

Short Communications

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The coefficient of thermal expansion of zirconium nitride. By T. W. BAKER, *Metallurgy Division, Atomic Energy Research Establishment, Harwell, Didcot, Berkshire, England*

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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

$$\begin{aligned} &4.5745 \pm 0.001 \text{ \AA} \text{ at } 17^\circ \text{ C.}, \\ &4.5865 \pm 0.001 \text{ \AA} \text{ at } 445^\circ \text{ C.}, \\ &4.5965 \pm 0.001 \text{ \AA} \text{ at } 680^\circ \text{ C.} \end{aligned}$$

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

$$\begin{aligned} &6.0 \pm 0.5 \times 10^{-6} \text{ C.}^{-1}, \text{ temperature range } 17\text{--}445^\circ \text{ C.}; \\ &7.0 \pm 0.5 \times 10^{-6} \text{ C.}^{-1}, \text{ temperature range } 17\text{--}680^\circ \text{ C.} \end{aligned}$$

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

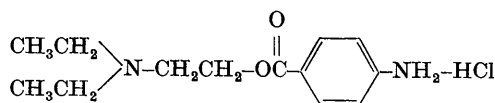
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Crystallographic properties of procaine hydrochloride. By HARRY A. ROSE, *Eli Lilly and Company, Indianapolis, Indiana, U.S.A.*

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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 (floatation), while the density calculated from X-ray data is 1.220 g.cm.⁻³. The unit-cell dimensions are:

$$a_0 = 14.35, \quad b_0 = 25.04, \quad c_0 = 8.28 \text{ \AA}.$$

The optical properties are:

$\alpha = 1.540$, $\beta = 1.564$, $\gamma > 1.70$ (all at 25° C., 5893 Å); (+)2*V* = 37°. The optic plane is 001, $\alpha = a$. Keenan (1944) gives $\alpha = 1.540$, $\beta = 1.566$, $\gamma > 1.690$.

The powder data (Table 1) were obtained using a