Short Communications

Contributions intended for publication under this heading should be expressly so marked; they should not exceed about 500 words; they should be forwarded in the usual way to the appropriate Co-editor; they will be published as speedily as possible; and proofs will not generally be submitted to authors. Publication will be quicker if the contributions are without illustrations.

Acta Cryst. (1958). **11**, 300

The coefficient of thermal expansion of zirconium nitride.

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(*Received* 25 *June* 1957)

An X-ray method of determining the thermal expansion was adopted.

The zirconium nitride used was prepared by heating about 10 g. of zirconium powder, surrounded by a nitrogen atmosphere of low oxygen content, for 4 hr. at 1250° C. in an alumina boat. A chemical examination of the compound formed gave the analysis of 52·7, 47·2, and 0·05 atomic% for zirconium, nitrogen and magnesium respectively.

The zirconium nitride in powder form was enclosed in an unsealed silica capillary tube, and examined in a Unicam S. 150 high-temperature camera under a vacuum of the order of 1×10^{-4} mm. Hg. The camera furnace was fed from a supply fitted with a Claude Lyons BMVR-1725 stabilizer, and its temperature was further controlled by a regulator utilizing the resistance properties of the platinum-wound furnace itself.

The temperature of the specimen was inferred from the readings of a platinum–platinum/rhodium thermocouple consisting of a 0·7 mm. bead situated just below the specimen and having 0·005 in. leads. This thermocouple was calibrated by X-ray measurements of the interplanar spacings of platinum, using thermo-pure filings, in a silica capillary, at various temperatures. These measurements were compared with the dilatometric data for platinum of Esser & Eusterbrock (1941), which were taken as the standard.

The measured unit-cell dimensions are

$4{\cdot}5745 \pm 0{\cdot}001$ Å at 17° C.,
$4{\cdot}5865 \pm 0{\cdot}001$ Å at 445° C.,
$4{\cdot}5965 \pm 0{\cdot}001$ Å at 680° C.

These yield the following values for the coefficients of thermal expansion:

$6{\cdot}0 \pm 0{\cdot}5 \times 10^{-6}$° C.$^{-1}$, temperature range 17–445° C.;
$7{\cdot}0 \pm 0{\cdot}5 \times 10^{-6}$° C.$^{-1}$, temperature range 17–680° C.

There is no indication of a phase change over this region, and the unit-cell dimension at 17° C. was unchanged by the thermal treatment of the specimen.

The unit-cell dimension at room temperature agrees with the value of Duwez & Odell (1950) of 4·576 Å (quoted as 4·567 kX. units), in contrast to the values of 4·63 kX. units (Becker & Ebert, 1925) and 4·61 kX. units (van Arkel, 1924) reported by previous investigators.

References

ARKEL, A. E. VAN (1924). *Physica*, **4**, 286.
BECKER, K. & EBERT, F. (1925). *Z. Phys.* **31**, 268.
DUWEZ, P. & ODELL, F. (1950). *J. Electrochem. Soc.* **97**, 305.
ESSER, E. & EUSTERBROCK, H. (1941). *Arch. Eisenhüttenw.* **14**, 341.

Acta Cryst. (1958). **11**, 300

Crystallographic properties of procaine hydrochloride.

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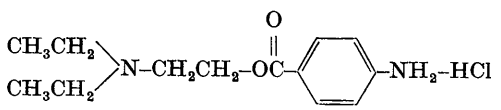
(*Received* 18 *October* 1957)

Procaine hydrochloride, used medicinally as a local anesthetic, has the chemical name 2-diethylaminoethyl-*p*-aminobenzoate hydrochloride. A mention of the optical crystallography is made by Keenan (1944). The compound is represented by the formula:



Crystallization from ethyl acetate–ethanol solution results in needles elongated parallel to *c* and showing {010}, {120} and small {100}. Crystallization from water gives plates lying on (010). The sample used for this study melted in the range 156·5–158·0° C. (Kofler hot stage). The crystal system is orthorhombic with space group D_{2h}^{15}–*Pcab* and eight molecules per cell. The observed density is 1·232 g.cm.$^{-3}$ (flotation), while the density calculated from X-ray data is 1·220 g.cm.$^{-3}$. The unit-cell dimensions are:

$$a_0 = 14{\cdot}35, \quad b_0 = 25{\cdot}04, \quad c_0 = 8{\cdot}28 \text{ Å}.$$

The optical properties are:

$\alpha = 1{\cdot}540$, $\beta = 1{\cdot}564$, $\gamma > 1{\cdot}70$ (all at 25° C., 5893 Å); $(+)2V = 37°$. The optic plane is 001, $\alpha = a$. Keenan (1944) gives $\alpha = 1{\cdot}540$, $\beta = 1{\cdot}566$, $\gamma > 1{\cdot}690$.

The powder data (Table 1) were obtained using a

Table 1. *X-ray powder diffraction data for procaine hydrochloride*

d_o (Å)	$I/I_{max.}$	*hkl*	d_c from *a*, *b*, *c* (Å)
12·52	0·04	020	12·52
6·91	0·40	111	6·89
		040	6·26
6·25	0·40	220	6·23
		121	6·22
		131	5·44
5·45	1·00	201	5·42
4·98	0·04	221	4·98
4·72	0·04	240	4·72
4·47	0·08	320	4·47
		002	4·14
4·11	1·00	151	4·11
		311	4·09
		321	3·93
3·94	0·20	112	3·93
		331	3·71
3·70	0·20	032	3·71
		251	3·68
		132	3·59
3·56	0·20	400	3·59
		341	3·45
3·44	0·04	420	3·45
		232	3·29
3·27	0·16	401	3·29
		171	3·20
3·20	0·20	351	3·19
		152	3·11
3·11	0·20	312	3·11

Table 1 (*cont.*)

d_o (Å)	$I/I_{max.}$	*hkl*	d_c from *a*, *b*, *c* (Å)
2·93	0·12	332	2·93
2·87	0·02	181	2·87
2·79	0·02	520	2·80
2·70	0·16	402	2·71
2·66	0·16	422	2·65
2·59	0·20	203	2·58
2·52	0·04	—	—
2·37	0·08	—	—
2·28	0·08	—	—
2·18	0·04	—	—
2·16	0·04	—	—
2·08	0·04	—	—
2·05	0·04	—	—
2·00	0·04	—	—
1·937	0·02	—	—
1·903	0·02	—	—
1·840	0·02	—	—
1·794	0·04	—	—
1·740	0·02	—	—
1·711	0·02	—	—

camera 114·6 mm. in diameter with copper radiation and nickel filter. A wavelength value of 1·540 Å was used in the calculations.

Reference

KEENAN, G. L. (1944). *J. Off. Agric. Chem.* **27**, 153.

Acta Cryst. (1958). **11**, 301

The unit cell and space group of thorium *tetrakis*-dibenzoylmethane.

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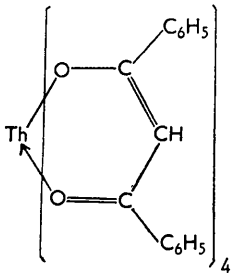
(*Received* 4 *October* 1957)

The preparation of thorium *tetrakis*-dibenzoylmethane (I) has been described elsewhere (Comyns, 1957). It crystal-



(I)

lized from toluene as pale yellow orthorhombic tablets showing straight extinction between crossed Nicols: elongated [001], tabular {100}.

The density was determined by flotation in aqueous K_2HgI_4.

Oscillation photographs, and zero-, first-, and second-layer equi-inclination Weissenberg photographs taken about the *c* axis confirmed the orthorhombic symmetry. Cu $K\alpha$ radiation (λ = 1·542 Å) was used.

The cell-dimensions were:

$$a = 20{\cdot}4 \pm 0{\cdot}1,\ b = 10{\cdot}33 \pm 0{\cdot}05,\ c = 23{\cdot}2 \pm 0{\cdot}1 \text{ Å}.$$

The calculated density, assuming 4 molecules per unit cell, is 1·53±0·01 g.cm.$^{-3}$; the experimental value was 1·52 g.cm.$^{-3}$. The following classes of reflexion were observed to be systematically absent: 0*kl*, $l \neq 2n$; *h*0*l*, $l \neq 2n$; *hk*0, $h+k \neq 2n$. Also, all reflexions *hkl* with $l \neq 2n$ were observed to be weak. The space group is thus *Pccn* (No. 56) and the thorium atoms lie in the fourfold special positions (*d*) or (*c*), these differing only in the choice of origin. The point symmetry of these positions is 2, and the molecules therefore each possess a twofold axis. This sheds little light on the stereochemistry of eight-fold coordination (Marchi, Fernelius & McReynolds, 1943; Nyholm, 1954), since most of the possible models have twofold axes.

No further work on this compound is contemplated.

References

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