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The unit cells of ($PtenX_4$) ($PtenX_2$), (X=Cl, Br, I).

By K. W. Browall, J. S. Kasper and L. V. Interrante,
General Electric Corporate Research and Development, Schenectady, New York 12301, *U.S.A.*

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Unit-cell data and indexed power X-ray diffraction patterns are reported for the complexes ($PtenX_4$) ($PtenX_2$), where en = $H_2NCH_2CH_2NH_2$, X = Cl, Br, I. The unit cell for the case where X = Br has been corrected from a previous report.

In the course of characterizing mixed-valence platinum complexes of the type [$PtenX_4$] [$PtenX_2$], hereafter referred to by the empirical formula $PtenX_3$, where en = $H_2NCH_2CH_2NH_2$ and X = Cl, Br, I, we have encountered difficulty in indexing X-ray powder diagrams on the basis of the unit cell given for $PtenBr_3$ (Ryan & Rundle, 1961), the only compound of this group for which structural data have been reported. Upon further investigation with single crystals as well as with powders, we have found that the cited lattice constants of $PtenBr_3$ are in error. Accordingly, we wish to report the corrected unit cell and the indexed powder pattern for $PtenBr_3$ and also the unit-cell data and powder patterns for the isomorphous compounds $PtenCl_3$ and $PtenI_3$.

Table 1. *Lattice constants for the* $PtenX_3$ *complexes*

Space group *Ama*2

	a	*b*	*c*
$PtenCl_3$	9·648 (6) Å	13·441 (8) Å	5·481 (4) Å
$PtenBr_3$	10·123 (4)	13·863 (6)	5·606 (5)
$PtenI_3$	11·029 (10)	14·329 (12)	5·862 (6)

The complexes were prepared by previously reported methods (Interrante, Browall & Bundy, 1974), based on the procedures of Watt & McCarley (1957*a*, *b*) and using $PtenCl_2$ as the starting material. In particular, $PtenBr_2$ and $PtenI_2$ were prepared from $PtenCl_2$ by halide exchange (Watt & McCarley, 1957*a*), and $PtenBr_4$ and $PtenCl_4$ were prepared by direct halogenation of the corresponding Pt(II) salts. The mixed-valence complexes were obtained by slow evaporation and cooling of aqueous solutions containing equimolar amounts of the Pt(II) and Pt(IV) complexes for the cases where X = Cl, Br. The method of Watt & McCarley (1957*b*) was used to prepare $PtenI_3$ from K_2PtI_6 and $PtenI_2$. An alternate method was also used to prepare $PtenBr_3$ from $PtenBr_2$ and $Pt(en)_2Br_4$ (Watt & McCarley, 1957*a*); the powder patterns for $PtenBr_3$ prepared by the two different techniques were identical. The powder patterns for each of the $PtenX_3$ compounds were in essential agreement with the partial powder patterns reported by Watt &

Table 2. *X-ray diffraction data for* $PtenX_3$ *complexes*

$PtenCl_3$			$PtenBr_3$			$PtenI_3$		
d (Å)	*I*/*I*₁	*hkl*	*d* (Å)	*I*/*I*₁	*hkl*	*d* (Å)	*I*/*I*₁	*hkl*
6·65	100	020	6·84	100	020	7·10	100	020
5·48	6	120	5·69	20	120	5·97	68	120
			5·16	17	011	5·43	56	011
4·82	18	200	5·06	30	200			
4·48	56	111	4·62	59	111	4·87	68	111
3·93	21	220	4·09	43	220	4·36	88	220
3·49	5	211	3·63	12	211	3·85	—	211
			3·56	10	031	3·70	—	031
3·36	7	040						
3·27	7	131						
3·17	7	140	3·27	13	140			
						3·26	—	320
2·753	24	240	2·853	37	240	3·00	72	240
2·734	18	002	2·801	37	002	2·937	64	002
2·536	8	022	2·600	17	022			
			2·535	12	400	2·740	—	400
2·381	9	202	2·454	30	202			
2·358	18	331				2·607	40	331
			2·419	12	151, 340			
						2·558	40	340
2·274	9	420	2·386	17	420			
2·245	27	222	2·309	33	222, 060	2·441	—	222, 411
						2·407	—	060
2·185	6	160				2·350	—	160
2·164	8	251	2·231	10	251			
2·036	6	260	2·097	7	260	2·192	—	260
						2·181	—	322
1·967	8	440	2·048	10	440			
1·943	18	242	2·004	33	242, 351	2·093	68	242
1·847	6	520	1·952	7	520			
			1·906	7	360	2·009	—	360, 402
1·815	9	071, 402, 013				1·937	—	013
						2·028	—	511
1·782	18	171, 113				1·918	—	113

McCarley (1957*b*). Elemental analyses for C, H, N gave satisfactory agreement with calculated values (in parentheses): $PtenCl_3$: C 6·8 (6·64), H 2·3 (2·23), N 7·9 (7·75); $PtenBr_3$: C 5·0 (4·85), H 1·6 (1·63), N 5·9 (5·66); $PtenI_3$: C 4·1 (3·78), H 1·2 (1·27), N 4·1 (4·41). Single crystals of $PtenCl_3$ and $PtenBr_3$ were grown by slowly cooling aqueous solutions (Interrante & Browall, 1974). The resulting crystals had a metallic sheen and were strongly dichroic.

The lattice constants in Table 1 were obtained by a least-squares analysis of the Debye–Scherrer patterns given in Table 2 (obtained with a 114·6 mm camera radius and Cu $K\alpha$ radiation). Intensities (peak heights) were obtained from densitometer traces. The space group, *Ama*2, was ascertained from X-ray precession photographs of single crystals of $PtenBr_3$ and $PtenCl_3$ and is equivalent to that reported for the disordered structure described by Ryan & Rundle (1961).

The lattice constants of $PtenBr_3$ given by Ryan & Rundle (1961) (after interchanging *a* and *c* to bring the reported space group, *C*2*cm*, into the standard setting, *Ama*2) are $a = 10·12$, $b = 14·77$, $c = 5·60$ Å. Referring to Table 1, the discrepancy between the sets of data is seen to be associated almost entirely with the value of *b*. It is difficult to check on the origin of the discrepancy because of an internal inconsistency in the paper by Ryan & Rundle. The reported X-ray density of 4·027 cm^{-3} does not follow from their (Ryan & Rundle, 1961) unit-cell data, which would correspond to a density of 3·93 g cm^{-3}. Our lattice constants call for a density of 4·18 g cm^{-3}. The measured density is 4·03 g cm^{-3}.

On the other hand, our single-crystal data appear to correspond to those of Ryan & Rundle (1961) with respect to systematic absences and in regard to agreement of individual structure factors so that no major difference in the crystal structure is suggested by our results. One difference in the single-crystal results is that we have not been able to observe the streaking between layer lines normal to **c*** as described by Ryan & Rundle (1961). Presumably, then, this would imply more complete disorder in the arrangement of the chains that are parallel to **c**.

It does not appear that a typographical error was responsible for the abnormally large value of *b* given by Ryan & Rundle (1961), since their interatomic distances follow from their lattice constants. In particular, the molecular plane (non-chain axis) Pt(IV)–Br and Pt(II)–Br distances of 2·51 Å are consistent with the reported *b* value. On the other hand, the new lattice constants indicate a value of 2·43 Å; this is in better agreement with the corresponding distance of 2·46 (6) Å in the related compound, $[Pt(NH_3)_2Br_4]$ $[Pt(NH_3)_2Br_2]$ (Wallen, Brosset & Vannerberg, 1962). The revised $PtenBr_3$ values also indicate that the in-plane Br–Br distance is 3·54 Å and the Br–Pt–Br angle is 86°24′.

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A reinvestigation of the crystal and molecular structure of hexachloroborazine. A correction.

By M. S. GOPINATHAN, M. A. WHITEHEAD, C. A. COULSON,* J. R. CARRUTHERS and J. S. ROLLETT, *Theoretical Chemistry Department, 1 South Parks Road, Oxford OX1 3TG, England*

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In the paper by Gopinathan, Whitehead, Coulson, Carruthers & Rollett [*Acta Cryst.* (1974). **B30**, 731–737] Fig. 5 represents the enantiomorph of the structure obtained from the parameters of Table 5 of the same paper.

The parameters resulting from our refinements in Table 5 (Gopinathan, Whitehead, Coulson, Carruthers & Rollett, 1974) have been inverted through a centre of symmetry so that they can be compared with the results of Haasnoot, Verschoor, Romers & Groeneveld (1972). In the preparation of Fig. 5 this inversion was not performed and the figure represents the enantiomorph of the structure obtained from the parameters of Table 5.

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* Deceased.