thermal Analyst 2100 for data analyses.

For single crystal investigations a four circle diffractometer CAD4 (NONIUS) and an imaging plate system IPDS (STOE & Cie) with graphite monochromated Mo-K\textsubscript{α} radiation were used.

**Preparations**

Because of the toxicity of mercury and its compounds all reactions were carried out in Schlenk tubes.

**Bis(\textit{n}-pentanethiolato)mercury(II), [Hg(SC\textsubscript{5}H\textsubscript{11})\textsubscript{2}]**

\textit{n}-Pentanethiol (0.60 ml, 5.06 mmol) was added to a solution of mercury(II) iodide (1.07 g, 2.36 mmol) in 30 ml of absolute ethanol. A voluminous precipitate formed immediately which became microcrystalline after adding triethylamine (1 ml, 3.20 mmol) to the reaction mixture. After stirring for 12 h in the dark, the colourless crystals were collected, washed twice with cold absolute ethanol and dried under vacuum without further purification. Yield: 0.75 g (78%), m. p. 68.6 °C.

C\textsubscript{16}H\textsubscript{22}HgS\textsubscript{2} (406,998)

Calcd C 29.51 H 5.45 S 15.76%,

Found C 29.00 H 5.34 S 15.67%.

IR (ν\textsubscript{max/cm\(^{-1}\)}): 2955vs, 2925vs, 2869vs, 2858vs, 2793w (sh), 1464s, 1457s, 1435s, 1370s, 1332m, 1297w, 1272m, 1257w, 1209s, 1189w, 1109m, 1060m, 1034w, 997w, 945w, 896m, 827m, 750w, 729s [ν(C-S)], 724s [ν(CH\textsubscript{2})], 460m, 377s [ν\textsubscript{asym}(Hg-S)], 349vw [ν\textsubscript{asym}(Hg-S)] and 225m (CsI, pellet).

Raman (ν\textsubscript{max/cm\(^{-1}\)}): 2958m, 2921s, 2898vs, 2871s, 2852m, 2721w, 2712w, 2659w, 1502w, 1462w (sh), 1445m, 1297w, 1272w, 1253w (sh), 1209w, 1186w, 1107w, 1056w, 1048w, 986w, 894m, 723m [ν(C-S)], 458m, 367w, 350s [ν\textsubscript{asym}(Hg-S)], 222m, 131s [δ(C-S-Hg)], 113m [δ(S-Hg-S); tentatively], 91w (sh) and 81s.

**n-Pentanethiolatomercurry(II) iodide, [IHgSC\textsubscript{5}H\textsubscript{11}]**

\textit{n}-Pentanethiol (0.29 ml, 2.34 mmol) was added to a solution of mercury(II) iodide (1.05 g, 2.31 mmol) in 30 ml of absolute ethanol. A voluminous precipitate was formed immediately which dissolved completely on adding triethylamine (0.32 ml, 2.31 mmol). The solution turned from colourless to pale yellow. After adding 20 ml of water an off-white crystalline solid formed which was collected, washed twice with cold absolute ethanol and dried under vacuum without further purification. Yield: 0.91 g (91%).
Bis(n-pentanethiolato)mercury(II), Hg(SC$_5$H$_n$)$_2$

Calcd C 13.94 H 2.57 S 7.45%, Found C 13.91 H 2.46 S 7.67%.

IR ($\nu_{\text{max}}$/$\text{cm}^{-1}$): 2956vs, 2926vs, 2870vs, 2857vs, 1465s, 1454s, 1441m (sh), 1417s, 1374m, 1359w, 1298w, 1214s, 1179w, 1107m, 1071m, 1036w, 971w, 950w, 893w, 828m, 765w, 721s

\[ \nu(C-S) \text{ and } \nu(CH_2) \text{ (CsI, pellet).} \] (max/cm$^{-1}$): 452w, 364m, 330s O a S ym(Hg-S)$_t$, 312m, 307m, 210s \[ \nu_{\text{asym}}(Hg-S)_b \text{ or } \nu_{\text{asym}}(Hg-I)_t \] and 142s \[ \nu(Hg-I)_b \] (PE, Nujol).

X-Ray structure determination of Hg(SC$_5$H$_n$)$_2$

Suitable crystals were obtained by recrystallization from absolute ethanol. Using a polarization microscope, various crystals, apparent to be single crystals, were selected, but in all cases there was a macroscopic deformity. This behaviour can be explained by the pronounced layer character of the crystal structure. Bis(n-pentanethiolato)mercury(II) crystallizes in the monoclinic space group C$_2$/c (No. 15 [56]) with lattice constants $a = 7.3684(17)$, $b = 5.2447(11)$, $c = 34.6177(82)$ Å and $\beta = 96.39(28)$. The determined lattice constants agree with those given in literature [22]. For the first investigations of the compound a four circle diffractometer (CAD4; NONIUS) was used. The same cell was found, but the determination of the space group by reflection conditions led to C$m$ (No. 12 [56]). Only in the acentric space group a solution and refinement of the structure was successful (final $R_f = 0.1300$ based on 1449 Fo > 4$\sigma$(Fo) [57] for 70 parameters). However, there is no reason for a polar space group. Therefore, additional experiments were carried out on an imaging plate system (IPDS; STOE & Cie) which gave a suitable explanation for the problem.

The very thin plates of Hg(SC$_5$H$_n$)$_2$ are not real single crystals. In the direction of the crystallographic b axis there are several specimen piled up and twisted in a range of about $\pm$ 3 - 5° in relation to the c axis. This behaviour leads to wrong intensity relations between the reflections as a function of the $\theta$ angle. For low angle reflections the integrated intensity comes from more specimen as for high angle reflections. The reflections are located on the corresponding diffraction rings. To explain this problem Fig. 5 shows a sketch of the h0l layer from the reciprocal space.

![Fig. 5. Sketch drawing (not scaled) of the h0l layer showing the condition which led to the wrong space group (l $\neq$ 2n → C2/m). Several specimen (many more then 5) are piled up and twisted in a range of about $\pm$ 3 - 5° in relation to the c* axis (rotation to the left).](#)

It gives an impression for the obtained reflections and the condition leading to the wrong space group (l $\neq$ 2n). Until now it was impossible to obtain the integrated intensity accumulated over all specimen from the images, because there is no tool available for such a special problem.

<table>
<thead>
<tr>
<th>Atom</th>
<th>x</th>
<th>y</th>
<th>z</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hg</td>
<td>0.5</td>
<td>0.1762(1)</td>
<td>0.75</td>
</tr>
<tr>
<td>S</td>
<td>0.2292(2)</td>
<td>0.1812(4)</td>
<td>0.7097(1)</td>
</tr>
<tr>
<td>C(1)</td>
<td>0.2640(7)</td>
<td>0.423(1)</td>
<td>0.6724(1)</td>
</tr>
<tr>
<td>H(1)A</td>
<td>0.2590(7)</td>
<td>0.592(1)</td>
<td>0.6835(1)</td>
</tr>
<tr>
<td>H(1)B</td>
<td>0.3831(7)</td>
<td>0.400(1)</td>
<td>0.6635(1)</td>
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<tr>
<td>C(2)</td>
<td>0.1220(8)</td>
<td>0.396(1)</td>
<td>0.6399(1)</td>
</tr>
<tr>
<td>H(2)A</td>
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<td>0.404(1)</td>
<td>0.6493(1)</td>
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<tr>
<td>H(2)B</td>
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<td>0.6278(1)</td>
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<tr>
<td>C(3)</td>
<td>0.1371(1)</td>
<td>0.616(2)</td>
<td>0.6082(2)</td>
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<tr>
<td>H(3)A</td>
<td>0.125(1)</td>
<td>0.780(2)</td>
<td>0.6205(2)</td>
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<tr>
<td>H(3)B</td>
<td>0.257(1)</td>
<td>0.609(2)</td>
<td>0.5994(2)</td>
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<tr>
<td>C(4)</td>
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<td>0.596(2)</td>
<td>0.5731(2)</td>
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<tr>
<td>H(4)B</td>
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<td>0.631(1)</td>
<td>0.5813(2)</td>
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<td>0.5402(2)</td>
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<td>0.5188(2)</td>
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<td>H(5)B</td>
<td>0.145(1)</td>
<td>0.745(2)</td>
<td>0.5317(2)</td>
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<tr>
<td>H(5)C</td>
<td>0.025(1)</td>
<td>0.952(2)</td>
<td>0.5494(2)</td>
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</table>
Table II. Selected bond lengths (Å) and angles (°) for bis-
(n-pentanethiolato)mercury(II) (Standard deviations in
brackets).

<table>
<thead>
<tr>
<th>Bond Lengths (Å)</th>
<th>Angles (°)</th>
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<tr>
<td>Hg-S (2x) 2.30(2)</td>
<td>S-Hg-S 179(1)</td>
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<tr>
<td>S-C(1) 1.85(5)</td>
<td>Hg-S-C(1) 105(2)</td>
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<tr>
<td>C(1)-C(2) 1.46(8)</td>
<td>S-C(1)-C(2) 109(4)</td>
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<tr>
<td>C(2)-C(3) 1.60(9)</td>
<td>C(1)-C(2)-C(3) 111(5)</td>
</tr>
<tr>
<td>C(3)-C(4) 1.5(1)</td>
<td>C(2)-C(3)-C(4) 114(6)</td>
</tr>
<tr>
<td>C(4)-C(5) 1.5(1)</td>
<td>C(3)-C(4)-C(5) 113(7)</td>
</tr>
</tbody>
</table>

But in spite of the harmed and therefore incomplete integrated data ($R_{int} = 0.2151$) the solution of the structure was successful using the programs SHELXS [58] and SIR 97 [59] All atoms, with the exception of the H atoms, were located by using direct methods. For the structure refinement the program SHELXL 93 [57] was applied. Because of the poor intensity data only the Hg and S atoms were refined anisotropically. The positions of the hydrogen atoms were calculated and refined under geometrical constraints. A final $R_1 = 0.1349$ (based on 433 Fo > 4σ(Fo) [57] for 35 parameters) was obtained. The arrangement of the molecules in the unit cell is shown in Fig. 1 [60]. Refined fractional co-ordinates and selected bond lengths and angles are given in Tables I and II.

Acknowledgements

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