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Crystal structure of *catena*-poly[bis(*N,O*-dimethylhydroxylammonium) [di- μ -bromido-dibromidostannate(II)]]

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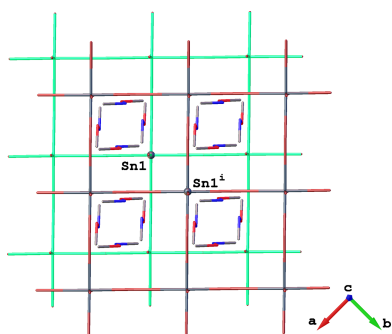
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The title compound, $\{(C_2H_8NO)_2[SnBr_4]\}_n$, is a layered hybrid perovskite crystallizing in the monoclinic space group $C2/c$. The asymmetric unit consists of one $H_3C-O-NH_2^+-CH_3$ cation (Me_2HA^+), one Sn^{II} atom located on a twofold rotation axis, and two Br atoms. The Sn^{II} atom has a distorted octahedral coordination environment formed by the bromido ligands. The $\{SnBr_6\}$ units corner-share their equatorial Br atoms, forming infinite mono-layers that extend parallel to the *ab* plane. These inorganic layers are sandwiched by the organic Me_2HA^+ cations organized in double-layers; stacking of the layers is along the *c*-axis direction. Consecutive inorganic layers, separated by the organic cations, are shifted relative to each other along the *b*-axis direction. Specifically, the Sn^{II} atom in one inorganic layer is offset by 3.148 Å along the *b* axis relative to the Sn^{II} atom in an adjacent inorganic layer. The *N,O*-dimethylhydroxylammonium cation forms two hydrogen bonds with the axial bromide anions of the inorganic layers as acceptors, and leads to the cohesion of the crystal structure. According to Hirshfeld surface analysis, the highest contributions to the crystal packing are from $H\cdots H$ (46.2%), $Br\cdots H$ (38.5%), and $H\cdots O$ (14.8%) contacts.

1. Chemical context

Hybrid perovskites are an important class of solution-processable semiconducting materials that exhibit interesting electrical and optical properties. Lead halide hybrid perovskites, the most prominent members of the hybrid perovskite family, have demonstrated high power conversion efficiency in solar cells and high photoluminescence quantum yields (Sun *et al.*, 2017; Shamsi *et al.*, 2019). Despite the promising potential of lead halide hybrid perovskites, the high toxicity of lead limits their commercialization. Lead can be easily absorbed into the bloodstream and cause damage to the cardiovascular system, kidneys, reproductive system, DNA, and the central nervous system, leading to brain disorders and, ultimately, death (Jadhav *et al.*, 2007). One approach to overcoming lead toxicity is to replace lead with less toxic elements like tin or germanium, which have ionic radii similar to that of lead (Hao *et al.*, 2014).

In the past decade, tin halide hybrid perovskites have gained significant attention for solar energy applications due to their low toxicity and tunable band gap (Milot *et al.*, 2018; Li *et al.*, 2023). However, a major challenge with tin halide hybrid perovskites is the ease with which Sn^{II} oxidizes to Sn^{IV} (Byrannvand *et al.*, 2022). One method to stabilize Sn^{II} is by incorporating it into the crystal structure of layered hybrid

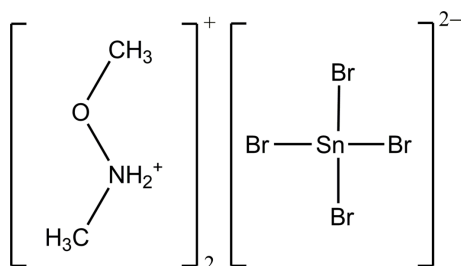


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perovskites (Byranvand *et al.*, 2022). Such layered hybrid perovskites, commonly denoted as ‘two-dimensional’ hybrid perovskites, are stoichiometric compounds composed of alternating inorganic metal halide layers and organic cationic layers. This class of materials offers exceptional flexibility in terms of composition, structure, and dimensionality (Straus & Kagan, 2018). It has been shown that the hydrophobic nature of the bulky organic cations that form the organic layers in these hybrid perovskites prevents moisture from interacting with the ionic inorganic layers composed of metal halide octahedra (Azmi *et al.*, 2024).

In this context, the search for new tin halide hybrid perovskites with a broad range of optoelectronic properties is crucial for the development of more efficient solar cells, optoelectronic, and spintronic devices. One approach to modifying the crystal structure and properties of layered hybrid perovskites is to use different templating organic cations. Hydroxylamines, which have been relatively under-explored, represent a promising class towards such cations. Only a few studies have reported on the synthesis of hydroxylamine-based metal halides (D’Annibale *et al.*, 2019; Froschauer *et al.*, 2013; Zhang *et al.*, 2018; Schottenberger *et al.*, 2015; Saal *et al.*, 2017; Ben Hmida *et al.*, 2019; Yuan *et al.*, 2019; Ban *et al.*, 1999).

In the current study, we synthesized a new layered hybrid perovskite using the reaction between *N,O*-dimethylhydroxylamine and tin(II) hydroxide in concentrated hydrobromic acid. Detailed structural and Hirshfeld surface analyses of the resulting compound, $(\text{C}_2\text{H}_8\text{NO}^+)_2[\text{SnBr}_4]^-$, (**1**), were performed.



2. Structural commentary

Compound (**1**) has a layered crystal structure. The asymmetric unit includes one Sn^{II} atom (located on a twofold rotation axis), two Br atoms and one *N,O*-dimethylhydroxylammonium cation (Fig. 1). The Sn^{II} atom is coordinated by six bromido ligands, forming a distorted $\{\text{SnBr}_6\}$ octahedron. The Sn—Br distances within the octahedron range from 2.7141 (8) Å to 3.2399 (9) Å (Table 1). Interestingly, a special type of octahedral distortion is realized, with two short equatorial Sn—Br2 bonds [2.7143 (8) Å], two long equatorial Sn—Br2 bonds [3.2395 (8) Å] and two middle-length axial Sn—Br1 bonds [2.9790 (8) Å]. The *cis*-Br—Sn—Br bond angles are in the range 84.82 (2)–98.52 (2)°, and the *trans*-Br—Sn—Br bond angles are 172.29 (3)° for equatorial Br[−] ligands and 175.25 (4)° for axial Br[−] ligands (Table 1), which

Table 1

Selected geometric parameters (Å, °).

Sn1—Br1	2.9790 (8)	Sn1—Br2 ⁱ	3.2395 (8)
Sn1—Br2	2.7143 (8)		
Br1—Sn1—Br1 ⁱⁱ	175.25 (4)	Br2 ⁱⁱⁱ —Sn1—Br2 ⁱ	88.972 (6)
Sn1—Br2 ⁱ —Sn1 ⁱ	172.29 (3)	Br1—Sn1—Br2 ⁱ	84.82 (2)
Br2—Sn1—Br1 ⁱⁱ	89.19 (3)	Br2—Sn1—Br2 ⁱⁱ	91.66 (4)
Br2—Sn1—Br1	87.50 (2)	Br1 ⁱⁱⁱ —Sn1—Br2 ⁱ	98.52 (2)

Symmetry codes: (i) $x + \frac{1}{2}, y + \frac{1}{2}, z$; (ii) $-x + 1, y, -z + \frac{3}{2}$.

indicates the distortion from ideal values of 90° and 180°, respectively. Quantitative octahedral distortion parameters can be calculated by formula $\Delta d = (1/6)\Sigma_{i=1}^6 (d_i - d)^2/d^2$ (1) and $\Sigma = \Sigma_{i=1}^{12} |90 - \alpha_i|$ (2), where d_i is the individual bond length and d is average bond length and α_i are twelve individual *cis*-angles in the coordination octahedron. The value of Δd is $6.41 \cdot 10^{-3}$, which is typical for layered tin halide hybrid perovskites (Sirenko *et al.*, 2024), and the Σ value is 39.21°.

The polymeric inorganic layers of (**1**) propagate parallel to the *ab* plane and are formed by sharing all equatorial corners of the $\{\text{SnBr}_6\}$ octahedra. These inorganic $[\text{SnBr}_{4/2}\text{Br}_{2/1}]_{\infty}^{2-}$ layers (where Br_{4/2} denotes four equatorial atoms bonded in a corner-sharing manner and Br_{2/1} denotes two axial halogen atoms bonded only to one Sn^{II} atom) are separated by organic Me_2HA^+ cations (Fig. 2); the stacking direction of the layers is along the *c*-axis direction. The organic cations are aligned parallel to the *c* axis and organized in double layers (Fig. 2).

3. Supramolecular features

The *N,O*-dimethylhydroxylammonium cations interact with the inorganic layers through the protonated secondary amino

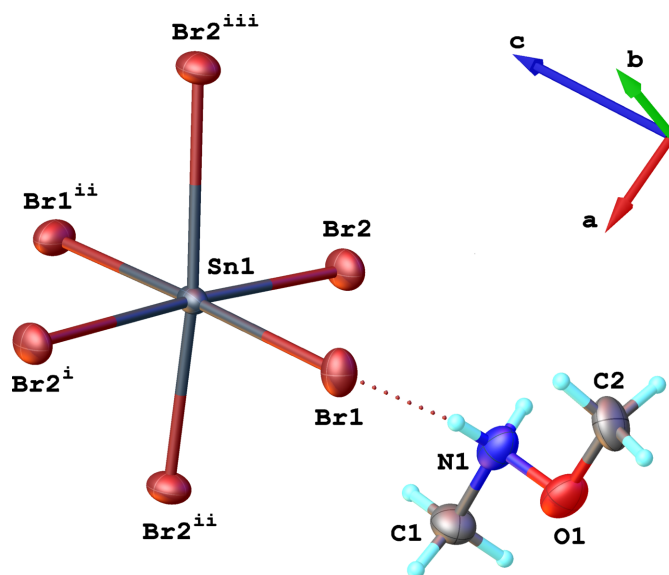


Figure 1

Representation of the building units in the crystal structure of compound (**1**), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are shown as small spheres of arbitrary radius. [Symmetry codes: (i) $\frac{1}{2} + x, \frac{1}{2} + y, z$; (ii) $1 - x, y, \frac{3}{2} - z$; (iii) $\frac{1}{2} - x, \frac{1}{2} + y, \frac{3}{2} - z$.]

Table 2

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N1-H1A\cdots Br1^{iii}$	0.80	2.58	3.358 (6)	165
$N1-H1B\cdots Br1$	0.80	2.58	3.358 (6)	165
$C1-H1D\cdots Br2$	0.96	3.16	3.778 (9)	123
$C2-H2B\cdots Br1$	0.96	3.04	3.818 (8)	139
$C2-H2C\cdots Br1^{iii}$	0.96	3.09	3.803 (9)	132

Symmetry code: (iii) $x - \frac{1}{2}, y - \frac{1}{2}, z$.

group, forming $N-H\cdots Br$ hydrogen bonds exclusively with the axially positioned Br^- anions (Fig. 3, Table 2). Additionally, in the crystal structure of (1), weak $C-H\cdots Br$ contacts are present. As was previously shown, $C-H\cdots Br$ hydrogen bonding can be assumed when the difference $(r_{C\cdots B}) - (r_{vdw}B + r_{C-H}) < 1.00$ Å, where r_{C-H} is the average $C-H$ bond length, and $r_{vdw}B$ is the van der Waals radius of the hydrogen-bond acceptor (Harmon *et al.*, 1992). For weak contacts $C1-H1D\cdots Br2$, $C2-H2B\cdots Br1$ and $C2-H2C\cdots Br1^{iii}$, this difference is less than 1.00 Å (0.868, 0.908 and 0.893 Å, respectively), which may indicate the presence of such weak hydrogen bonds (Table 2). While the angle for $C1-H1D\cdots Br2$ (123.3°) is less characteristic of hydrogen bonding, the angles for $C2-H2B\cdots Br1$ and $C2-H2C\cdots Br1^{iii}$ deviate less from the ideal value of 180° (Table 2), further supporting the possible presence of weak hydrogen bonding (Harmon *et al.*, 1992).

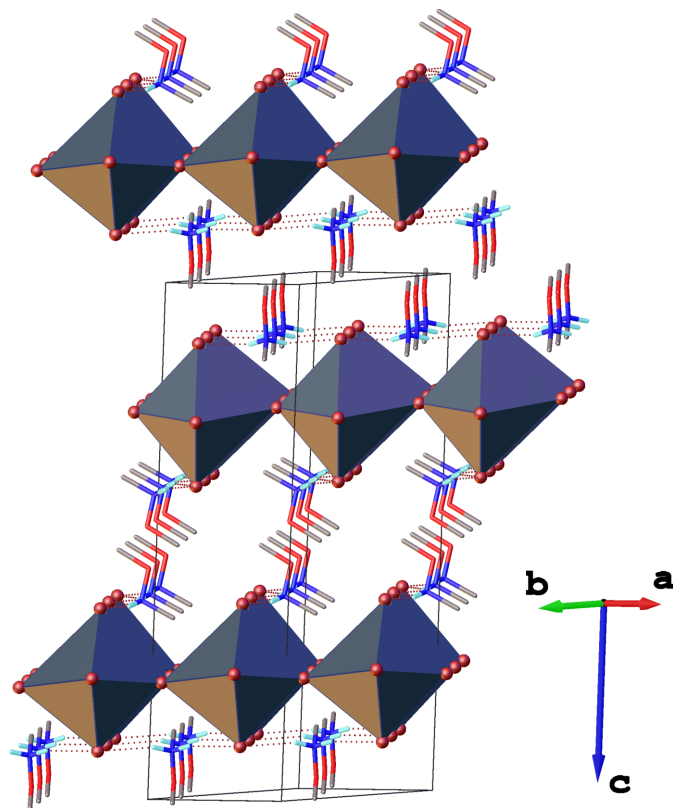


Figure 2

Projection of the crystal structure of (1) approximately along [110], showing its layered crystal structure.

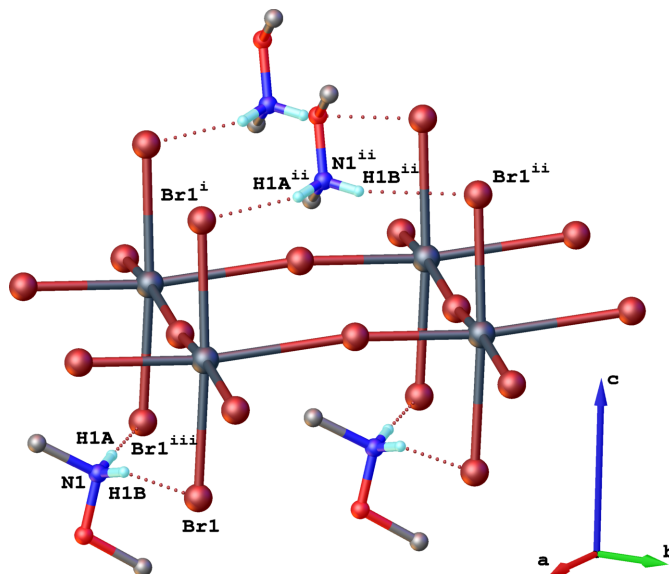


Figure 3

Side view of a fragment of the crystal structure of (1), showing the orientation of organic cations and the hydrogen-bonding scheme (dotted lines). [Symmetry codes: (i) $1 - x, y, \frac{3}{2} - z$; (ii) $\frac{1}{2} - x, \frac{1}{2} + y, \frac{3}{2} - z$; (iii) $-\frac{1}{2} + x, -\frac{1}{2} + y, z$.]

It is worth noting that the *N,O*-dimethylhydroxylammonium cations are oriented perpendicularly to each other on opposite sides of the inorganic layer (Fig. 3). Such an orientation of the organic cations leads to a less distorted inorganic layer compared to the case where these cations were aligned parallel to each other on both sides of the inorganic layer. This orientation of the organic cations results from the

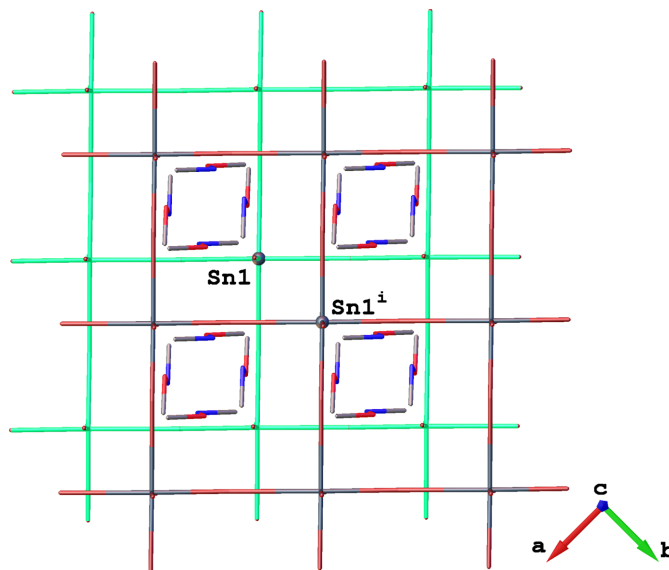


Figure 4

View along [001] of a fragment of inorganic layers in the crystal structure of (1). The upper inorganic layer is offset along the b axis relative to the adjacent inorganic layer (highlighted in green). In this projection, the distance between the Sn^{II} atom of the first inorganic layer is offset by 3.148 Å along the b axis relative to the adjacent layer. [Symmetry code: (i) $x, 2 - y, \frac{1}{2} + z$.]

neighbouring inorganic layers positioning themselves in a manner that minimizes the free space between the Me_2HA^+ cations within the organic layers. The two neighbouring inorganic layers, separated by the double layers of organic cations, are shifted along the b axis; the Sn^{II} atom in one inorganic layer is offset by 3.148 Å along the b axis relative to the Sn^{II} atom located in the adjacent inorganic layer (Fig. 4).

4. Hirshfeld surface analysis

A Hirshfeld surface analysis was performed and the associated two-dimensional fingerprint plots were generated using *CrystalExplorer* (Spackman *et al.*, 2021), with standard resolution of the three-dimensional d_{norm} surfaces, revealing two prominent red spots and several white regions (Fig. 5). Visualizations were performed using a red–white–blue colour scheme, where red regions highlight shorter contacts, white regions indicate contacts around the van der Waals (vdW) separation, and blue areas depict longer contacts. The red spots on the Hirshfeld surface are attributed to the rather strong intermolecular $\text{N}—\text{H} \cdots \text{Br}$ hydrogen bonds, while the white regions mainly correspond to $\text{H} \cdots \text{H}$ and $\text{O} \cdots \text{H}/\text{H} \cdots \text{O}$ contacts.

The overall two-dimensional fingerprint plot (Fig. 5b), along with plots showing $\text{H} \cdots \text{H}$, $\text{Br} \cdots \text{H}/\text{H} \cdots \text{Br}$, $\text{O} \cdots \text{H}/\text{H} \cdots \text{O}$, $\text{O} \cdots \text{O}$, $\text{Br} \cdots \text{O}/\text{O} \cdots \text{Br}$, and $\text{Sn} \cdots \text{H}/\text{H} \cdots \text{Sn}$ contacts and their relative contributions to the Hirshfeld surface are illustrated in Fig. 5c–j. The most abundant interaction is $\text{H} \cdots \text{H}$, which contributes 46.2% to the crystal structure of the

title compound (Fig. 5c), with a characteristic tip at $d_e = d_i = 1.3$ Å. The $\text{Br} \cdots \text{H}/\text{H} \cdots \text{Br}$ and $\text{O} \cdots \text{H}/\text{H} \cdots \text{O}$ interactions contribute 38.5% and 14.8%, respectively, to the crystal structure (Fig. 5d–e).

The $\text{Br} \cdots \text{H}/\text{H} \cdots \text{Br}$ contacts form a distinctive spike on the corresponding two-dimensional plot at $(d_i, d_e) = (0.84, 1.53$ Å), corresponding to the closest $\text{N}—\text{H} \cdots \text{Br}$ contact near 2.37 Å, indicating relevant intermolecular interactions. Similarly, the $\text{O} \cdots \text{H}/\text{H} \cdots \text{O}$ contacts appear as a pair of spikes at $(d_i, d_e) = (1.55, 1.32$ Å), corresponding to the closest $\text{C}—\text{H} \cdots \text{O}$ contact near 2.87 Å in the crystal structure. The contributions of the remaining $\text{O} \cdots \text{O}$, $\text{Br} \cdots \text{O}/\text{O} \cdots \text{Br}$, and $\text{Sn} \cdots \text{H}/\text{H} \cdots \text{Sn}$ interactions are smaller than 1.0%. The contributions of $\text{Br} \cdots \text{H}/\text{H} \cdots \text{Br}$ and $\text{O} \cdots \text{H}/\text{H} \cdots \text{O}$ contacts highlight the significant role of hydrogen bonding and van der Waals interactions in the crystal packing of (**1**).

5. Database survey

A search of the Cambridge Structure Database (CSD version 5.44, last update June 2023; Groom *et al.*, 2016) revealed 73 structures containing $\{\text{SnBr}_6\}$ octahedra and cations of organic amines. Selected examples include EKIWUS (Lorena *et al.*, 2014) and MINNOQ (Fu *et al.*, 2023), which are both layered hybrid perovskites. EKIWUS is a tin bromide compound with inorganic layers built from corner-sharing $\{\text{SnBr}_6\}$ octahedra, while the organic cations are organized in double layers separating the inorganic layers, similar to the title compound. By contrast, MINNOQ is a mixed lead-tin bromide hybrid perovskite featuring inorganic layers composed of corner-sharing $\{\text{SnBr}_6\}$ and $\{\text{PbBr}_6\}$ octahedra. Unlike EKIWUS, the organic layers in MINNOQ are organized in single layers, resulting in shorter distances between consecutive inorganic layers.

6. Synthesis and crystallization

$\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ (0.1 g, 0.44 mmol) was dissolved in 0.7 ml of distilled water with the addition of a few drops of hydrochloric acid (HCl) to prevent hydrolysis, and the mixture was stirred until complete dissolution. Then 140 µL of $\text{NH}_3 \cdot \text{H}_2\text{O}$ (35% w/w) was added to this solution, which resulted in the colourless precipitate of $\text{SnO} \cdot x\text{H}_2\text{O}$. The solution obtained was centrifuged at 12000 RPM for 2 min to separate $\text{SnO} \cdot x\text{H}_2\text{O}$ from the mother liquor. The resulting solid was washed first with water and then with methanol. After each washing step, the solution was centrifuged again at 12000 RPM for 2 min. Subsequently, the obtained $\text{SnO} \cdot x\text{H}_2\text{O}$ was allowed to dry at 303 K for 5 min before it was dissolved in a mixture of 0.5 ml concentrated HBr acid (48% w/v) and 50 µL H_3PO_2 under heating and continuous stirring. Then, *N,O*-dimethylhydroxylamine hydrochloride (0.89 mmol) was added to the solution. The reagents were stirred until the solution became homogenous. Light-yellow crystals were precipitated upon cooling the solution to room temperature. The crystals were separated and kept in the mother solution prior to the diffraction measurements.

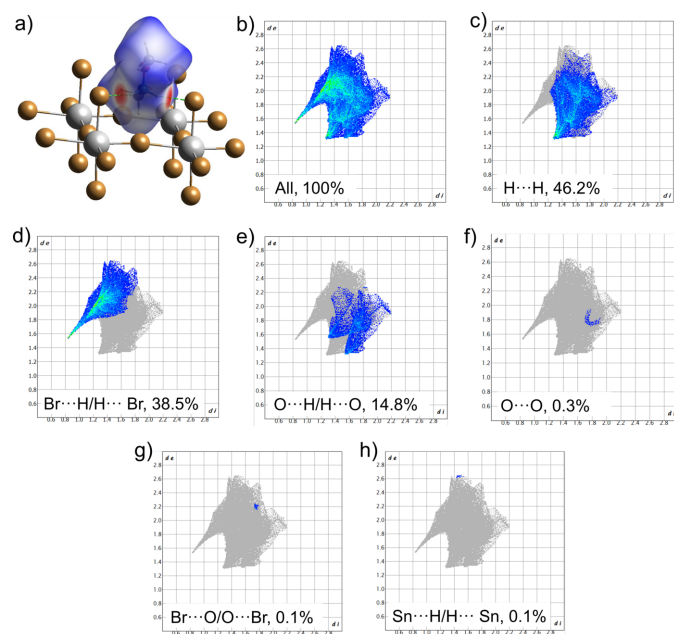


Figure 5

(a) Hirshfeld surface representation with the function d_{norm} plotted onto the surface for the different interactions; two-dimensional fingerprint plots from a Hirshfeld surface analysis of (**1**) showing: (b) all contacts; (c) $\text{H} \cdots \text{H}$ (46.2%); (d) $\text{Br} \cdots \text{H}/\text{H} \cdots \text{Br}$ (38.5%); (e) $\text{O} \cdots \text{H}/\text{H} \cdots \text{O}$ (14.8%); (f) $\text{O} \cdots \text{O}$ (0.3%); (g) $\text{Br} \cdots \text{O}/\text{O} \cdots \text{Br}$ (0.1%) and (h) $\text{Sn} \cdots \text{H}/\text{H} \cdots \text{Sn}$ (0.1%).

7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. Methyl H atoms were positioned geometrically and refined as a rotating group, with C—H = 0.96 Å and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$. The H atoms of the N—H groups were positioned geometrically and refined as riding atoms with N—H = 0.80 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{N})$.

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References

- Azmi, R., Zhumagali, S., Bristow, H., Zhang, S., Yazmaciyan, A., Pininti, A. R., Utomo, D. S., Subbiah, A. S. & De Wolf, S. (2024). *Adv. Mater.* **36**, e2211317.
- Ban, I., Kristl, M., Volavšek, B. & Golič, L. (1999). *Monatsh. Chem.* **130**, 401–408.
- Ben Hmida, W., Jellali, A., Abid, H., Hamdi, B., Naili, H. & Zouari, R. (2019). *J. Mol. Struct.* **1184**, 604–614.
- Byranvand, M. M., Zuo, W., Imani, R., Pazoki, M. & Saliba, M. (2022). *Chem. Sci.* **13**, 6766–6781.
- Clark, R. C. & Reid, J. S. (1995). *Acta Cryst.* **A51**, 887–897.
- D'Annibale, A., Panetta, R., Tarquini, O., Colapietro, M., Quaranta, S., Cassetta, A., Barba, L., Chita, G. & Latini, A. (2019). *Dalton Trans.* **48**, 5397–5407.
- Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.
- Froschauer, C., Salchner, R., Laus, G., Weber, H. K., Tessadri, R., Griesser, U., Wurst, K., Kahlenberg, V. & Schottenberger, H. (2013). *Aust. J. Chem.* **66**, 391.
- Fu, P., Quintero, M. A., Vasileiadou, E. S., Raval, P., Welton, C., Kepenekian, M., Volonakis, G., Even, J., Liu, Y., Malliakas, C., Yang, Y., Laing, C., Dravid, V. P., Reddy, G. N. M., Li, C., Sargent, E. H. & Kanatzidis, M. G. (2023). *J. Am. Chem. Soc.* **145**, 15997–16014.
- Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* **B72**, 171–179.
- Hao, F., Stoumpos, C. C., Cao, D. H., Chang, R. P. H. & Kanatzidis, M. G. (2014). *Nat. Photon.* **8**, 489–494.
- Harmon, K. M., De Santis, N. J. & Brandt, D. O. (1992). *J. Mol. Struct.* **265**, 47–57.
- Jadhav, S. H., Sarkar, S. N., Patil, R. D. & Tripathi, H. C. (2007). *Arch. Environ. Contam. Toxicol.* **53**, 667–677.
- Li, Y., Zhou, H., Xia, M., Shen, H., Wang, T., Gao, H., Sheng, X., Han, Y., Chen, Z., Dou, L., Zhu, H. & Shi, E. (2023). *Sci. Adv.* **9**, eadh0517.
- Lorena, G. S., Hasegawa, H., Takahashi, Y., Harada, J. & Inabe, T. (2014). *Chem. Lett.* **43**, 1535–1537.
- Milot, R. L., Klug, M. T., Davies, C. L., Wang, Z., Kraus, H., Snaith, H. J., Johnston, M. B. & Herz, L. M. (2018). *Adv. Mater.* **30**, e1804506.
- Rigaku OD (2023). *CrysAlis PRO*. Rigaku Oxford Diffraction, Yarnton, England.

Table 3

Experimental details.

Crystal data	
Chemical formula	(C ₂ H ₈ NO) ₂ [SnBr ₄]
M_r	562.52
Crystal system, space group	Monoclinic, C2/c
Temperature (K)	293
a, b, c (Å)	8.4947 (4), 8.3064 (5), 21.6425 (14)
β (°)	96.150 (5)
V (Å ³)	1518.31 (15)
Z	4
Radiation type	Mo $K\alpha$
μ (mm ⁻¹)	12.19
Crystal size (mm)	0.20 × 0.11 × 0.09
Data collection	
Diffractometer	Xcalibur, Eos
Absorption correction	Analytical [<i>CrysAlis PRO</i> (Rigaku OD, 2023) based on expressions derived by Clark & Reid, 1995]
$T_{\text{min}}, T_{\text{max}}$	0.227, 0.438
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	6678, 1832, 1320
R_{int}	0.047
$(\sin \theta/\lambda)_{\text{max}}$ (Å ⁻¹)	0.687
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.046, 0.091, 1.06
No. of reflections	1832
No. of parameters	64
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e Å ⁻³)	0.81, -0.91

Computer programs: *CrysAlis PRO* (Rigaku OD, 2023), *SHELXT* (Sheldrick, 2015a), *SHELXL* (Sheldrick, 2015b), *OLEX2* (Dolomanov *et al.*, 2009) and *publCIF* (Westrip, 2010).

- Saal, T., Rahm, M., Christe, K. O. & Haiges, R. (2017). *Angew. Chem. Int. Ed.* **56**, 9587–9591.
- Schottenberger, H., Nerdinger, S., Laus, G., Salchner, R., Haslinger, S., Kahlenberg, V., Wurst, K. E., Braun, D., Vergeiner, S., Kopacka, H., Puckowski, A. & Stolte, S. (2015). *Heterocycles*, **90**, 1018–1037.
- Shamsi, J., Urban, A. S., Imran, M., De Trizio, L. & Manna, L. (2019). *Chem. Rev.* **119**, 3296–3348.
- Sheldrick, G. M. (2015a). *Acta Cryst.* **A71**, 3–8.
- Sheldrick, G. M. (2015b). *Acta Cryst.* **C71**, 3–8.
- Sirenko, V. Y., Kucheriv, O. I., Shova, S. & Gural'skiy, I. A. (2024). *Mater. Today*, **41**, 102452.
- Spackman, P. R., Turner, M. J., McKinnon, J. J., Wolff, S. K., Grimwood, D. J., Jayatilaka, D. & Spackman, M. A. (2021). *J. Appl. Cryst.* **54**, 1006–1011.
- Straus, D. B. & Kagan, C. R. (2018). *J. Phys. Chem. Lett.* **9**, 1434–1447.
- Sun, H., Yang, Z., Wei, M., Sun, W., Li, X., Ye, S., Zhao, Y., Tan, H., Kynaston, E. L., Schon, T. B., Yan, H., Lu, Z., Ozin, G. A., Sargent, E. H. & Seferos, D. S. (2017). *Adv. Mater.* **29**, 1701153.
- Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.
- Yuan, W., Zeng, Y., Tan, Y.-Y., Zhou, J.-H., Xu, W.-J., Zhang, W.-X. & Chen, X.-M. (2019). *Chem. Commun.* **55**, 8983–8986.
- Zhang, T., Chen, C., Zhang, W.-Y., Ye, Q. & Fu, D.-W. (2018). *Inorg. Chem. Front.* **5**, 2340–2345.

supporting information

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Crystal structure of *catena*-poly[bis(*N,O*-dimethylhydroxylammonium) [di- μ -bromido-dibromidostannate(II)]]

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Computing details

catena-Poly[bis(*N,O*-dimethylhydroxylammonium) [di- μ -bromido-dibromidostannate(II)]]

Crystal data

(C₂H₈NO)₂[SnBr₄]

$M_r = 562.52$

Monoclinic, C2/c

$a = 8.4947$ (4) Å

$b = 8.3064$ (5) Å

$c = 21.6425$ (14) Å

$\beta = 96.150$ (5)°

$V = 1518.31$ (15) Å³

$Z = 4$

$F(000) = 1040$

$D_x = 2.461$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 1867 reflections

$\theta = 3.5$ – 26.2°

$\mu = 12.19$ mm⁻¹

$T = 293$ K

Prism, clear light colourless

$0.20 \times 0.11 \times 0.09$ mm

Data collection

Xcalibur, Eos

diffractometer

Radiation source: fine-focus sealed X-ray tube,

Enhance (Mo) X-ray Source

Graphite monochromator

Detector resolution: 16.1593 pixels mm⁻¹

ω scans

Absorption correction: analytical

[CrysAlisPro (Rigaku OD, 2023) based on
expressions derived by Clark & Reid, 1995]

$T_{\min} = 0.227$, $T_{\max} = 0.438$

6678 measured reflections

1832 independent reflections

1320 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.047$

$\theta_{\max} = 29.2^\circ$, $\theta_{\min} = 1.9^\circ$

$h = -10 \rightarrow 10$

$k = -11 \rightarrow 10$

$l = -28 \rightarrow 27$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.046$

$wR(F^2) = 0.091$

$S = 1.06$

1832 reflections

64 parameters

0 restraints

Primary atom site location: dual

Hydrogen site location: inferred from
neighbouring sites

H atoms treated by a mixture of independent
and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0255P)^2 + 5.9957P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.81$ e Å⁻³

$\Delta\rho_{\min} = -0.91$ e Å⁻³

Extinction correction: SHELXL-2018/3

(Sheldrick 2015b),

$F_c^* = kF_c[1 + 0.001 \times F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.00064 (10)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($\text{\AA}^2$)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Sn1	0.500000	0.81057 (7)	0.750000	0.0296 (2)
Br1	0.49095 (9)	0.79570 (10)	0.61214 (3)	0.0527 (3)
Br2	0.26948 (8)	0.58288 (9)	0.74077 (4)	0.0525 (3)
O1	0.3786 (7)	0.3817 (7)	0.5434 (3)	0.0657 (16)
N1	0.3672 (7)	0.4115 (8)	0.6048 (3)	0.058 (2)
H1A	0.280 (6)	0.3942 (13)	0.6130 (6)	0.069*
H1B	0.3886 (15)	0.503 (6)	0.6129 (6)	0.069*
C2	0.2663 (9)	0.4871 (10)	0.5057 (4)	0.065 (2)
H2A	0.291904	0.488360	0.463565	0.097*
H2B	0.273286	0.594386	0.522265	0.097*
H2C	0.160523	0.447150	0.506703	0.097*
C1	0.4823 (9)	0.3014 (10)	0.6406 (4)	0.060 (2)
H1C	0.461883	0.192512	0.627173	0.090*
H1D	0.470815	0.310042	0.684117	0.090*
H1E	0.588140	0.330831	0.633490	0.090*

Atomic displacement parameters ($\text{\AA}^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sn1	0.0243 (3)	0.0241 (3)	0.0401 (4)	0.000	0.0021 (3)	0.000
Br1	0.0573 (5)	0.0561 (6)	0.0440 (5)	−0.0174 (4)	0.0018 (4)	0.0028 (4)
Br2	0.0414 (4)	0.0440 (5)	0.0722 (6)	−0.0117 (3)	0.0061 (4)	−0.0013 (4)
O1	0.075 (4)	0.069 (4)	0.058 (4)	−0.003 (3)	0.026 (3)	−0.012 (3)
N1	0.062 (4)	0.052 (4)	0.063 (5)	−0.016 (3)	0.022 (4)	−0.014 (4)
C2	0.067 (5)	0.071 (6)	0.051 (5)	−0.008 (5)	−0.021 (4)	0.013 (5)
C1	0.056 (5)	0.055 (5)	0.067 (6)	0.014 (4)	−0.003 (4)	0.007 (5)

Geometric parameters ($\text{\AA}$, $^\circ$)

Sn1—Br1 ⁱ	2.9791 (8)	N1—H1B	0.80 (5)
Sn1—Br1	2.9790 (8)	N1—C1	1.493 (9)
Sn1—Br2 ⁱ	2.7144 (8)	C2—H2A	0.9600
Sn1—Br2	2.7143 (8)	C2—H2B	0.9600
Sn1—Br2 ⁱⁱ	3.2395 (8)	C2—H2C	0.9600
O1—N1	1.365 (8)	C1—H1C	0.9600
O1—C2	1.476 (9)	C1—H1D	0.9600
N1—H1A	0.80 (5)	C1—H1E	0.9600
Br1—Sn1—Br1 ⁱ	175.25 (4)	C1—N1—H1A	110.4

Sn1—Br2 ⁱⁱ —Sn1 ⁱⁱ	172.29 (3)	C1—N1—H1B	110.4
Br2—Sn1—Br1 ⁱ	89.19 (3)	O1—C2—H2A	109.5
Br2—Sn1—Br1	87.50 (2)	O1—C2—H2B	109.5
Br2 ⁱ —Sn1—Br2 ⁱⁱ	88.972 (6)	O1—C2—H2C	109.5
Br1—Sn1—Br2 ⁱⁱ	84.82 (2)	H2A—C2—H2B	109.5
Br2 ⁱ —Sn1—Br1	89.19 (3)	H2A—C2—H2C	109.5
Br2 ⁱ —Sn1—Br1 ⁱ	87.50 (2)	H2B—C2—H2C	109.5
Br2—Sn1—Br2 ⁱ	91.66 (4)	N1—C1—H1C	109.5
Br1 ⁱ —Sn1—Br2 ⁱⁱ	98.52 (2)	N1—C1—H1D	109.5
N1—O1—C2	108.8 (6)	N1—C1—H1E	109.5
O1—N1—H1A	110.4	H1C—C1—H1D	109.5
O1—N1—H1B	110.4	H1C—C1—H1E	109.5
O1—N1—C1	106.4 (6)	H1D—C1—H1E	109.5
H1A—N1—H1B	108.6		
C2—O1—N1—C1	179.7 (6)		

Symmetry codes: (i) $-x+1, y, -z+3/2$; (ii) $x+1/2, y+1/2, z$.

Hydrogen-bond geometry ($\text{\AA}$, $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1A $\cdots$ Br1 ⁱⁱⁱ	0.80	2.58	3.358 (6)	165
N1—H1B $\cdots$ Br1	0.80	2.58	3.358 (6)	165
C1—H1D $\cdots$ Br2	0.96	3.16	3.778 (9)	123
C2—H2B $\cdots$ Br1	0.96	3.04	3.818 (8)	139
C2—H2C $\cdots$ Br1 ⁱⁱⁱ	0.96	3.09	3.803 (9)	132

Symmetry code: (iii) $x-1/2, y-1/2, z$.