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## **A monoclinic modification of $K_3[InCl_6]$**

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A monoclinic modification of  $K_3[InCl_6]$ Lin Chen,<sup>a\*</sup> Bo-Lin Wu,<sup>a</sup> Xiao-Yi He<sup>a</sup> and Jin-Xiao Mi<sup>b</sup>

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## Key indicators

Single-crystal X-ray study  
 $T = 295$  K  
 Mean  $\sigma(n-CI) = 0.001$  Å  
 $R$  factor = 0.031  
 $wR$  factor = 0.093  
 Data-to-parameter ratio = 27.1

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The crystal structure of monoclinic tripotassium indium(III) hexachloride,  $K_3[InCl_6]$ , obtained by the solvent evaporation method, has been determined from single-crystal X-ray diffraction data. The crystal structure is characterized by isolated  $[InCl_6]$  octahedra located in the centre of the cell and at the centre of each of the edges of the cell, linked with  $K^+$  cations to form a three-dimensional structure.

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## Comment

In the anhydrous potassium indium chloride system, four modifications of  $K_3[InCl_6]$  have previously been reported from powder diffraction data (Atkinson *et al.*, 1968; Wignacourt, 1981). It is interesting that all known modifications of  $K_3[InCl_6]$  polymorphs belong to the tetragonal system. To our knowledge, the title compound reported here is the first monoclinic modification in the polymorph series. The new modification of  $K_3[InCl_6]$  was synthesized as an intermediate in an investigation of the influence of  $K^+$  on the formation and particle size of indium tin oxide (ITO) nanopowders.

The crystal structure of the title compound is characterized by isolated  $[InCl_6]$  octahedra, linked with  $K^+$  cations to form a three-dimensional structure. Isolated  $[InCl_6]$  octahedra are located in the centre of the cell and at the centre of each of the edges of the cell (Fig. 1). Large cations K1, K2, K3 are eight-

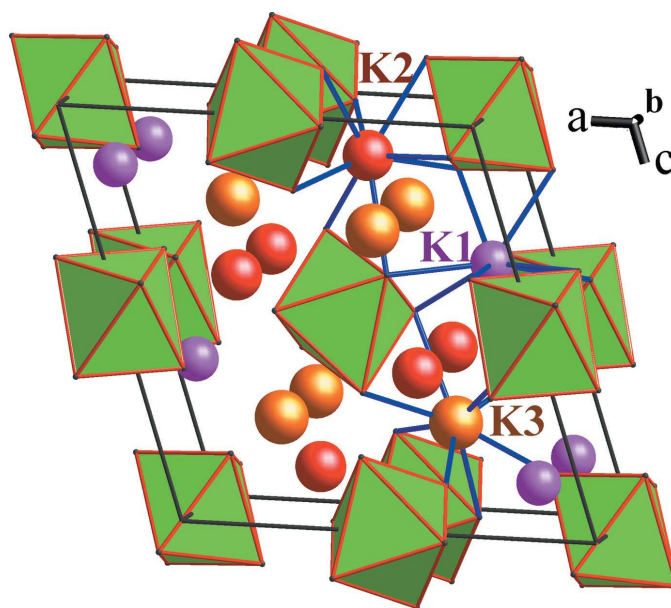
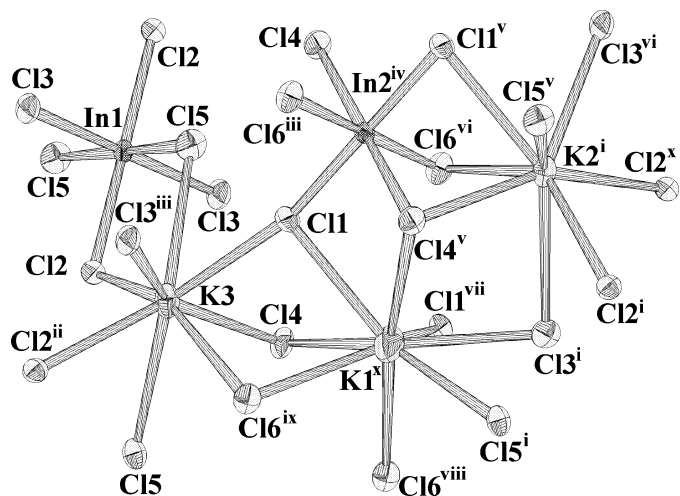


Figure 1

The crystal structure of  $K_3[InCl_6]$ . The  $[InCl_6]$  octahedra are shown in a polyhedral representation.



**Figure 2**

The coordination environment of the metal atoms in  $K_3[InCl_6]$ , with displacement ellipsoids drawn at the 50% probability level. Symmetry codes: (i)  $x - 1, -y + \frac{3}{2}, z - \frac{1}{2}$ ; (ii)  $x, -y + \frac{3}{2}, z - \frac{1}{2}$ ; (iii)  $-x + 1, -y + 2, -z + 1$ ; (iv)  $x, y + 1, z$ ; (v)  $x - 1, y + 1, z$ ; (vi)  $x - 1, y, z$ ; (vii)  $x - 1, -y + \frac{1}{2}, z - \frac{1}{2}$ ; (viii)  $x - 1, -y + \frac{3}{2}, -z + \frac{1}{2}$ ; (ix)  $x + 1, y + \frac{1}{2}, -z + \frac{1}{2}$ ; (x)  $x, -y + 1, -z + 1$ .

coordinate with respect to nearby ( $d < 3.70$  Å) Cl atoms, with mean K—Cl distances of 3.386, 3.224 and 3.251 Å, respectively (Fig. 2). Cations In1 and In2 are coordinated octahedrally by six nearby Cl atoms and the  $[InCl_6]$  octahedra are quite regular.

## Experimental

The title compound was synthesized by the solvent evaporation method. The reaction solution was prepared by mixing  $InCl_3$  (analytical grade),  $K_2CO_3$  (analytical grade) and hydrochloric acid (analytical grade) in the molar ratio  $InCl_3:K_2CO_3:HCl = 2:3:6$ . Colourless transparent crystals were obtained by drying the reaction solution in a constant temperature oven at 325 K for several days. Crystal growth was affected by crystallization of KCl and the amount of  $K_2CO_3$ . Crystals of the title compound are sensitive to moisture and change to powder in several days in air. The powder was proved to consist of  $K_2[InCl_5(H_2O)]$  (Wignacourt *et al.*, 1976) and the tetragonal modification of  $K_3[InCl_6]$  (Atkinson *et al.*, 1968) by X-ray powder diffraction data. The chemical composition of the single crystal was confirmed by a chemical semi-quantitative energy-dispersive X-ray analysis.

### Crystal data

$K_3[InCl_6]$   
 $M_r = 444.82$   
 Monoclinic,  $P2_1/c$   
 $a = 12.188$  (3) Å  
 $b = 7.5530$  (17) Å  
 $c = 12.703$  (3) Å  
 $\beta = 108.957$  (4)°  
 $V = 1106.0$  (5) Å<sup>3</sup>

$Z = 4$   
 $D_x = 2.671$  Mg m<sup>-3</sup>  
 Mo  $K\alpha$  radiation  
 $\mu = 4.65$  mm<sup>-1</sup>  
 $T = 295$  (2) K  
 Plate, colourless  
 $0.50 \times 0.28 \times 0.09$  mm

### Data collection

Bruker SMART CCD area-detector diffractometer  
 $\omega$  scans  
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)  
 $T_{min} = 0.199, T_{max} = 0.659$   
 6419 measured reflections  
 2548 independent reflections  
 2341 reflections with  $I > 2\sigma(I)$   
 $R_{int} = 0.023$   
 $\theta_{max} = 28.0^\circ$

### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.031$   
 $wR(F^2) = 0.093$   
 $S = 1.12$   
 2548 reflections  
 94 parameters

$w = 1/[\sigma^2(F_o^2) + (0.0527P)^2 + 0.923P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{max} < 0.001$   
 $\Delta\rho_{max} = 0.92$  e Å<sup>-3</sup>  
 $\Delta\rho_{min} = -1.20$  e Å<sup>-3</sup>

**Table 1**

Selected bond lengths (Å).

In1—Cl5	2.4096 (9)	K2—Cl6 <sup>vii</sup>	3.0556 (13)
In1—Cl3	2.4695 (9)	K2—Cl4 <sup>viii</sup>	3.0912 (13)
In1—Cl2	2.6005 (10)	K2—Cl5 <sup>viii</sup>	3.1374 (13)
In2—Cl1 <sup>i</sup>	2.4595 (10)	K2—Cl2	3.1511 (12)
In2—Cl4 <sup>ii</sup>	2.4806 (9)	K2—Cl1 <sup>viii</sup>	3.3151 (15)
In2—Cl6 <sup>iii</sup>	2.5463 (9)	K2—Cl3 <sup>vii</sup>	3.3636 (14)
K1—Cl3 <sup>iv</sup>	3.2518 (16)	K2—Cl3	3.6261 (15)
K1—Cl6 <sup>v</sup>	3.2540 (16)	K3—Cl4 <sup>iii</sup>	2.9279 (12)
K1—Cl4 <sup>ii</sup>	3.2577 (16)	K3—Cl3 <sup>iv</sup>	3.0954 (13)
K1—Cl1 <sup>i</sup>	3.2694 (17)	K3—Cl2	3.1063 (12)
K1—Cl6 <sup>iv</sup>	3.2898 (16)	K3—Cl2 <sup>vi</sup>	3.1299 (13)
K1—Cl5 <sup>iv</sup>	3.4642 (18)	K3—Cl1 <sup>vi</sup>	3.1991 (13)
K1—Cl4 <sup>v</sup>	3.6101 (17)	K3—Cl5 <sup>vi</sup>	3.4552 (15)
K1—Cl1 <sup>vi</sup>	3.6918 (19)	K3—Cl6 <sup>iii</sup>	3.4916 (13)
K2—Cl2 <sup>vi</sup>	3.0516 (12)	K3—Cl5 <sup>iii</sup>	3.6022 (16)

Symmetry codes: (i)  $x - 1, y, z$ ; (ii)  $-x + 1, -y, -z + 1$ ; (iii)  $-x + 1, -y + 1, -z + 1$ ; (iv)  $-x + 1, y - \frac{1}{2}, -z + \frac{3}{2}$ ; (v)  $x - 1, -y + \frac{1}{2}, z + \frac{3}{2}$ ; (vi)  $-x + 1, y + \frac{1}{2}, -z + \frac{3}{2}$ ; (vii)  $x, -y + \frac{3}{2}, z + \frac{1}{2}$ ; (viii)  $x, -y + \frac{1}{2}, z + \frac{1}{2}$ .

The deepest hole is 0.78 Å from atom In1.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 2004); software used to prepare material for publication: *SHELXL97*.

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