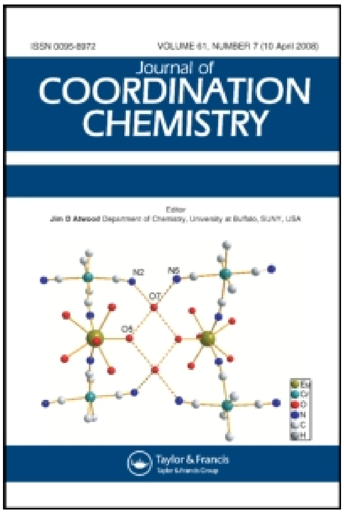

Journal of Coordination Chemistry
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713455674

The Crystal structures of tetra-*n*-Butylammonium salts of $GaCl_4^-$, $GaBr_4^-$ and GaI_4^-

K. Rudawska[a]; H. Ptasiewicz-Bąk[a]
[a] Institute of Nuclear Chemistry and Technology, 03-195 Warszawa, Poland

To cite this Article Rudawska, K. and Ptasiewicz-Bąk, H.(2003) 'The Crystal structures of tetra-*n*-Butylammonium salts of $GaCl_4^-$, $GaBr_4^-$ and GaI_4^-', Journal of Coordination Chemistry, 56: 18, 1567 — 1574
To link to this Article: DOI: 10.1080/00958970310001636453
URL: http://dx.doi.org/10.1080/00958970310001636453

J. Coord. Chem., Vol. 56, No. 18, 15 December 2003, pp. 1567–1574

THE CRYSTAL STRUCTURES OF TETRA-*n*-BUTYLAMMONIUM SALTS OF $GaCl_4^-$, $GaBr_4^-$ AND GaI_4^-

K. RUDAWSKA* and H. PTASIEWICZ-BĄK

Institute of Nuclear Chemistry and Technology, ul. Dorodna 16, 03-195 Warszawa, Poland

(Received 19 August 2002; In final form 10 October 2003)

Tetra-*n*-butylammonium salts of $GaCl_4^-$, $GaBr_4^-$ and GaI_4^- have been prepared from stoichiometric amounts of GaX_3 and Bu_4NX in methanol. Their crystal structures have been determined by single crystal X-ray diffraction. The $Bu_4N[GaCl_4]$ and $Bu_4N[GaBr_4]$ salts crystallize in the orthorhombic system, space group Pnna, whereas the $Bu_4N[GaI_4]$ salt crystallizes in the monoclinic system, space group $P2_1/n$. For all compounds there are four molecules per unit cell. The structures consist of distorted tetrahedral GaX_4^- ions and Bu_4N^+ cations. In the tetrahedral $GaCl_4^-$ and $GaBr_4^-$ anions there are two pairs of Ga–X distances: 2.163(2), 2.174(2) Å and 2.297(2), 2.316(3) Å, respectively. In the GaI_4^- ion there are four Ga–I distances: 2.537(2), 2.539(2), 2.549(2) and 2.554(2) Å. The results show that the significantly greater increase in Ga–X distance on passing from Br to I than from Cl to Br results in lower symmetry and is accompanied by greater distortion of the tetrahedron.

Keywords: Tetra-*n*-butylammonium salts; Tetrachlorogallate(III); Tetrabromogallate(III); Tetraiodogallate(III); X-ray crystal structure

INTRODUCTION

In our previous paper [1] the $Bu_4N[InI_4]$ salt was structurally characterized and compared with the tetra-*n*-butylammonium salts of $InCl_4^-$ and $InBr_4^-$ previously studied [2]. We found that the symmetry of the space group decreases with increasing size of the halide ion, from Pnna for the tetrachloride and tetrabromide salts to $P2_1/n$ for the tetraiodide salt, and that greater distortion of the tetrahedron is observed for lower symmetry. Our objective in this study was to find more evidence for the correlation between the size of the MX_4^- anion, the space group and distortion of the tetrahedron for the example of the title compounds. X-ray investigations of the title compounds reported so far have been carried out by the powder diffraction and Weissenberg methods using Cu Kα radiation [3]. However, only the unit cell parameters with an error of 0.1% and the possible space groups have been determined [3]. The pri-

---

*Corresponding author. E-mail: krudaw@orange.ichtj.waw.pl

ISSN 0095-8972 print: ISSN 1029-0389 online © 2003 Taylor & Francis Ltd
DOI: 10.1080/00958970310001636453

mary purpose of this investigation was to unequivocally determine the space groups of the title compounds and the distortion of the GaX_4^- tetrahedra as a function of the X atom (X = Cl, Br and I). The second objective was to provide data that, together with results previously reported [1,2] and data on thallium and aluminum salts [4], are needed to check whether the M–X distances in the $Bu_4N[MX_4]$ salts show the so-called secondary periodicity [5]. According to this effect, which was observed for the M–C distance in the trimethyl compounds of Group 13 elements [6], the increase in the M–X distance should vary from small to large and again to small within the Al–Ga, Ga–In and In–Tl pairs, respectively. However, this would happen only in the case of an essentially covalent M–X bond. Thus, changes in the M–X distances down Group 13 elements could serve as a measure of the M–X bond character.

EXPERIMENTAL

Preparation of $Bu_4N[GaX_4]$

The $Bu_4N[GaX_4]$ salts (X = Cl, Br and I) were prepared from analytical grade GaX_3 and Bu_4NX in methanol [7]. Because of the susceptibility of gallium trihalides to moisture, the reactions were carried out in a dry box. In the first step the GaX_3 salt was dissolved in a minimum volume of dry methanol and then a methanol solution containing a stoichiometric amount of Bu_4NX was added. Precipitation of small crystals was observed immediately. The crystals were filtered off, dissolved in methanol and left to crystallize, yielding single crystals that were stable in air.

X-ray Studies

X-ray reflections for all compounds were collected at room temperature on a KUMA KM4 (Mo Kα radiation) four-circle diffractometer operating in the ω–2θ mode. Two standard reflections were monitored every 200 reflections and were found to remain constant. Unit cell dimensions and standard deviations were obtained by least-squares fit to 25 reflections (average $15 < 2\theta < 30$). Reflections were processed using profile analysis in each case and were corrected for Lorentz and polarization effects. An absorption correction based on a ψ-scan was applied. The atoms in the anions and almost all nitrogen and carbon atoms in the cations were located by the Patterson method. The few remaining nitrogen and carbon atoms were located by successive Fourier syntheses. However, using difference Fourier maps we could not determine the positions of all hydrogen atoms. Hydrogen atoms were therefore located using standard geometrical criteria and restrained to be bound to C-atoms with C–H distances of 0.97 Å. Final refinement was carried out on the positional parameters of all atoms, using anisotropic temperature factors for all non-hydrogen atoms and isotropic temperature factors for hydrogen atoms. The structure was refined by full-matrix least-squares methods on F^2. A weighting scheme was used in the form: $w = 1/[\sigma^2(F_0^2) + (AP)^2 + BP]$, where $P = (\mathrm{Max}(F_0^2) + 2F_c^2)/3$ and A, B are the refined parameters listed in Table I. Calculations were carried out using the SHELXL97 program [8]. Listings of observed and calculated structure factors and anisotropic thermal parameters are available from the authors on request.

TABLE I Crystal data and structure refinement details for Bu$_4$N[GaCl$_4$], Bu$_4$N[GaBr$_4$] and Bu$_4$N[GaI$_4$]

Empirical formula	$C_{16}H_{36}NCl_4Ga$	$C_{16}H_{36}NBr_4Ga$	$C_{16}H_{36}NI_4Ga$
Formula weight	452.09	631.58	819.49
Temperature (K)	293 K	293 K	293 K
Wavelength (Å)	0.71073	0.71073	0.71073
Crystal system	Orthorhombic	Orthorhombic	Monoclinic
Space group	Pnna	Pnna	$P2_1/n$
Unit cell dimensions (Å)	$a = 18.521(11)$ $b = 11.540(10)$ $c = 11.441(10)$	$a = 18.638(9)$ $b = 11.773(5)$ $c = 11.689(4)$	$a = 11.646(6)$ $b = 20.846(6)$ $c = 11.678(9)$ $\beta = 98.59(6)$
Volume (Å)	$V = 2445.2$	$V = 2565.0$	$V = 2803.3$
Z	4	4	4
Calculated density (g cm^3)	10.041	1.636	1.942
μ(Mo Kα) (mm^{-1})	1.34	7.3	5.39
$F(000)$	816	1240	1528
Crystal dimensions (mm)	0.7 × 0.2 × 0.3	0.6 × 0.15 × 0.15	0.4 × 0.4 × 0.4
Max 2θ for data collection (°)	59.99	59.94	43.96
Index range	$-25 \leq h \leq 0$, $0 \leq k \leq 16$, $0 \leq l \leq 15$	$0 \leq h \leq 26$, $0 \leq k \leq 16$, $0 \leq l \leq 16$	$-12 \leq h \leq 10$, $-21 \leq k \leq 0$, $-12 \leq l \leq 0$
No. of measured reflections	863	814	1976
No. of unique reflections	862	800	1858
No. of reflections with $F_o > 4\sigma(F_o)$	439	310	1512
R_{int}	0.0234	0.0141	0.0165
Method of structure solution		Patterson method	
Method of structure refinement		Lease squares on F^2	
No. of parameters refined	104	104	204
Goodness-of-fit on F^2	1.102	1.138	1.070
Final $R1$ $[F_o > 4\sigma(F_o)]$	0.0245	0.0277	0.0319
Final $wR2$ index (for all unique reflections)	0.0772	0.0972	0.0937
Absorption correction		Experimental ψ-scan	
Min. and max. transmission factors	0.62, 0.79	0.59, 0.80	0.60, 0.78
Larger diff. Peak and hole (e Å^{-3})	0.06 and –0.06	0.18 and −0.19	0.50 and −0.23
Weight parameters (A, B)	0.0248, 0.00	0.0298, 0.00	0.0636, 0.7168
Mean shift/esd	0.022	0.007	0.013

Table I shows crystal and refinement data. Tables II and III show the positional and equivalent isotropic displacements, and interatomic distances and bond angles are shown in Tables IV and V. Figures 1 and 2 show the Bu$_4$N[GaCl$_4$] molecule (which is isostructural with the Bu$_4$N[GaBr$_4$] molecule) and the Bu$_4$N[GaI$_4$] molecule (which is isostructural with the Bu$_4$N[InI$_4$] [1] molecule) with the numbering of atoms.

DISCUSSION

Our results show that the Bu$_4$N[GaCl$_4$] and Bu$_4$N[GaBr$_4$] salts crystallize in the orthorhombic space group Pnna, whereas Bu$_4$N[GaI$_4$] crystallizes in the monoclinic space group $P2_1/n$. The a, b and c parameters of the unit cells determined previously [3] are, within their rather large error, equal to ours, whereas the space groups suggested [3] are wrong.

Comparison of the molecular volumes (see Table I, V/Z) for the title compounds shows that the difference in the cell volume between the iodide and bromide salts is twice as large as that between the bromide and chloride salts. However, a plot of V

TABLE II Fractional atomic coordinates and equivalent isotropic displacement ($Å^2$) for $Bu_4N[GaC1_4]$, $Bu_4N[GaBr_4]$

Atom	*x*	*y*	*z*	U_{eq}
Ga	0.09264(5)	0.25(0)	0.75(0)	0.1298(5)
Cl(1)	0.1589(1)	0.1087(1)	0.6873(2)	0.1611(7)
Cl(2)	0.0266(2)	0.1831(3)	0.8918(2)	0.230(2)
N	0.0634(3)	0.75(0)	0.75(0)	0.100(1)
C(11)	0.1098(3)	0.8238(4)	0.8285(4)	0.109(1)
C(12)	0.1605(3)	0.7673(7)	0.9108(4)	0.123(2)
C(13)	0.2047(4)	0.8559(7)	0.9774(6)	0.160(2)
C(14)	0.2592(5)	0.8022(7)	1.0613(7)	0.180(3)
C(21)	0.0170(3)	0.6724(4)	0.8243(4)	0.112(1)
C(22)	−0.0405(4)	0.6028(6)	0.7687(7)	0.133(2)
C(23)	−0.0779(5)	0.5269(8)	0.8547(8)	0.169(3)
C(24)	−0.1456(6)	0.466(1)	0.8036(9)	0.217(5)
Ga	0.0974(1)	0.25(0)	0.75(0)	0.097(1)
Br(1)	0.1656(1)	0.1045(1)	0.6770(2)	0.1212(9)
Br(2)	0.0260(1)	0.1742(2)	0.8929(3)	0.216(2)
N	0.0613(9)	0.75(0)	0.75(0)	0.084(5)
C(11)	0.1049(8)	0.8295(9)	0.821(2)	0.091(5)
C(12)	0.1511(9)	0.774(1)	0.911(1)	0.097(5)
C(13)	0.1936(8)	0.850(1)	0.977(1)	0.107(6)
C(14)	0.248(1)	0.813(2)	1.068(1)	0.157(8)
C(21)	0.0142(8)	0.6755(9)	0.837(1)	0.071(4)
C(22)	−0.4449(7)	0.612(1)	0.770(2)	0.125(7)
C(23)	−0.081(1)	0.536(1)	0.857(2)	0.162(8)
C(24)	−0.149(1)	0.485(2)	0.817(2)	0.20(1)

TABLE III Fractional atomic coordinates and equivalent isotopic displacement ($Å^2$) for $Bu_4N[GaI_4]$

Atom	*x*	*y*	*z*	U_{eq}
Ga	0.4693(1)	0.81845(6)	1.2323(1)	0.0940(4)
I(1)	0.5176(1)	0.92645(5)	1.14883(9)	0.1392(4)
I(2)	0.49169(8)	0.73286(4)	1.08074(7)	0.1182(4)
I(3)	0.60885(8)	0.79769(5)	1.41702(7)	0.1223(4)
I(4)	0.26140(9)	0.81762(7)	1.2736(1)	0.1565(5)
N	0.3677(8)	0.8628(5)	0.7204(7)	0.100(3)
C(11)	0.272(1)	0.8619(7)	0.800(1)	0.119(4)
C(12)	0.248(1)	0.9195(8)	0.863(1)	0.131(4)
C(13)	0.153(2)	0.910(1)	0.933(2)	0.20(1)
C(14)	0.139(2)	0.960(1)	1.018(3)	0.30(2)
C(21)	0.355(1)	0.9212(8)	0.642(1)	0.134(5)
C(22)	0.236(1)	0.9314(9)	0.570(2)	0.156(6)
C(23)	0.252(4)	0.995(2)	0.484(2)	0.31(2)
C(24)	0.145(5)	1.017(3)	0.463(6)	0.40(3)
C(31)	0.481(1)	0.8725(6)	0.8046(8)	0.104(3)
C(32)	0.591(1)	0.8694(8)	0.752(1)	0.132(5)
C(33)	0.698(1)	0.8757(9)	0.851(2)	0.147(6)
C(34)	0.806(2)	0.883(1)	0.820(2)	0.23(1)
C(41)	0.366(1)	0.8024(7)	0.654(1)	0.113(4)
C(42)	0.394(2)	0.7421(6)	0.722(1)	0.114(4)
C(43)	0.374(2)	0.6843(8)	0.655(2)	0.142(5)
C(44)	0.394(2)	0.6219(8)	0.721(2)	0.191(8)

TABLE IV Bond lengths (Å) and bond angles (°) for Bu$_4$N[GaC1$_4$] and Bu$_4$N[GaBr$_4$]

GA–Cl(1)	2.163(2)	Cl(1)–Ga–Cl(2)	107.4(1)
Ga–Cl(2)	2.174(2)	Cl(1)–Ga–Cl(2)^a	109.8(1)
N–C(11)	1.51(1)	Cl(1)–Ga–Cl(1)^a	110.9(1)
N–C(21)	1.50(1)	Cl(2)–Ga–Cl(2)^a	111.5(2)
C(11)–C(12)	1.48(1)	C(11)–N–C(21)	109.0(3)
C(12)–C(13)	1.52(1)	C(11)–N–C(21)^b	109.0(3)
C(13)–C(14)	1.52(1)	C(11)–N–C(11)^b	110.4(5)
C(21)–C(22)	1.48(1)	C(21)–N–C(21)^b	110.3(5)
C(22)–C(23)	1.49(1)	N–C(11)–C(12)	119.4(4)
C(23)–C(24)	1.55(1)	N–C(21)–C(22)	119.5(5)
		C(11)–C(12)–C(13)	111(1)
		C(12)–C(13)–C(14)	114(1)
		C(21)–C(22)–C(23)	112(1)
		C(22)–C(23)–C(24)	113(1)
Ga–Br(1)	2.297(2)	Br(1)–Ga–Br(2)	107.4(1)
Ga–Br(2)	2.316(3)	Br(1)–Ga–Br(2)^a	109.7(1)
N–C(11)	1.49(2)	Br(1)–Ga–Br(1)^a	112.9(2)
N–C(21)	1.53(1)	Br(2)–Ga–Br(2)^a	109.8(2)
C(11)–C(12)	1.51(2)	C(11)–N–C(21)	110(1)
C(12)–C(13)	1.43(2)	C(11)–N–C(21)^b	106(1)
C(13)–C(14)	1.53(2)	C(11)–N–C(11)^b	114(2)
C(21)–C(22)	1.49(2)	C(21)–N–C(21)^b	110(2)
C(22)–C(23)	1.51(2)	N–C(11)–C(12)	115(1)
C(23)–C(24)	1.47(2)	N–C(21)–C(22)	117(1)
		C(11)–C(12)–C(13)	115(1)
		C(12)–C(13)–C(14)	124(1)
		C(21)–C(22)–C(23)	109(1)
		C(22)–C(23)–C(24)	114(1)

Symmetry codes: $^a x, -y+1/2, -z+3/2$; $^b x, -y+3/2; -z+3/2$.

TABLE V Bond lengths (Å) and bond angles (°) for Bu$_4$N[GaI$_4$]

Ga–I(1)	2.549(2)	I(1)–Ga–I(2)	107.5(1)
Ga–I(2)	2.554(2)	I(1)–Ga–I(3)	109.0(1)
Ga–I(3)	2.537(2)	I(1)–Ga–I(4)	110.5(1)
Ga–I(4)	2.539(2)	I(2)–Ga–I(3)	110.6(1)
N–C(11)	1.55(1)	I(2)–Ga–I(4)	109.0(1)
N–C(21)	1.52(2)	I(3)–Ga–I(4)	110.2(1)
N–C(31)	1.54(1)	C(11)–N–C(21)	111(1)
N–C(41)	1.49(2)	C(21)–N–C(31)	106(1)
C(11)–C(12)	1.46(2)	C(31)–N–C(41)	113(1)
C(12)–C(13)	1.49(2)	C(41)–N–C(11)	111(1)
C(13)–C(14)	1.46(3)	C(41)–N–C(21)	112(1)
C(21)–C(22)	1.52(2)	C(11)–N–C(31)	104(1)
C(22)–C(23)	1.70(4)	N–C(11)–C(12)	120(1)
C(23)–C(24)	1.31(4)	N–C(21)–C(22)	116(1)
C(31)–C(32)	1.50(2)	N–C(31)–C(32)	116(1)
C(32)–C(33)	1.57(2)	N–C(41)–C(42)	117(1)
C(33)–C(34)	1.37(2)	C(11)–C(12)–C(13)	113(1)
C(41)–C(42)	1.50(2)	C(12)–C(13)–C(14)	117(2)
C(42)–C(43)	1.44(2)	C(21)–C(22)–C(23)	105(1)
C(43)–C(44)	1.52(2)	C(22)–C(23)–C(24)	101(4)
		C(31)–C(32)–C(33)	109(1)
		C(32)–C(33)–C(34)	119(2)
		C(41)–C(42)–C(43)	114(1)
		C(42)–C(43)–C(44)	116(2)

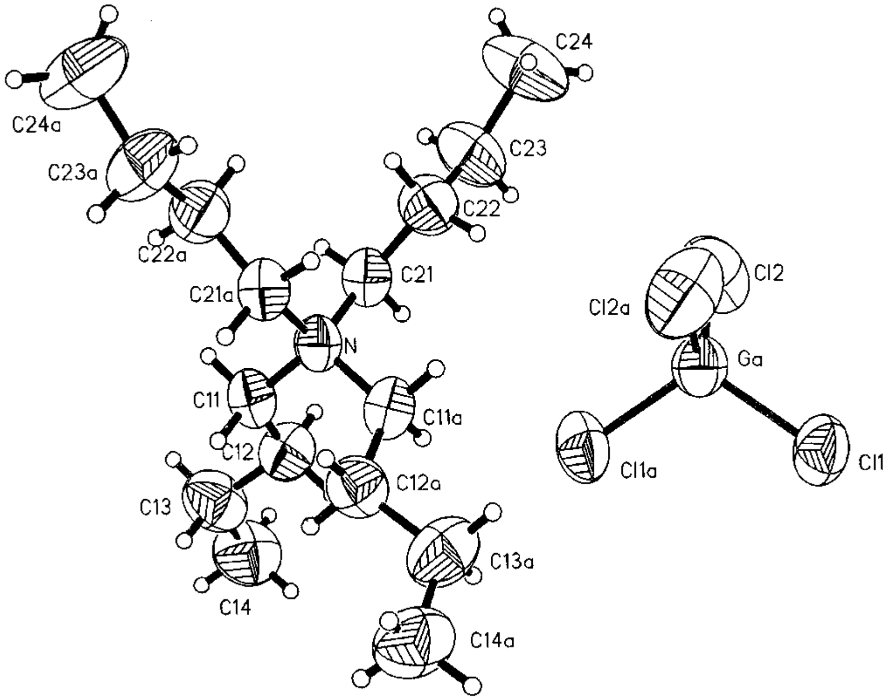

FIGURE 1 The molecular structure of $Bu_4N[GaCl_4]$ with atomic numbering.

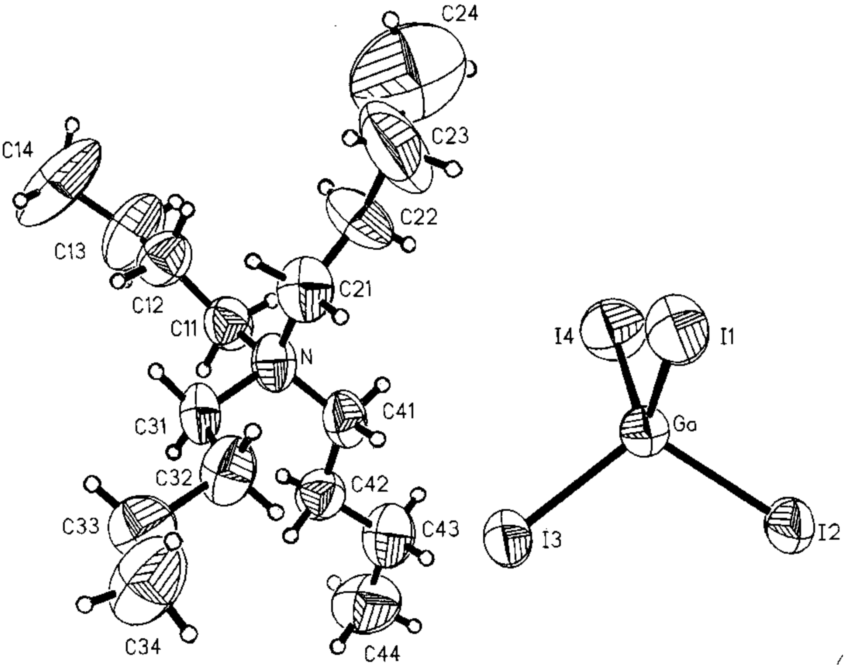

FIGURE 2 The molecular structure of $Bu_4N[GaI_4]$ with atomic numbering.

as a function of $(M–X)^3$ where M–X is the metal-to-halide distance shows that for the GaI_4^- salt the unit cell volume is slightly smaller than expected from the increase between the chloride and bromide salts.

The structures of the salts $Bu_4N[GaCl_4]$, $Bu_4N[GaBr_4]$ and $Bu_4N[GaI_4]$ consist of tetrahedral Bu_4N^+ cations and more or less distorted tetrahedral GaX_4^- anions. In each salt the Bu_4N^+ cation has almost the same structure and the average N–C distance is 1.51 Å. The C–C distance changes in the range from 1.37(2) to 1.57(2) Å (average 1.50 Å) but in this respect the $Bu_4N[GaI_4]$ salt provides two exceptions. In this salt the C(22)–C(23) distance, which is the longest, is 1.70(4) Å, while the C(23)–C(24) distance, which is the shortest, is 1.31(4) Å. It should be noted that C(23) and C(24) are two end atoms in one of the chains and have the biggest equivalent isotropic displacement. The data in Table III show that in each chain the two end atoms always have large equivalent isotropic displacements. It should be added that the structure of the $Bu_4N[InI_4]$ salt also shows short C(23)–C(24) and long C(22)–C(23) distances, which are accompanied by large equivalent isotropic displacements of the two end atoms [1]. The average C–N–C and N–C–C angles are similar for all compounds at 110 and 117°, respectively. The average C–C–C angle in the chloride and iodide salts is 113°, whereas in $Bu_4N[GaBr_4]$ it is equal to 116°. This is probably because the C(12)–C(13)–C(14) angle in the bromide salt, 124°, is much larger than in the remaining salts.

The crystal structures of the anions in the $Bu_4N[GaX_4]$ salts show significant differences. In the orthorhombic $Bu_4N[GaCl_4]$ and $Bu_4N[GaBr_4]$ salts the four M–X distances are grouped into pairs of identical distances: 2.163(2), 2.174(2) Å and 2.297(2), 2.316(3) Å, respectively. The way in which the relatively small $GaCl_4$ and $GaBr_4$ tetrahedra are packed in the unit cell results in the Pnna symmetry. In contrast, in the monoclinic $Bu_4N[GaI_4]$ salt the large GaI_4 tetrahedron shows one pair of statistically equal distances (2.537(2), 2.539(2) Å) and one pair of similar distances (2.549(2) and 2.554(2) Å). Moreover, the difference between the longest and shortest M–X distances is distinctly greater in the iodide salt than in the chloride and bromide salt. Splitting of the distances is accompanied by splitting of the X–Ga–X angles which vary from 107.4(1) to 112.9(2)°. The average X–X distance in the tetrahedron increases from 3.50 to 3.766 to 4.155 Å for the chloride, bromide and iodide salts, respectively. In each case it differs only slightly from the distance calculated for a regular tetrahedron with the center-to-vertex distance equal to the average M–X distance. The Ga atoms in the tetrahedra always have five nearest N atoms. In the GaI_4^- salt all the Ga–N distances are different and are in the range 5.990(8) to 7.943(9) Å (the average distance is 6.581 Å). In the $GaCl_4^-$ and $GaBr_4^-$ salts there are two pairs of distances, 5.795(5), 6.409(5) Å and 5.925(3), 6.550(8) Å, respectively. The fifth distance is 6.370(7) or 6.362(17) Å and the average distance is 6.156 or 6.262 Å, for the tetrachloride and the tetrabromide. Thus, the lower symmetry of the salt and the greater distortion of the tetrahedron are accompanied by a much larger range of Ga–N distances.

For the M^{3+} ions of Group 13 elements the crystal structures of tetra-*n*-butylammonium salts of the following anions have been solved: AlI_4^- [9], $InCl_4^-$ and $InBr_4^-$ [2], InI_4^- [1] and TlI_4^- [10,11]. The reported structures, together with the data for the gallium salts presented in this paper, confirm our presumption that in the tetra-*n*-butylammonium salts of MCl_4^- and MBr_4^- there are always two statistically different pairs of identical M–X distances, whereas in salts of MI_4^- there are four different M–I distances with a significantly greater difference between the longest and shortest distances. A comparison of the existing data shows that the tetrachloride and tetrabromide salts crystallize in the orthorhombic space group Pnna, whereas salts with the larger iodine atom crystallize in the monoclinic space group $P2_1/n$.

References

[1] K. Rudawska, H. Ptasiewicz-Bąk and S. Siekierski, *J. Coord. Chem.* **55**, 403 (2002).
[2] A. Masood, M.A. Khan and D.G. Tuck, *Acta Cryst.* **B38**, 803 (1982).
[3] A.N. Grigor'ev, F.M. Spiridonov, V.M. Ionov, V.A. Kravcov, L.M. Mikheeva and L.H. Komissareeva, *Zhur. Neorg. Khim.* **17**, 664 (1977).
[4] K. Rudawska and S. Siekierski, *J. Coord. Chem.*, in press.
[5] P. Pyykkö, *Chem. Rev.* **88**, 563 (1988).
[6] A. Haaland, *J. Mol. Struct.* **97**, 115 (1983).
[7] L.M. Mikheeva and A.N. Grigor'ev, *Vestnik Moskov. Univ.*, *Khimia* **13**, 480 (1972).
[8] G.M. Sheldrick, SHELXL97, Program for Crystal Structure Determination (University of Göttingen, Germany 1997).
[9] R.D. Rogers, J.C. Backer and J.L. Atwood, *J. Cryst. Spect. Res.* **14**, 333 (1984).
[10] J. Glaser, P.L. Gogin M. Sandström and V. Lustko, *Acta Chem. Scand.* **A36**, 55 (1982).
[11] J. Glaser, P.L. Gogin, M. Sandström and V. Lustko, *Acta Chem. Scnd.* **A37**, 437 (1982).