

At present, the evidence for  $D^+$  order is inconclusive; and a further neutron powder study combined with MASNMR of a carefully deammoniated sample of Linde Y-zeolite is planned in an attempt to resolve this point.

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#### References

- BAUR, W. H. (1964). *Am. Mineral.* **49**, 697–704.
- BEYER, H. K. & BELENYKAJA, I. (1980). *Catalysis in Zeolites*, edited by B. IMELIK, C. NACCACHE, Y. BEN TAARIT, J. C. VEDRINE, G. LOUDURIER & H. PRALIAUD, pp. 203–210. Amsterdam: Elsevier.
- BOSÁČEK, V., BERAN, S. & JIRÁK, Z. (1981). *J. Phys. Chem.* **85**, 3856–3859.
- BRECK, D. W. & FLANIGEN, E. M. (1968). *Molecular Sieves*, pp. 47–60. London: The Chemical Society.
- FINGER, L. W. & PRINCE, E. (1975). *Natl Bur. Stand. (US) Tech. Note* No. 854.
- GALLEZOT, P., BEAUMONT, R. & BARTHOMEUF, D. (1974). *J. Phys. Chem.* **78**(15), 1550–1553.
- HEWAT, A. W. (1973). UK Atomic Energy Authority Research Group Report RLL 73/897 (unpublished).
- JIRÁK, Z., VRATISLAV, S. & BOSÁČEK, V. (1980). *J. Phys. Chem. Solids*, **41**, 1089–1095.
- JIRÁK, Z., VRATISLAV, S., ZAJÁČEK, J. & BOSÁČEK, V. (1977). *J. Catal.* **49**, 112–114.
- JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794, revised. Oak Ridge National Laboratory, Oak Ridge, Tennessee.
- KLINOWSKI, J., THOMAS, J. M., FYFE, C. A. & GOBBI, G. C. (1982). *Nature (London)*, **296**, 533–536.
- KLINOWSKI, J., THOMAS, J. M., FYFE, C. A., GOBBI, G. C. & HARTMAN, J. S. (1983). *Inorg. Chem.* **22**, 63–66.
- KOESTER, L. & RAUCH, H. (1981). International Atomic Energy Agency, Contract 2517/RB (unpublished).
- MAXWELL, I. E., VAN ERP, W. A., HAYS, G. R., COUPERUS, T., HUIS, R. & CLAGUE, A. D. H. (1982). *J. Chem. Soc. Chem. Commun.* pp. 523–524.
- PLUTH, J. J. & SMITH, J. V. (1982). *J. Am. Chem. Soc.* **104**, 6977–6982.
- RIETVELD, H. M. (1969a). Reactor Centrum Netherland Research Report RCN 104 (unpublished).
- RIETVELD, H. M. (1969b). *J. Appl. Cryst.* **2**, 65–71.
- SCHERZER, J. (1978). *J. Catal.* **54**, 285–288.

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## Structure of $\text{Zr}_2\text{Be}_{17}$

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**Abstract.**  $M_r = 335.64$ , hexagonal,  $R\bar{3}m$ ,  $a = 7.538$  (1),  $c = 11.015$  (1) Å,  $V = 541.9$  (5) Å<sup>3</sup>,  $Z = 3$ ,  $D_x = 3.09$  g cm<sup>-3</sup>, Mo  $K\alpha$ ,  $\lambda = 0.71073$  Å,  $\mu = 27.1$  cm<sup>-1</sup>,  $F(000) = 444$ ,  $T = 295$  K,  $R = 0.020$  for 951 unique reflections, e.s.d. for an observation of unit weight = 1.054. The compound is isostructural with the analogous niobium compound [Zalkin, Sands & Krikorian (1959). *Acta Cryst.* **12**, 713–715] with all the atoms of the structure located on special positions. The Zr atoms are positioned in a barrel-shaped cavity defined by 18 Be atoms with a 19th Be atom and another Zr atom capping both ends of the cavity.

**Introduction.** Beryllium forms intermetallic compounds with most of the transition metals in a rich variety of combining ratios. Although the stoichiometric atomic ratios of the binary compounds are relatively simple, their crystal structures generally are more complex as indicated by high numbers of formula units per unit cell. The correspondingly large unit cells lead to relatively dense sampling of reciprocal space in a diffraction

experiment. The resulting dense set of structure factors is well suited to carry explicitly the subtle differences in valence electron density which distinguish different transition metals in the same structure type, or perhaps even the same transition metal in a variety of environments.

The current study is part of a systematic program of accurate structure determination of Be intermetallic compounds. This series of studies is designed to identify and evaluate compounds which could be used in detailed electron density analyses. In addition to the considerations already mentioned, other favorable properties of Be intermetallic compounds are their very small thermal parameters and low X-ray absorption coefficients when the Be:metal ratio is high. A tendency toward twinning is dealt with in these studies by careful photographic examination of each potential specimen crystal to ensure its suitability for data collection.

The title compound was originally studied as a powder by Zalkin, Bedford & Sands (1959). The present work is the single-crystal structure determination of  $\text{Zr}_2\text{Be}_{17}$ .

**Experimental.** Be and Zr mixed in 16:1 atomic ratio and melted under inert atmosphere in copper-hearth tungsten arc furnace. Zr-Be ingot formed was remelted in an attempt to ensure sample homogeneity. Several samples from crushed ingot examined by precession photographic techniques before suitable single crystal chosen for data collection. Irregularly shaped crystal (0.6 × 0.3 × 0.2 mm); Enraf-Nonius CAD-4 computer-automated diffractometer; lattice parameters from least-squares fit of 25 reflections in the range  $8 < \theta < 20^\circ$ ; empirical absorption correction (max. transmission coefficient = 0.998, min. = 0.866, av. = 0.954); data corrected for Lorentz and polarization effects; full shell of data for  $0 < \theta < 20^\circ$ ; in the range  $20 < \theta < 60^\circ$   $h, k \geq 0$ ;  $l$  data collected with reflections related by  $R$  centering systematically omitted ( $-7 \leq h \leq 15$ ,  $-7 \leq k \leq 15$ ,  $-26 \leq l \leq 26$ ); 3104 intensities measured (max.  $\sin\theta/\lambda = 1.22 \text{ \AA}^{-1}$ ) of which 1033 unique; root-mean-square variations in intensity for standard reflections are:  $\overline{426} = 1.2$ ;  $\overline{213} = 1.9$ ;  $\overline{213} = 2.5\%$ ; total loss in intensity = 0.6%;  $R_{\text{int}}$  for merging data = 0.033; 951 reflections observed based on  $I > 3\sigma(I)$ ; structure solved using heavy-atom technique; structure refined anisotropically using full-matrix weighted least squares [ $\omega = 1/\sigma^2(F)$ ] based on  $F$ ; scattering factors of Cromer & Waber (1974); convergence at  $R = 0.020$ ;  $R_w = 0.026$ ; max.  $\Delta/\sigma$  in final cycle of least squares  $< 0.01\sigma$ ; max. residual in final difference synthesis = 0.03, min. =  $-0.8 e \text{ \AA}^{-3}$ ; secondary-extinction parameter refined to  $6.6 (4) \times 10^{-6}$ ; anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974) applied to  $F_c$  during refinement but to  $F_o$  after refinement complete; all calculations performed using Enraf-Nonius SDP system of programs (Frenz, 1982).

**Discussion.** Refined atomic coordinates are listed in Table 1. Selected interatomic distances are given in Table 2.\*

There are no major structural differences from the niobium analog. The Zr atoms reside in a cage defined by 20 atoms; 19Be and 1Zr. Coordination about the Zr atom consists of three six-membered rings of Be atoms as shown in Fig. 1. Two of the Be rings are located above and below the Zr atoms along the  $z$  direction. The third, and somewhat larger ring of Be atoms, is located about the equator of the Zr cage. The Be rings found above and below the Zr atom are capped by a 19th Be atom on one end and another Zr atom at the opposite end, thus completing the 20-atom cage.

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\* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39462 (6 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Atomic positions for Zr<sub>2</sub>Be<sub>17</sub>

E.s.d.'s are given in parentheses.

		$x$	$y$	$z$	$B_{\text{eq}}^*(\text{\AA}^2)$
Zr	6(c)	0.0	0.0	0.16274 (1)	0.266 (1)
Be(1)	6(c)	0.0	0.0	0.4034 (2)	0.54 (2)
Be(2)	9(e)	0.1667	0.3333	0.3333	0.98 (2)
Be(3)	18(h)	0.16563 (9)	$-x$	$-0.0082 (1)$	0.48 (1)
Be(4)	18(g)	0.2875 (2)	$x$	0.5	0.52 (1)

$$* B_{\text{eq}} = \frac{4}{3}(a^2\beta_{11} + b^2\beta_{22} + c^2\beta_{33} + ab\beta_{12}\cos\gamma + ac\beta_{13}\cos\beta + bc\beta_{23}\cos\alpha).$$

Table 2. Interatomic distances ( $\text{\AA}$ ) for Zr<sub>2</sub>Be<sub>17</sub>

E.s.d.'s are given in parentheses.

Zr-Zr	3.585 (1)	Be(3)-1Zr	2.867 (1)
Zr-1Be(1)	2.652 (2)	Be(3)-1Zr'	2.752 (1)
Zr-3Be(2)	2.875 (1)	Be(3)-1Zr''	2.827 (1)
Zr-3Be(3)	2.868 (1)	Be(3)-1Be(1)	2.353 (1)
Zr-3Be(3')	2.752 (1)	Be(3)-2Be(2)	2.188 (1)
Zr-3Be(3'')	2.827 (1)	Be(3)-2Be(3)	2.170 (1)
Zr-6Be(4)	2.702 (1)	Be(3)-2Be(4)	2.232 (1)
Be(1)-1Zr	2.652 (2)	Be(3)-2Be(4')	2.369 (1)
Be(1)-1Be(1)	2.126 (5)	Be(4)-2Zr	2.702 (1)
Be(1)-3Be(2)	2.309 (1)	Be(4)-2Be(1)	2.413 (1)
Be(1)-3Be(3)	2.353 (1)	Be(4)-2Be(2)	2.152 (1)
Be(1)-6Be(4)	2.413 (1)	Be(4)-2Be(3)	2.232 (1)
Be(2)-2Zr	2.875 (1)	Be(4)-2Be(3')	2.369 (1)
Be(2)-2Be(1)	2.309 (1)	Be(4)-2Be(4)	2.167 (1)
Be(2)-4Be(3)	2.185 (1)		
Be(2)-4Be(4)	2.152 (1)		

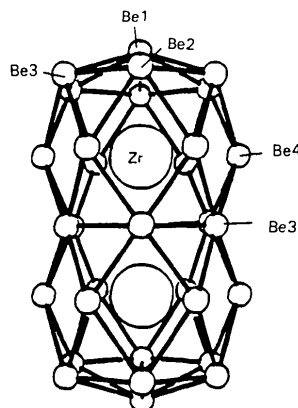


Fig. 1. Coordination cage of Zr in Zr<sub>2</sub>Be<sub>17</sub>.

## References

- CROMER, D. T. & WABER, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.2B. Birmingham: Kynoch Press.
- FRENZ, B. A. (1982). *Enraf-Nonius Structure Determination Package*. Enraf-Nonius, Delft.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press.
- ZALKIN, A., BEDFORD, R. G. & SANDS, D. E. (1959). *Acta Cryst.* **12**, 700.
- ZALKIN, A., SANDS, D. E. & KRİKORIAN, O. H. (1959). *Acta Cryst.* **12**, 713-715.