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A monoclinic modification of $K_3[InCl_6]$

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(n–Cl) = 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.

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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.

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-

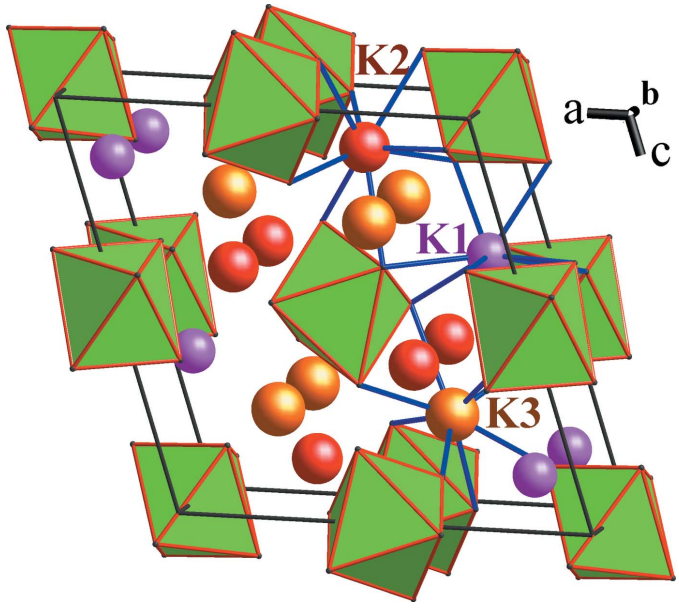

Figure 1
The crystal structure of $K_3[InCl_6]$. The $[InCl_6]$ octahedra are shown in a polyhedral representation.

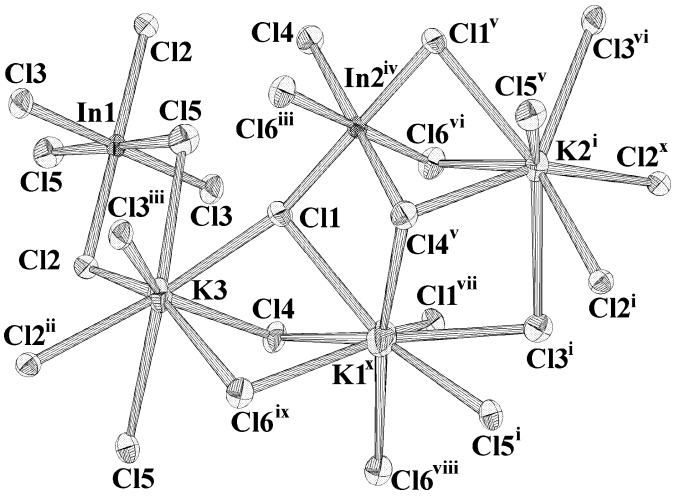

Figure 2
The coordination environment of the metal atoms in $K_3[InCl_6]$, with displacement ellipsoids drawn at the 50% probability level. Symmetry codes: (i) $x-1, -y+\frac{3}{2}, z-\frac{1}{2}$; (ii) $x, -y+\frac{3}{2}, z-\frac{1}{2}$; (iii) $-x+1, -y+2, -z+1$; (iv) $x, y+1, z$; (v) $x-1, y+1, z$; (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
β = 108.957 (4)°
V = 1106.0 (5) Å^3
Z = 4
D_x = 2.671 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 4.65 mm^{-1}
T = 295 (2) K
Plate, colourless
0.50 × 0.28 × 0.09 mm

Data collection

Bruker SMART CCD area-detector diffractometer
ω 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
θ_{max} = 28.0°

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^{-3}
$\Delta\rho_{min}$ = −1.20 e Å^{-3}

Table 1
Selected bond lengths (Å).

In1—Cl5	2.4096 (9)	K2—Cl6^{vii}	3.0556 (13)
In1—Cl3	2.4695 (9)	K2—Cl4^{viii}	3.0912 (13)
In1—Cl2	2.6005 (10)	K2—Cl5^{viii}	3.1374 (13)
In2—Cl1^{i}	2.4595 (10)	K2—Cl2	3.1511 (12)
In2—Cl4^{ii}	2.4806 (9)	K2—Cl1^{viii}	3.3151 (15)
In2—Cl6^{iii}	2.5463 (9)	K2—Cl3^{vii}	3.3636 (14)
K1—Cl3^{iv}	3.2518 (16)	K2—Cl3	3.6261 (15)
K1—Cl6^{v}	3.2540 (16)	K3—Cl4^{iii}	2.9279 (12)
K1—Cl4^{ii}	3.2577 (16)	K3—Cl3^{iv}	3.0954 (13)
K1—Cl1^{i}	3.2694 (17)	K3—Cl2	3.1063 (12)
K1—Cl6^{iv}	3.2898 (16)	K3—Cl2^{vi}	3.1299 (13)
K1—Cl5^{iv}	3.4642 (18)	K3—Cl1^{vi}	3.1991 (13)
K1—Cl4^{v}	3.6101 (17)	K3—Cl5^{vi}	3.4552 (15)
K1—Cl1^{vi}	3.6918 (19)	K3—Cl6^{iii}	3.4916 (13)
K2—Cl2^{vi}	3.0516 (12)	K3—Cl5^{iii}	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) $x-1, -y+\frac{1}{2}, z+\frac{1}{2}$; (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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