## References

Chen, B.-H. & Eichhorn, B. (1991). *Mater. Res. Bull.* **26**, 1035–1040.
Chen, B.-H., Eichhorn, B. & Fanwick, P. (1992). *Inorg. Chem.* **31**, 1788–1791.
Chen, B.-H., Eichhorn, B. W. & Wong-Ng, W. (1994). *Acta Cryst.* C**50**, 161–164.
Chen, B.-H., Wong-Ng, W. & Eichhorn, B. W. (1993). *J. Solid State Chem.* **103**, 75–84.
Eichhorn, B. W. (1994). *Progress in Inorganic Chemistry*, Vol. 42, edited by K. D. Karlin, 139–237, see p. 200. New York: John Wiley & Sons.
Enraf–Nonius (1994). *CAD*-4 *EXPRESS*. Version 5.1/1.2. Enraf–Nonius, Delft, The Netherlands.
Harms, K. (1993). *XCAD*-4. *Program for the Lp Correction of Nonius CAD*-4 *Diffractometer Data*. University of Marburg, Germany.
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Ruddlesden, S. N. & Popper, P. (1957). *Acta Cryst.* **10**, 538–539.
Ruddlesden, S. N. & Popper, P. (1958). *Acta Cryst.* **11**, 54–55.
Saeki, M., Yajima, Y. & Onoda, M. (1991). *J. Solid State Chem.* **92**, 286–294.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1993). *SHELXL*93. *Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.
Sheldrick, G. M. (1994). *SHELXTL. Structure Determination Programs*. Version 5.03. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

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# Hexa-$\mu$-nitrato-$\mu_4$-oxo-tetraberyllium

Martin J. Haley,† Stephen C. Wallwork,* Bryan Duffin, Norman Logan and C. Clifford Addison†

*Department of Chemistry, The University of Nottingham, University Park, Nottingham NG7 2RD, England. E-mail: s.c.wallwork@nott.ac.uk*

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## Abstract

Crystal structure determination of the title compound, [$Be_4O(NO_3)_6$], confirms the expectation of a molecular structure similar to that of basic beryllium acetate [Tulinsky & Worthington (1959). *Acta Cryst.* **12**, 626–634].

## Comment

The basic nitrate is the first crystallographically characterized nitrato–metal complex in which all nitrate ligands within the (tetranuclear) molecule are bridging bidentate (*syn–syn*) (Addison, Logan, Wallwork & Garner, 1971).

Each molecule lies on a crystallographic threefold axis in the cubic unit cell. This imposes just one threefold axis on the molecule but it is found that each has full tetrahedral symmetry within experimental error. A central O atom (lying approximately at a quarter or three-quarters of the way along a body diagonal of the cell) is surrounded by a tetrahedron of Be atoms, each pair of which is bridged in a bidentate manner by a planar nitrate group, resulting in the expected tetrahedral coordination of each Be. However, the plane of each such nitrate group is slightly twisted and folded relative to the line of the two bridged Be atoms (clockwise, when viewed along the terminal N—O bond towards the centre of the tetrahedron), as shown in Fig. 1. This permits a reduction in the Be—O—N angles which, nevertheless, are all still greater than 120°.

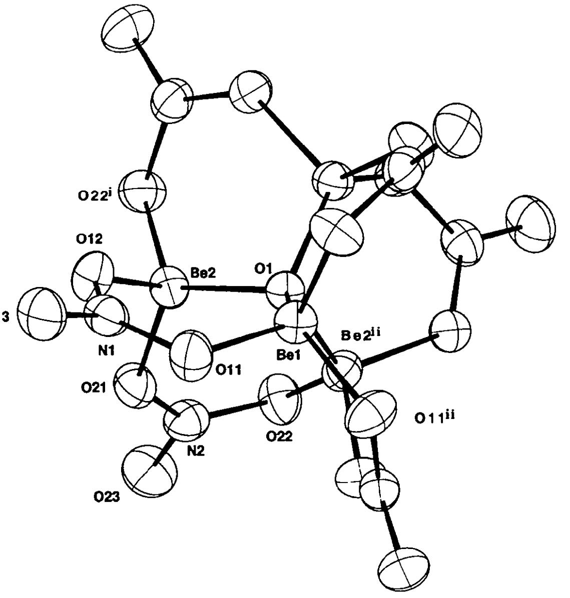

Fig. 1. The molecule of basic beryllium nitrate with displacement ellipsoids drawn at the 50% probability level. Symmetry codes: (i) *z*, *x*, *y*; (ii) *y*, *z*, *x*.

The eight molecules in the cell are centred near the eight permutations of 1/4 and 3/4 in groups of three. If they were exactly at these positions and all had the same orientation, the arrangement would correspond to simple cubic with a repeat distance equal to half of the actual cell dimension. The strength of some of the reflections with odd indices is an indication of the distortion from that arrangement. The two molecules situated at approximately 1/4 and 3/4 of the way along each body diagonal are centrosymmetrically related to each other, so the three nitrate planes on the molecule at 1/4 that face the corresponding three on the molecule at 3/4 along the same diagonal are staggered relative to each other. This must allow closer packing than if they were eclipsed and, combined with the overall molecular shape, results in an atomic pattern very different from a translational repeat.

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† Deceased.

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## Experimental

The title compound was obtained as a colourless crystalline solid (Addison & Walker, 1961) when the 2:1 dinitrogen tetraoxide adduct of beryllium nitrate was thermally decomposed at 398 K in a static vacuum. A crystal sublimed at 398 K onto a cold finger at 288 K under a static vacuum of $10^{-3}$ mm Hg (1.333 Pa) was sealed into a 0.3 mm diameter glass capillary under dry nitrogen in view of its hygroscopic nature.

*Crystal data*

[$Be_4O(NO_3)_6$]
$M_r = 424.1$
Cubic
$Pa\bar{3}$
$a = 14.025\,(2)$ Å
$V = 2759\,(1)$ Å$^3$
$Z = 8$
$D_x = 2.04$ Mg m$^{-3}$
$D_m = 2.0\,(1)$ Mg m$^{-3}$
$D_m$ measured by flotation in tetrabromoethane/tetrachloroethane

Cu $K\alpha$ radiation
$\lambda = 1.54184$ Å
Cell parameters from 12 reflections
$\theta = 20$–$40°$
$\mu = 2.01$ mm$^{-1}$
$T = 293$ K
Irregular
0.3 × 0.3 × 0.2 mm
Colourless

*Data collection*

Hilger & Watts four-circle diffractometer
$\omega$–$2\theta$ scans
Absorption correction: none
3183 measured reflections
992 independent reflections
748 reflections with $I > 3\sigma(I)$

$R_{\rm int}$ not recorded
$\theta_{\rm max} = 75.5°$
$h = 1 \rightarrow 17$
$k = 0 \rightarrow 12$
$l = 0 \rightarrow 11$
3 standard reflections every 100 reflections intensity decay: none

*Refinement*

Refinement on $F$
$R = 0.047$
$wR = 0.025$
$S = 1.076$
748 reflections
88 parameters
$(\Delta/\sigma)_{\rm max} = 0.00025$

$\Delta\rho_{\rm max} = 0.28$ e Å$^{-3}$
$\Delta\rho_{\rm min} = -0.36$ e Å$^{-3}$
Extinction correction: none
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å$^2$)*

$$U_{\rm eq} = (U_iU_jU_k)^{1/3}.$$

| | $x$ | $y$ | $z$ | $U_{\rm eq}$ |
|---|---|---|---|---|
| O1 | 0.2668 (1) | $x$ | $x$ | 0.0286 (6) |
| Be1 | 0.1997 (2) | $x$ | $x$ | 0.033 (1) |
| Be2 | 0.2383 (2) | 0.2534 (3) | 0.3780 (2) | 0.033 (2) |
| N1 | 0.1211 (2) | 0.1158 (2) | 0.3434 (2) | 0.036 (1) |
| O11 | 0.1143 (1) | 0.1488 (1) | 0.2581 (1) | 0.042 (1) |
| O12 | 0.1809 (1) | 0.1556 (1) | 0.4006 (1) | 0.039 (1) |
| O13 | 0.0734 (1) | 0.0498 (1) | 0.3689 (2) | 0.046 (1) |
| N2 | 0.1938 (2) | 0.4283 (2) | 0.4033 (2) | 0.036 (1) |
| O21 | 0.1734 (1) | 0.3404 (1) | 0.4187 (1) | 0.039 (1) |
| O22 | 0.2475 (1) | 0.4481 (1) | 0.3308 (1) | 0.039 (1) |
| O23 | 0.1644 (2) | 0.4899 (2) | 0.4539 (1) | 0.050 (1) |

Table 2. *Selected geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| O1—Be1 | 1.629 (6) | Be2—O12 | 1.621 (4) |
| O1—Be2 | 1.622 (3) | Be2—O21 | 1.625 (4) |
| Be1—O11 | 1.617 (3) | Be2—O22$^{\rm i}$ | 1.631 (4) |
| Be1—O1—Be2 | 110.3 (2) | O1—Be2—O22$^{\rm i}$ | 112.9 (2) |
| Be2—O1—Be2$^{\rm ii}$ | 108.7 (2) | O12—Be2—O22$^{\rm i}$ | 103.6 (2) |
| O1—Be1—O11 | 113.0 (2) | O21—Be2—O22$^{\rm i}$ | 105.8 (2) |
| O11—Be1—O11$^{\rm ii}$ | 105.8 (2) | Be1—O11—N1 | 125.1 (2) |
| O1—Be2—O12 | 114.1 (2) | Be2—O12—N1 | 124.6 (2) |
| O1—Be2—O21 | 122.9 (2) | Be2—O21—N2 | 122.5 (2) |
| O12—Be2—O21 | 106.8 (2) | Be2$^{\rm ii}$—O22—N2 | 121.6 (2) |

Symmetry codes: (i) $z, x, y$; (ii) $y, z, x$.

After preliminary examination by oscillation and Weissenberg photography, the crystal was oriented on the diffractometer and reflections collected with peak counts for 30 s and background counts for 15 s. Intensities were corrected for Lorentz and polarization effects but not for absorption. The direct methods used for structure solution had no routines for cubic symmetry, so that for the lower orthorhombic symmetry *Pbca* was used, having the same translational symmetry elements but not the threefold axis of the true space group $Pa\bar{3}$. The threefold symmetry was recovered by ensuring equal intensities for reflections *hkl*, *klh*, *lhk*. An *E* map for the solution with the highest figure of merit showed all the expected atoms. Refinement was carried out in the true space group $Pa\bar{3}$, with O1 and Be1 in special positions on the threefold axis. Full-matrix weighted least-squares refinement with anisotropic displacement parameters was used for the final cycles. A five-term Chebychev polynomial (Carruthers & Watkin, 1979) with coefficients 0.944, −2.56, −0.324, −1.34 and −0.145 was used as the weighting scheme.

Data collection: Hilger & Watts diffractometer software. Cell refinement: Hilger & Watts diffractometer software. Data reduction: *MANTAP* (King, 1973). Program(s) used to solve structure: *MULTAN* (Germain, Main & Woolfson, 1971). Program(s) used to refine structure: *CRYSTALS* (Watkin, Prout, Carruthers & Betteridge, 1996). Molecular graphics: *CAMERON* (Watkin, Prout & Pearce, 1996). Software used to prepare material for publication: local programs.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: JZ1166). Services for accessing these data are described at the back of the journal.

## References

Addison, C. C., Logan, N., Wallwork, S. C. & Garner, C. D. (1971). *Chem. Soc. Q. Rev.* **25**, 289–322.

Addison, C. C. & Walker, A. (1961). *Proc. Chem. Soc.* p. 242.

Carruthers, J. R. & Watkin, D. J. (1979). *Acta Cryst.* **A35**, 698–699.

Germain, G., Main, P. & Woolfson, M. M. (1971). *Acta Cryst.* **A27**, 368–376.

King, T. J. (1973). *MANTAP. Data Reduction Program.* University of Nottingham, England.

Watkin, D. J., Prout, C. K., Carruthers, J. R. & Betteridge, P. W. (1996). *CRYSTALS*. Issue 10. Chemical Crystallography Laboratory, University of Oxford, England.

Watkin, D. J., Prout, C. K. & Pearce, L. J. (1996). *CAMERON*. Chemical Crystallography Laboratory, University of Oxford, England.