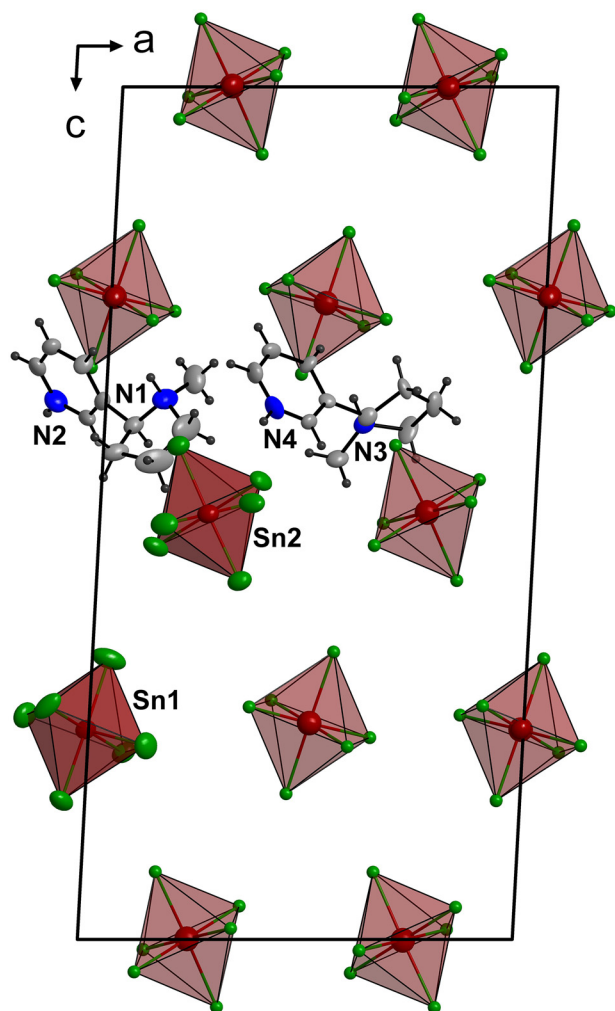


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# The pseudosymmetric crystal structure of 3-((1R,2S)-1-methylpyrrolidin-1-ium-2-yl)pyridin-1-ium hexachloridostannate(IV), $C_{10}H_{16}N_2SnCl_6$

**Table 1:** Data collection and handling.

|                                                                            |                                                               |
|----------------------------------------------------------------------------|---------------------------------------------------------------|
| Crystal:                                                                   | Colorless block                                               |
| Size:                                                                      | 0.31 × 0.13 × 0.07 mm                                         |
| Wavelength:                                                                | Mo K $\alpha$ radiation (0.71073 Å)                           |
| $\mu$ :                                                                    | 2.39 mm <sup>-1</sup>                                         |
| Diffractometer, scan mode:                                                 | Xcalibur, $\omega$                                            |
| $\theta_{\max}$ , completeness:                                            | 26.8°, >99%                                                   |
| $N(hkl)_{\text{measured}}$ , $N(hkl)_{\text{unique}}$ , $R_{\text{int}}$ : | 18,562, 7301, 0.044                                           |
| Criterion for $I_{\text{obs}}$ , $N(hkl)_{\text{gt}}$ :                    | $I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$ , 6213            |
| $N(\text{param})_{\text{refined}}$ :                                       | 345                                                           |
| Programs:                                                                  | CrysAlis <sup>PRO</sup> [1], SHELX [2–4], [5],<br>Diamond [6] |

**Abstract**

$C_{10}H_{16}N_2SnCl_6$ , monoclinic,  $I2$  (no. 5),  $a = 14.0477(4)$  Å,  $b = 8.9140(2)$  Å,  $c = 27.6199(6)$  Å,  $\beta = 93.060(2)^\circ$ ,  $Z = 8$ ,  $V = 3453.67(15)$  Å<sup>3</sup>,  $R_{\text{gt}}(F) = 0.0395$ ,  $wR_{\text{ref}} = 0.0858$ ,  $T = 290$  K.

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A packing diagram of the title crystal structure is shown in the figure with view along  $[0\bar{1}0]$ . This figure contains the asymmetric unit shown as displacement ellipsoids and additional  $[SnCl_6]^{2-}$  anions documenting a layered stacking of the cationic and anionic layers in the structure along  $[001]$ . Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

**Source of material**

All chemicals were obtained from commercial sources and used as purchased.

In a representative experiment 0.437 g  $SnCl_4$  (1.24 mmol; Riedel-de Haen) were dissolved in 1 ml concentrated hydrochloric acid (37%, Sigma–Aldrich). To this mixture 0.2 ml (0.202 g; 1.24 mmol) *S*-nicotine (Acros Organics; systematic name: *S*-3-[1-methylpyrrolidin-2-yl]pyridine) was added. In the next step additional hot concentrated hydrochloric acid was added until the precipitated solids are dissolved. For instant crystallization the solution was manipulated with a

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**Table 2:** Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å<sup>2</sup>).

| Atom | <i>x</i>     | <i>y</i>    | <i>z</i>     | <i>U</i> <sub>iso</sub> <sup>*</sup> / <i>U</i> <sub>eq</sub> |
|------|--------------|-------------|--------------|---------------------------------------------------------------|
| Sn1  | −0.00653 (6) | 0.01949 (9) | 0.75381 (3)  | 0.01665 (14)                                                  |
| Sn2  | 0.24846 (6)  | 0.27716 (9) | 0.50087 (3)  | 0.01657 (14)                                                  |
| Cl1  | −0.0540 (2)  | 0.0570 (3)  | 0.83703 (9)  | 0.0232 (6)                                                    |
| Cl2  | 0.08369 (18) | −0.2034 (3) | 0.78080 (8)  | 0.0184 (5)                                                    |
| Cl3  | 0.1336 (2)   | 0.1710 (3)  | 0.77285 (10) | 0.0257 (6)                                                    |
| Cl4  | −0.1475 (2)  | −0.1351 (3) | 0.73679 (10) | 0.0263 (6)                                                    |
| Cl5  | −0.0969 (2)  | 0.2395 (3)  | 0.72606 (9)  | 0.0254 (6)                                                    |
| Cl6  | 0.0380 (2)   | −0.0159 (3) | 0.67088 (9)  | 0.0316 (7)                                                    |
| Cl7  | 0.1491 (2)   | 0.4960 (3)  | 0.51294 (9)  | 0.0247 (6)                                                    |
| Cl8  | 0.1319 (2)   | 0.1147 (3)  | 0.53921 (9)  | 0.0211 (6)                                                    |
| Cl9  | 0.3290 (2)   | 0.3230 (3)  | 0.57950 (9)  | 0.0209 (6)                                                    |
| Cl10 | 0.17165 (19) | 0.2361 (3)  | 0.42098 (9)  | 0.0199 (6)                                                    |
| Cl11 | 0.36559 (19) | 0.4381 (3)  | 0.46222 (9)  | 0.0193 (6)                                                    |
| Cl12 | 0.3476 (2)   | 0.0556 (3)  | 0.48830 (9)  | 0.0237 (6)                                                    |
| N1   | 0.1352 (6)   | 0.6924 (10) | 0.3662 (3)   | 0.033 (2)                                                     |
| H1   | 0.099369     | 0.749257    | 0.340861     | 0.039*                                                        |
| N2   | −0.1152 (7)  | 0.3285 (10) | 0.3684 (3)   | 0.028 (2)                                                     |
| H2   | −0.133300    | 0.247836    | 0.382316     | 0.033*                                                        |
| N3   | 0.5965 (6)   | 0.7806 (8)  | 0.3951 (3)   | 0.0231 (18)                                                   |
| H3   | 0.568927     | 0.732869    | 0.365633     | 0.028*                                                        |
| N4   | 0.3882 (7)   | 1.1919 (10) | 0.3762 (3)   | 0.025 (2)                                                     |
| H4   | 0.365196     | 1.264961    | 0.392203     | 0.030*                                                        |
| C1   | 0.1870 (12)  | 0.803 (2)   | 0.4004 (5)   | 0.062 (5)                                                     |
| H1A  | 0.244586     | 0.757805    | 0.414929     | 0.074*                                                        |
| H1B  | 0.204268     | 0.892561    | 0.383086     | 0.074*                                                        |
| C2   | 0.1183 (11)  | 0.8406 (14) | 0.4390 (5)   | 0.060 (4)                                                     |
| H2A  | 0.102276     | 0.946367    | 0.437803     | 0.072*                                                        |
| H2B  | 0.146729     | 0.817800    | 0.470967     | 0.072*                                                        |
| C3   | 0.0284 (9)   | 0.7444 (11) | 0.4285 (4)   | 0.033 (3)                                                     |
| H3A  | −0.021216    | 0.801231    | 0.411042     | 0.039*                                                        |
| H3B  | 0.003914     | 0.705691    | 0.458242     | 0.039*                                                        |
| C4   | 0.0663 (8)   | 0.6161 (12) | 0.3968 (4)   | 0.027 (3)                                                     |
| H4A  | 0.101755     | 0.545552    | 0.418210     | 0.032*                                                        |
| C5   | −0.0086 (7)  | 0.5307 (13) | 0.3685 (4)   | 0.023 (2)                                                     |
| C6   | −0.0414 (9)  | 0.4023 (14) | 0.3882 (4)   | 0.027 (3)                                                     |
| H6   | −0.011029    | 0.365223    | 0.416434     | 0.033*                                                        |
| C7   | −0.0566 (8)  | 0.5786 (12) | 0.3256 (4)   | 0.024 (3)                                                     |
| H7   | −0.035991    | 0.665383    | 0.310688     | 0.029*                                                        |
| C8   | −0.1324 (8)  | 0.5025 (15) | 0.3050 (4)   | 0.027 (2)                                                     |
| H8   | −0.163309    | 0.535682    | 0.276342     | 0.032*                                                        |
| C9   | −0.1624 (9)  | 0.3723 (14) | 0.3284 (4)   | 0.029 (3)                                                     |
| H9   | −0.214674    | 0.317998    | 0.315898     | 0.035*                                                        |
| C10  | 0.2006 (9)   | 0.5910 (15) | 0.3430 (5)   | 0.045 (4)                                                     |
| H10A | 0.164888     | 0.522523    | 0.322255     | 0.067*                                                        |
| H10B | 0.236880     | 0.535587    | 0.367418     | 0.067*                                                        |
| H10C | 0.243079     | 0.648172    | 0.324096     | 0.067*                                                        |
| C11  | 0.6987 (9)   | 0.7324 (14) | 0.4024 (5)   | 0.037 (3)                                                     |
| H11A | 0.706365     | 0.629582    | 0.391700     | 0.044*                                                        |
| H11B | 0.719783     | 0.739310    | 0.436301     | 0.044*                                                        |
| C12  | 0.7551 (7)   | 0.8376 (11) | 0.3723 (4)   | 0.031 (3)                                                     |
| H12A | 0.780808     | 0.784583    | 0.345220     | 0.037*                                                        |
| H12B | 0.807577     | 0.881156    | 0.391769     | 0.037*                                                        |
| C13  | 0.6851 (7)   | 0.9610 (11) | 0.3539 (4)   | 0.021 (2)                                                     |
| H13A | 0.714282     | 1.059379    | 0.356729     | 0.025*                                                        |
| H13B | 0.664249     | 0.943856    | 0.320260     | 0.025*                                                        |

**Table 2:** (continued)

| Atom | <i>x</i>   | <i>y</i>    | <i>z</i>   | <i>U</i> <sub>iso</sub> <sup>*</sup> / <i>U</i> <sub>eq</sub> |
|------|------------|-------------|------------|---------------------------------------------------------------|
| C14  | 0.6031 (8) | 0.9475 (11) | 0.3864 (4) | 0.023 (2)                                                     |
| H14  | 0.621425   | 0.996212    | 0.417276   | 0.027*                                                        |
| C15  | 0.5085 (7) | 1.0113 (12) | 0.3680 (3) | 0.017 (2)                                                     |
| C16  | 0.4692 (8) | 1.1317 (12) | 0.3929 (4) | 0.019 (2)                                                     |
| H16  | 0.500153   | 1.168934    | 0.420975   | 0.023*                                                        |
| C17  | 0.4606 (8) | 0.9630 (12) | 0.3255 (4) | 0.022 (2)                                                     |
| H17  | 0.485622   | 0.885839    | 0.307418   | 0.026*                                                        |
| C18  | 0.3738 (8) | 1.0318 (14) | 0.3100 (3) | 0.025 (2)                                                     |
| H18  | 0.340635   | 0.998178    | 0.282004   | 0.030*                                                        |
| C19  | 0.3389 (8) | 1.1458 (12) | 0.3355 (4) | 0.026 (3)                                                     |
| H19  | 0.281914   | 1.192126    | 0.325324   | 0.031*                                                        |
| C20  | 0.5386 (8) | 0.7378 (12) | 0.4364 (3) | 0.030 (3)                                                     |
| H20A | 0.542115   | 0.631202    | 0.441143   | 0.045*                                                        |
| H20B | 0.473499   | 0.766648    | 0.429470   | 0.045*                                                        |
| H20C | 0.562923   | 0.787848    | 0.465245   | 0.045*                                                        |

glass pipette. The title compound decomposes as 270 °C (monitored by differential scanning calorimetry [DSC]).

## Experimental details

A crystal of the title compound was directly selected from the mother liquor and mounted on a Xcalibur four-circle diffractometer equipped with the EOS detector [1]. An absorption correction was applied [1]. The structure solution and the refinement were successfully carried out using the SHELX program system [2, 3, 5]. Hydrogen atoms were finally included in the refinement using standard riding models implemented in the SHELXL software package [2, 3]. The maximum residual peak of 0.79 e Å<sup>−3</sup> is located near Cl2 (0.70 Å) and the deepest hole is −0.72 e Å<sup>−3</sup>, which is 0.70 Å apart from Sn2. The figure was created using the Diamond software [6].

## Comment

### Introduction

A recently performed survey for nicotine-containing crystal structures in the Cambridge Structural Database [7] gave approximately 50 hits. As discussed before, structural data are available for (a) metal complexes, which contain neutral nicotine ligands; (b) a small number of co-crystals containing neutral nicotine as one of the components; (c) some examples for pyrrolidiny-monoprotonated nicotinium salts; and (d) mono-protonated nicotinium as a cationic ligand [8]. In detail only a limited number of

examples of salt structures containing doubly protonated nicotinium cations  $NicH_2$  have been reported so far [8]. This contribution is part of our continuing interest in synthesis, characterization and understanding of hydrogen-bonding as well as packing schemes of nicotinium salts [8–10]. The  $[SnCl_6]^{2-}$  counterion used for this study is a bulky, medium-strong hydrogen-bond acceptor, which is often used by us [11] and many other groups [12–15]. This contribution is also part of our longstanding interest in the structures and hydrogen-bonding schemes of hexahalo-genidometallates, their packing schemes and associated pseudosymmetry problems [11, 16–18].

## Structural comments

The title structure crystallizes in the non-centrosymmetric space group  $I2$  (no. 5). There are two doubly protonated nicotiniums cations (systematic name: 3-((1*R*,2*S*)-1-methylpyrrolidin-1-ium-2-yl)pyridin-1-ium;  $NicH_2$ ) and two  $[SnCl_6]^{2-}$  anions in the asymmetric unit (see the figure). All ions are in general positions. Bond lengths and angles within the two crystallographically independent  $NicH_2$  cations are all in the expected ranges [8–10]. The same is true for the  $[SnCl_6]^{2-}$  anion [11–15]. In detail, the protonation at the nitrogen of the pyrrolidiny moiety creates a chiral center with the descriptor *R* according to the Cahn–Ingold–Prelog priority rules. This finding came as no surprise, as all quality structure determinations containing  $NicH_2$  by now show this configuration. Thus the title structure is another example, which pronounces the preference of the enantiomeric protonation of the pyrrolidiny moiety [8–10]. The Sn–Cl distances in the  $[SnCl_6]^{2-}$  anions range from 2.421(3)–2.469(2) Å and the *cis*-angles are in the range of 89.14(10)–91.73(10)°. In detail there is an internal correspondence between the weak hydrogen bonds accepted by the  $[SnCl_6]^{2-}$  anions and the Sn–Cl distances. For example the Sn2–Cl8 of 2.466(2) Å corresponds to the shortest  $NH\cdots Cl$  hydrogen bonds ( $N\cdots Cl$ : 3.204(9) Å).

## Group-subgroup relation and pseudosymmetry

The title structure shows an almost centrosymmetric arrangement of the  $[SnCl_6]^{2-}$  anions. We have shown several times that the presence of pseudosymmetry in a structure requires special procedures of data collection and structure refinement [16, 17, 19–21]. Thus we refined the diffraction data suggesting the monoclinic space group  $I2/a$  (no. 15), even though it is quite clear that this space group is not allowed, as

we used the pure enantimorphic *S*-nicotine as the starting material.

The test refinement in the centrosymmetric space group  $I2/a$  (no. 15) showed that the asymmetric unit contains one cation in general position and two halves of  $[SnCl_6]^{2-}$  anions located on two inversion centers (0, 0, 0; and 0.25, 0.25, 0.75). This finding came as no surprise, as in related cases [16, 22], the hexahalogenidometallate anions reside on two different inversion centers to produce the well known stacking of alternating hexahalogenidometallate layers. In the title structure the aforementioned layer are stacked along the crystallographic *c* direction. These layers are characterized by the fact that the octahedra of neighboring layers are tilted towards each other (see the figure). It was more surprising that the pyridylum moiety of the cation also shows a centrosymmetric arrangement.

Thus the true non-centrosymmetric structure (space group  $I2$ ) is the sub group of the hypothetical centrosymmetric structure in the space group  $I2/a$ . They are connected by a *translationengleiche* group-subgroup relation with the index 2 (Origin shift: 0, 0, 0.25; see Table 2) [23, 24].

The pseudosymmetry problem in this structure of cause affects the intensity distribution of the reciprocal lattice. Consequently reflections that would be absent in the case of an *a*-glide plane ( $h0l$ :  $h, l = 2n - 1$ ) are much weaker than general reflections. Furthermore the calculated Flack parameters are near zero, but the relatively large standard uncertainties are results of pseudo-symmetrically arrangements of most atoms in the structure (Flack parameter: 0.08(4) by classical fit to all intensities; 0.05(2) from 2471 selected quotients (Parsons' method) [5]).

## Packing

As mentioned before the  $[SnCl_6]^{2-}$  anions are stacked along the crystallographic *c* direction. The  $NicH_2$  cations are located between these layers (see the figure). Each anionic layer consists of only one of the two crystallographically independent  $[SnCl_6]^{2-}$  anions (Sn1 at *c* = 0.25; 0.75; Sn2 at *c* = 0, 0.5; see the figure). The octahedra within both layers are tilted to each other as a consequence of the space group symmetry. The connections between cations and anions are furnished by weak  $NH\cdots Cl$  hydrogen bonds. Consequently the title structure can be attributed as layered with separated cationic and anionic layers.

## The Raman spectroscopy

Raman spectra were recorded using a Bruker MultiRAM FT-Raman spectrometer (apodized resolution: 4  $cm^{-1}$ ). The

typical signals [25–27] for the  $[SnCl_6]^{2-}$  anions are found at  $310\text{ cm}^{-1}$  (very strong,  $\nu_1$ );  $238\text{ cm}^{-1}$  (medium,  $\nu_2$ );  $163\text{ cm}^{-1}$  (strong,  $\nu_4/\nu_5$ );  $115\text{ cm}^{-1}$  (strong), respectively.

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