Tabelle 1. *Atomkoordinaten und isotrope Temperaturfaktoren* (×10²) *für* $Sn_5F_9BF_4$

$$U_{äq} = \tfrac{1}{3}\textstyle\sum_i\sum_j U_{ij}a^*_i a^*_j \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{äq}$(Å²)
Sn(1)	0,5518 (2)	0,4005 (3)	0,3457 (2)	2,64 (8)
Sn(2)	0,3725 (2)	0,0960 (3)	0,4860 (2)	2,44 (8)
Sn(3)	0,7283 (2)	0,0882 (2)	0,1812 (2)	2,07 (7)
Sn(4)	0,8788 (2)	0,3339 (3)	0,9977 (2)	2,21 (8)
Sn(5)	0,2083 (2)	0,1227 (3)	0,1779 (2)	2,34 (8)
F(1)	0,198 (2)	0,499 (3)	0,130 (1)	3,1 (6)
F(2)	0,308 (2)	0,318 (2)	0,318 (2)	2,9 (5)
F(3)	0,360 (1)	0,126 (2)	0,176 (2)	2,7 (5)
F(4)	0,376 (2)	0,523 (3)	0,497 (1)	3,5 (6)
F(5)	0,520 (1)	0,134 (2)	0,460 (2)	3,4 (6)
F(6)	0,571 (1)	0,100 (3)	0,177 (2)	2,9 (5)
F(7)	0,694 (1)	0,484 (2)	0,170 (1)	2,8 (5)
F(8)	0,738 (2)	0,359 (3)	0,011 (2)	3,9 (7)
F(9)	0,772 (2)	0,348 (2)	0,379 (2)	3,6 (6)
F(10)	0,117 (3)	0,452 (5)	0,426 (3)	9,1 (14)
F(11)	0,123 (3)	0,171 (5)	0,410 (4)	12,1 (18)
F(12)	0,961 (4)	0,295 (9)	0,340 (6)	14,5 (20)
F(13)	0,057 (5)	0,328 (8)	0,265 (3)	12,1 (18)
B	0,067 (3)	0,303 (6)	0,363 (4)	4,8 (13)

Tabelle 2. Sn—F- *und* B—F-*Abstände* (Å) *im* $Sn_5F_9BF_4$ (*Standardabweichung bei* Sn—F-*Abständen* 0,02 Å)

Sn(1)—F(3)	2,21	Sn(4)—F(1)	2,02
—F(4)	2,01	—F(8)	2,03
—F(6)	2,17	—F(9)	2,11
Sn(2)—F(5)	2,18	Sn(3)—F(1)	2,44
—F(5′)	2,23	—F(2)	2,13
—F(7)	2,11	—F(4)	2,25
—F(8)	2,37	—F(6)	2,11
Sn(5)—F(2)	2,31	B—F(10)	1,39 (6)
—F(3)	2,09	—F(11)	1,24 (6)
—F(7)	2,16	—F(12)	1,32 (7)
—F(9)	2,31	—F(13)	1,34 (8)

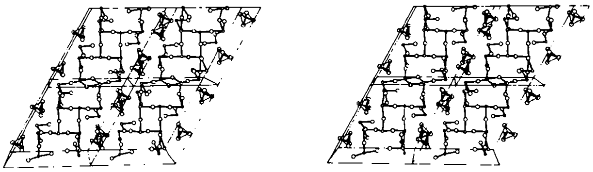

Fig. 1. Stereobild des $Sn_5F_9BF_4$ mit vier Elementarzellen in Blickrichtung [010]. (Kleine Kreise: Sn, grosse Kreise: F; die F-Atome der BF_4-Gruppen sind zu Tetraedern verbunden.)

vierseitigen Pyramiden und den Abständen der Tabelle 2 liegen alle Werte im Rahmen des üblichen (Brown, 1974). Die BF_4-Gruppen liegen zwischen den Schichten. Ihre Verzerrung (Tabelle 2) dürfte jedoch auf Grund der grossen Standardabweichung der Atomabstände und der hohen Temperaturfaktoren nur eine scheinbare sein.

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Potassium Tetrabromoindate(III), K[InBr$_4$]

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Abstract. $M_r = 473{\cdot}5$, orthorhombic, $Pnna$, $a = 7{\cdot}891$ (12), $b = 10{\cdot}390$ (5), $c = 10{\cdot}390$ (5) Å, $V = 851{\cdot}9$ Å³, $Z = 4$, $D_m = 3{\cdot}63$, $D_x = 3{\cdot}69$ g cm^{-3}, λ(Mo $K\bar{\alpha}$) = 0·7107 Å, $\mu = 2{\cdot}29$ cm^{-1}, $F(000) = 832$, room temperature, $R = 0{\cdot}052$ for 730 unique observed reflexions. This is the first alkaline halo-indate in which the In atom has a tetrahedral coordination, involving four Br atoms at distances 2·494 (2) and 2·520 (2) Å, and Br—In—Br angles ranging from 105·1 (1) to 113·2 (2)°. The K^+ ion is surrounded by eight Br^- ions with an average distance of 3·428 (3) Å.

Introduction. A previous investigation (Wignacourt, Mairesse, Barbier, Lorriaux-Rubbens & Wallart, 1982) of the indium tribromide–potassium bromide–water system at 293 K has shown the existence of the following hydrated double salts: $K_3[InBr_6].1{\cdot}5H_2O$, $K_2[InBr_5(H_2O)]$ and $K[InBr_4(H_2O)_2]$, all characterized by complete structural studies (Wignacourt, 1981). The existence of a non-hydrated salt has been pointed out, which was supposed to be formulated $K_3[In_2Br_9]$; the obtention of single crystals in the related diagram area enabled a structural investigation to be performed,

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proving the existence of a new compound K[$InBr_4$], not even given in the binary diagram $InBr_3$–KBr (Fedorov & Il'ina, 1964).

Experimental. Preparation by evaporation of $InBr_3$–KBr aqueous solutions in an appropriate ratio (Wignacourt, 1983). D_m measured pycnometrically using CCl_4 as reference. Needle-shaped crystal inside capillary filled with Nujol, preventing any precise absorption correction. Unit-cell dimensions refined from 25 reflexions ($5 < \theta < 7°$). Philips PW1100 diffractometer. 2802 reflexions with $2 < \theta < 30°$, $0 < h < 10$, $0 < k < 14$, $-14 < l < 14$, 1362 with $I > 3\sigma(I)$. No fourfold axis in spite of $b = c$; 730 unique reflexions, $R_{\text{int}} = 0{\cdot}035$. Three standards (210, $0\bar{2}\bar{2}$, $\bar{2}\bar{1}0$) stable throughout data collection. In atoms located from Patterson map, Br and K identified on successive Fourier syntheses. Least-squares refinement of F magnitudes with isotropic, then anisotropic temperature factors. Final $R = 0{\cdot}052$, $R_w = 0{\cdot}058$; $w = 1/\sigma^2(F_o)$. Max. Δ/σ in final refinement cycle: 0·01. Heights in final difference Fourier synthesis: max.: 0·8 e Å^{-3}, min.: −0·8 e Å^{-3}. Atomic scattering factors from Cromer & Waber (1965), anomalous-dispersion correction from Cromer & Liberman (1970). Local version of *ORFLS* program (Busing, Martin & Levy, 1962).

Discussion. Atomic positions are given in Table 1,* characteristic bond lengths and angles in Table 2. The anionic configuration and the molecular packing are shown in Figure 1.

The tetrahedral anion [$InBr_4$]$^-$ is characterized for the first time in the solid state.

The K^+ ion is surrounded by eight Br^- ions, provided by six [$InBr_4$]$^-$ ions. In the [$InBr_4$]$^-$ ion, two Br^- ions interact with only one K^+ ion, while the two others interact with two different K^+ ions.

In the literature, the only available information about In—Br bonds is from octahedral indium clusters: 2·686 Å (Wignacourt, 1981) or 2·670 Å (Khan & Tuck, 1981) in [$InBr_6$]$^{3-}$; 2·629 Å (Wignacourt, 1981) in [$InBr_5(H_2O)$]$^{2-}$; 2·613 Å (Wignacourt, Mairesse & Barbier, 1980) in [$InBr_4(H_2O)_2$]$^-$. In going from the octahedral [$InBr_6$]$^{3-}$ to the tetrahedral [$InBr_4$]$^-$, the shortening of the In—Br bond length is 0·19 Å. It is of about the same order as the 0·20 Å noted earlier for In—Cl bonds (Wignacourt, 1981; Contreras, Einstein, Gilbert & Tuck, 1977).

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* (Å^2)

$$B_{\text{eq}} = \tfrac{4}{3}\textstyle\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
In	$\frac{1}{4}$	0	1808 (1)	2·35 (8)
Br(1)	3428 (2)	1875 (2)	486 (2)	3·80 (10)
Br(2)	−18 (2)	544 (1)	3197 (2)	3·35 (10)
K	6792 (6)	$\frac{1}{4}$	$\frac{1}{4}$	3·40 (20)

Table 2. *Characteristic bond lengths* (Å) *and angles* (°)

In—Br(1)	2·494 (2)	In—Br(2)	2·520 (2)
Br(1)—In—Br(1)	113·2 (2)	Br(1)—In—Br(2)	105·1 (1)
Br(2)—In—Br(2)	110·0 (1)		111·8 (1)
K—Br(1^{i})	3·442 (4)	K—Br(2^{vi})	3·315 (4)
K—Br(1^{iii})	3·422 (3)	K—Br(2^{ii})	3·534 (2)
K—Br(1^{iv})	3·442 (4)	K—Br(2^{vii})	3·315 (4)
K—Br(1^{v})	3·422 (3)	K—Br(2^{viii})	3·534 (2)

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

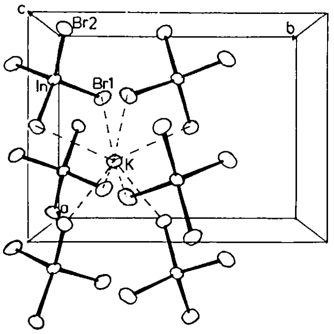

Fig. 1. Molecular packing in K[$InBr_4$].

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39719 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.