tion patterns. Because the crystals were attacked to some degree by all of the greases and adhesives commonly used for mounting, polyvinyl alcohol in aqueous solution was used to glue the crystals to glass wool fibers. The mounted crystals were enclosed in thin-wall capillary tubes that contained an atmosphere saturated with benzene. Precession camera data for two zones were then obtained in addition to powder diffraction data.

From the single crystal data, it was found that diphenyl picrylhydrazine, as grown from benzene solution, is monoclinic. The powder patterns were indexed on the basis of the cell obtained from the single crystal data and were used in a least-squares refinement of the cell size. Patterns taken on a diffractometer with Cu $K\alpha$ radiation were used in a preliminary refinement, the final refinement being a least-squares fit of the cell parameters to data from a Debye–Scherrer pattern taken with Cr $K\alpha$ radiation. Since all of the observable lines are in the forward reflection region, and because no corrections were made for the displacement of lines by absorption, it is felt that the internal consistency of the data set is not a fair measure of accuracy; the limits of error given below are approximately twice that indicated by this internal consistency. The cell dimensions are:

$$a = 9{\cdot}37 \pm 0{\cdot}02,\ b = 10{\cdot}78 \pm 0{\cdot}04,\ c = 11{\cdot}87 \pm 0{\cdot}02\ \text{Å}\ ,\quad \beta = 111^\circ\ 14' \pm 10'.$$

The observed interplanar spacings are compared with those calculated on the basis of this cell in Table 1.

The systematic absence of the $0k0$ reflections for odd values of k, together with a readily observable pyroelectric effect indicates the non-centrosymmetric space group $P2_1$.

The benzene of crystallization was determined by heating freshly crystallized material in a vacuum at 60 °C. to constant weight. A weight loss of 16·7% was found. The value calculated for one benzene per diphenyl picrylhydrazine is 16·5%. The density, calculated on the basis of two molecules of diphenyl picrylhydrazine plus two benzenes of crystallization per unit cell, is 1·406 g.cm.$^{-3}$, in good agreement with pycnometric density measurements of 1·40 g.cm.$^{-3}$.

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The crystal structures of some plutonium borides.

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A study of the plutonium–boron system has led to the identification of four plutonium borides, the structures being consistent with compounds of chemical formulae PuB, PuB_2, PuB_4, and PuB_6.

All the borides were prepared by heating powder-mixtures of elemental boron and plutonium in argon at 0·25 atmospheres. Preparations were carried out at 1200 °C. using a range of Pu:B ratios equivalent to 40–95 at.% boron; and also at 800 °C. in the more limited range 60–65 at.% boron. X-ray powder-photographs showed that the products of reaction contained unidentified phases, in addition to the borides already mentioned.

Plutonium monoboride was formed at 1200 °C. and in the composition range 40–70 at.% boron. The amount of monoboride formed decreased as the percentage of boron increased. This phase has a cubic NaCl-type structure (similar to that of TiB, ZrB and HfB) with lattice parameter 4·92 Å; the Pu–B distance is 2·46 Å. The existence of uranium and thorium monoborides has not been reported.

Plutonium diboride was formed in preparations at 800 °C., but not at 1200 °C. At 800 °C. the effect of changes in Pu:B ratio has not yet been fully investigated, but in the limited composition range studied (60–65 at.% B), PuB_2 was the only boride phase formed. It is simple hexagonal and is isomorphous with uranium diboride. Lattice parameters are $a = 3{\cdot}18$ and $c = 3{\cdot}90$ Å, with Pu atoms at (0, 0, 0) and boron atoms at (1/3, 2/3, 1/2) and (2/3, 1/3, $\bar{1}$/2). The structure is the AlB_2-type, in which boron atoms form two-dimensional hexagonal networks with planar layers of metal atoms interposed. Thus plutonium, uranium and several transition metals (Post, Glaser & Moskowitz, 1954) form a series of isocrystalline

Table 1. *Interatomic distances for some diborides* (Å)

Compound	Metal atomic radius	Lattice parameters		Metal-boron dist. expl.	Metal–boron dist. contact	Expl.-contact	Boron–boron distance in planes expl.	Expl.-contact (*B* diam.)
		a	*c*					
CrB_2	1·27	2·97	3·07	2·30	2·23	0·07	1·73	0
VB_2	1·34	3·00	3·06	2·31	2·24	0·07	1·73	0
TiB_2	1·47	3·03	3·23	2·38	2·32	0·06	1·75	0·01
MoB_2	1·39	3·05	3·08	2·32	2·28	0·04	1·76	0·02
TaB_2	1·46	3·08	3·27	2·41	2·35	0·06	1·78	0·04
NbB_2	1·46	3·09	3·30	2·43	2·35	0·08	1·78	0·04
HfB_2	1·59	3·14	3·47	2·51	2·44	0·07	1·81	0·07
ZrB_2	1·60	3·17	3·53	2·54	2·46	0·08	1·83	0·09
UB_2	1·52	3·14	4·00	2·70	2·36	0·34	1·81	0·07
PuB_2	1·60	3·18	3·90	2·68	2·47	0·21	1·83	0·09

diborides. Lattice parameters and interatomic distances of this series are listed in Table 1. A notable feature of the series is the unusually large metal–boron separation in both PuB_2 and UB_2. For the transition-metal diborides the experimental metal–boron distance exceeds the 'contact' distance (i.e. the sum of the close packed radii) by only 0·06–0·08 Å, whereas in UB_2 and PuB_2, the measured metal–boron distances exceed the calculated distances by 0·34, and 0·21 Å respectively.

A tetraboride phase was formed at 1200 °C. when the atomic percentage of boron was greater than 70. The proportion of this phase formed increased rapidly with boron content up to 85 at.% boron, but thereafter decreased with increasing boron content. The PuB_4 phase is isomorphous with UB_4 and is tetragonal, with $a = 7{\cdot}10$ and $c = 4{\cdot}014$ Å. The structure of UB_4 is given by Zalkin & Templeton (1950) who showed the space group to be *P*4/*mbm* and the atomic positions:

Metal in ±(0·31, 0·81, 0; 0·21, 0·31, 0).
4 B I in ±(0, 0, 0·2; 0·5, 0·5, 0·2).
4 B II ±(0·1, 0·6, 0·5; 0·4, 0·1, 0·5).
8 B III in ±(0·2, 0·04, 0·5; 0·7, 0·46, 0·5; 0·04, 0·2, 0·5; 0·54, 0·7, 0·5).

Using the lattice parameters found for plutonium tetraboride, the metal-boron distances are Pu–B I 2·77, Pu–B II 2·84, Pu–B III 2·47 Å i.e. the plutonium and the B III boron atoms are in contact.

Plutonium hexaboride was also formed at 1200 °C. when the amount of boron present exceeded 70 at.%. The proportion of the hexaboride formed increased steadily with increasing boron content. The structure is simple-cubic CaB_6-type and is isomorphous with ThB_6. With metal atoms at (0, 0, 0) and boron atoms at $\pm[(\frac{1}{2}, \frac{1}{2}, 0{\cdot}21), (\frac{1}{2}, 0{\cdot}21, \frac{1}{2}), (0{\cdot}21, \frac{1}{2}, \frac{1}{2})]$ the plutonium–boron distance is 2·92 Å, whereas the 'contact' distance is 2·47 Å. The boron atoms are in close contact since the boron–boron distance is 1·75 Å. The lattice parameter varies from $4{\cdot}115 \pm 0{\cdot}001$ to $4{\cdot}140 \pm 0{\cdot}001$ Å, suggesting that the hexaboride phase is not stoichiometric, and that excess boron is present in the structure.

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Heat treatment of evaporated carbon films.

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The structure analysis of evaporated carbon films deposited at room temperature was reported in the previous paper (Kakinoki *et al.*, 1960). In this note, we will report the structural change of these films when subjected to heat treatment.

Carbon films deposited at room temperature are attached on specimen holders made of thin carbon plates and heated to desired temperatures in a vacuum furnace. The film temperature is estimated by a small thermocouple placed near the specimen. The specimens used are taken from the same lot deposited at the same time, and they have a thickness of 240 Å. The highest temperatures attained in each treatment are 500, 700, 800, 900, 1000, and 1200 °C., respectively. The rate of temperature raise is about 100 °C. per min. Each specimen is kept at the highest temperature for 1 min., and then allowed to cool to room temperature, the rate of temperature decrease being very rapid.

Electron-diffraction diagrams show gradual change in the structure with increase of the temperature in heat treatment. These behaviours are shown in Figs. 1 and 2. Photographs in Fig. 1 are taken with s^2-sector under normal incidence, their intensity curves being represented in Fig. 2. At about 1200 °C., the diffraction pattern changes into sharp Debye–Scherrer rings with asymmetric line profiles, and they are all indexed with (*hk*0) of graphite having a lattice constant of $a = 2{\cdot}456$ Å, their spacings being listed in Table 1. No (*hkl*) ($l \neq 0$)

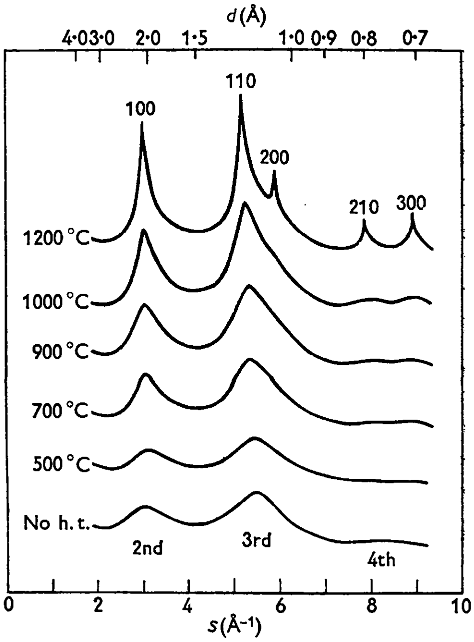

Fig. 2. Intensity curves of the photographs in Fig. 1 ($s = 2\pi/d = 4\pi \sin\theta/\lambda$).