Table 3. *Comparison of bond lengths* (Å) *in* YB_{12}, ZrB_{12} *and* UB_{12}

	YB_{12}	ZrB_{12}	UB_{12}
Unit-cell edge	7·500	7·408	7·473
B–B (intercubo-octahedral)	1·809 ± 0·024	1·780 ± 0·026	1·803 ± 0·026
B–B (intracubo-octahedral)	1·684 ± 0·024	1·678 ± 0·026	1·675 ± 0·026
Metal–B	2·783 ± 0·012	2·750 ± 0·013	2·771 ± 0·013
Metallic radius (Pauling) (coordination number 12)	1·797	1·597	1·516

atomic size of the metal atoms. Thus, in the cubic dodecaboride series, variations in the unit-cell dimensions are controlled by the internal B–B distances in the cubo-octahedron. These, in turn, are probably affected by the electron transfer between the boron and the metal atoms.

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On the preparation and crystallography of tetramethylammonium chloride hydrogen chloride*.

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A number of studies of compounds which appear to contain the bichloride ($Cl\text{-}H\text{-}Cl^-$) ion have been reported recently (Herbrandson, Dickerson & Weinstein, 1952; Waddington, 1958; Sharp, 1958; Vallee & McDaniel, 1962; Chang & Westrum, 1962). No X-ray studies have appeared, however, with the exception of the brief report of Waddington (1958) which included results of X-ray powder measurements on tetramethylammonium chloride hydrogen chloride (hereafter: TMAC1-HC1). An orthorhombic unit cell containing 8 molecules with $a = 14{\cdot}81$, $b = 11{\cdot}46$, $c = 10{\cdot}38$ Å, was reported for TMAC1-HC1 by the above author.

We have prepared TMAC1–HC1 by adding anhydrous hydrogen chloride to tetramethylammonium chloride in anhydrous methanol. Needle crystals of TMAC1-HC1, stable in sealed capillaries, were obtained from the latter solvent. Analysis of single-crystal material gave C,32·3; H,9·13; Cl,47·98 %. $(CH_3)_4N^+HCl_2^-$ requires C,32·9; H,8·9; Cl,48·5 %.

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Crystals sealed in glass capillaries were examined with Cu $K\alpha$ radiation, employing Weissenberg and precession techniques. The needle crystals were observed to be orthorhombic with the needle axis the b axis of the cell, $a = 9{\cdot}27 \pm 0{\cdot}01$, $b = 7{\cdot}73 \pm 0{\cdot}01$, $c = 11{\cdot}59 \pm 0{\cdot}01$ Å. The diffraction symbol is *mmmPn* . a indicating possible space groups *Pnma* and $Pn2_1a$. The calculated crystal density for 4 molecules per unit cell is 1·168; $\varrho_{obs} = 1{\cdot}15$.

This cell, slightly less than half the size of the unit cell chosen by Waddington, is considerably more amenable to X-ray structure study. We do not, at present, plan further X-ray structural work on this material.

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