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Short Communications

On the Crystal Structure of the Tetrabutylammonium Tetraiodothallate(III) $[N(C_4H_9)_4][TlI_4]$

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We have previously published results of the crystal structure determination of the title compound.[1] Following publication, we received a letter from Dr. R. E. Marsh,* in which he kindly pointed out that the crystal structure which we described in the space group $P2_1$, could possibly be better described in $P2_1/n$ (non-standard setting of $P2_1/c$, equivalent positions: $\pm(x, y, z)$, $\pm(1/2-x, 1/2+y, 1/2-z)$). The additional glide plane in $P2_1/n$ requires the systematic absence of all reflexions $h0l$ with $(h+l)$ odd.

In our original data set, the $h0l$'s with $(h+l)$ odd included 11 reflections having $F_{obs} \geq 3.92\sigma(F_{obs})$, leading us to describe the structure in $P2_1$. We have now measured all the equivalent $h0l$ reflections, and are convinced that they are systematically extinguished; the intensities that appear to be non-zero arise, we believe, from splitting of the crystal or from a small satellite. As a consequence, we have re-refined the structure in $P2_1/n$, using the same data as previously but with the eleven odd $h0l$'s removed.

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Table 1. Final fractional atomic positional parameters for the heavy atoms. Estimated standard deviations are given in parentheses.

$P2_1/n$	($P2_1$)	x	y	z
Tl	(Tl1, Tl2)	0.23476(8)	0.68332(5)	0.46103(9)
I1	(I1, I7)	0.2805(2)	0.6848(1)	0.2368(2)
I2	(I2, I5)	0.4325(2)	0.7030(1)	0.6133(2)
I3	(I3, I6)	0.0711(2)	0.7742(1)	0.4883(2)
I4	(I4, I8)	0.1435(2)	0.5669(1)	0.5099(2)

Table 2. Final anisotropic thermal parameters with estimated standard deviations in parentheses. The expression used is $\exp[-2\pi^2(U_{11}h^2a^{*2}+\ldots+2U_{12}hka^*b^*+\ldots)]$.

Atom	U_{11}	U_{12}	U_{33}	U_{12}	U_{13}	U_{23}
Tl1	0.0719(6)	0.0775(6)	0.0754(6)	0.0009(6)	0.0082(5)	0.0013(6)
I1	0.1469(20)	0.1673(23)	0.0840(14)	0.0024(18)	0.0287(14)	0.0028(15)
I2	0.0675(11)	0.1526(20)	0.1017(15)	0.0081(11)	0.0056(10)	–0.0252(14)
I3	0.0778(11)	0.0899(14)	0.1145(15)	0.0103(10)	–0.0043(10)	–0.0120(11)
I4	0.1088(15)	0.0818(14)	0.1463(19)	–0.0022(13)	0.0019(14)	0.0279(14)

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Table 3. Some interatomic distances in Ångströms and angles in degrees. Estimated standard deviations are given in parentheses.

Distance	Å [a]	Å [b]	Angle	deg.[a]
Tl–I1	2.752(2)	2.782(3)	I1–Tl–I2	110.72(8)
Tl–I2	2.748(2)	2.766(2)	I1–Tl–I3	109.62(7)
Tl–I3	2.764(2)	2.775(3)	I1–Tl–I4	109.93(8)
Tl–I4	2.770(2)	2.791(2)	I2–Tl–I3	110.95(7)
			I2–Tl–I4	108.43(7)
			I3–Tl–I4	107.11(7)

[a] Non-corrected values. [b] Corrected for thermal motion assuming the I atom to ride on the Tl atom.

The refinements were performed in the same way as in the previous paper.[1] Some, mostly terminal, carbon atoms obtained rather high temperature factors and probably have alternative positions. However, it was not necessary to keep positional parameters constant for any carbon atom, as was the case for $P2_1$ (probably because of the near singularities which arise when an approximately centrosymmetric structure is refined in a non-centrosymmetric space group). The final refinement cycle (114 refined parameters, the heavy atoms anisotropic, the nitrogen and carbon atoms isotropic) led to $R=5.4$ % ($R_w=8.0$ %, $S=1.82$) thus not significantly better than for $P2_1$. The results are given in Tables 1–3. Full parameter listings and structure factors are available from the authors on request. As can be seen, the distances and angles between the heavy atoms are not significantly different from the values of $P2_1$ when the mean values are taken in $P2_1$ (*e.g.* r_{Tl-I1} in $P2_1/n$ corresponds to $(r_{Tl-I1}+r_{Tl-I7})/2$ in $P2_1$), but the TlI_4 tetrahedron is more regular in $P2_1/n$. The standard deviations are about 50 % lower in the latter space group.

1. Glaser, J., Goggin, P. L., Sandström, M. and Lutsko, V. *Acta Chem. Scand. A 36* (1982) 55.

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