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# $RbGd(PO_3)_4$

**Hasna Ettis, Houcine Naïli* and Tahar Mhiri**

Laboratoire de l'État Solide, Département de Chimie, Faculté des Sciences de Sfax, BP 802, 3018 Sfax, Tunisia

Correspondence e-mail: houcine_naili@yahoo.com

**Key indicators**

Single-crystal X-ray study
$T$ = 298 K
Mean $\sigma$(P–O) = 0.004 Å
$R$ factor = 0.023
$wR$ factor = 0.056
Data-to-parameter ratio = 13.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.



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Single crystals of rubidium gadolinium polyphosphate were grown from a polyphosphate flux. The structure of the title compound is isotypic with $CsGd(PO_3)_4$ and consists of helical polyphosphate chains running along the [101] direction with a period of eight $PO_4$ tetrahedra. These polyphosphate chains are connected by isolated $GdO_8$ dodecahedra and irregularly shaped $RbO_{11}$ polyhedra, forming a three-dimensional framework.

## Comment

The structures of condensed phosphates of monovalent and rare earth metals corresponding to the general formula $M^{I}RE^{III}(PO_3)_4$ (where $M^{I}$ = alkali metal or $NH_4^+$, $RE^{III}$ = rare earth) are well known (Hong, 1975*a*,*b*; Koizumi, 1976; Masse *et al.*, 1977; Palkina *et al.*, 1979; Ferid *et al.*, 1987; Jaouadi *et al.*, 2003, 2005; Ettis *et al.*, 2003; Rekik *et al.*, 2004; El Masloumi *et al.*, 2005; Naïli & Mhiri, 2005, and references therein). At room temperature, the corresponding representatives exhibit mainly two different structure types for the cyclotetraphosphates, $P_4O_{12}^{4-}$, in the space groups $C2/c$ and $I\bar{4}3d$, and for the polyphosphates, $(PO_3)_4^{4-}$, seven different structure types with different space groups are known (the structure type is denoted in roman numerals): $C2/c$ (I), $P2_1/n$ (II), $P2_1$ (III), $P2_1/n$ (IV), $P2_1/n$ (V), $P2_1$ (VI), and $C222_1$ (VII). We report here a new rubidium gadolinium polyphosphate, $RbGd(PO_3)_4$, which is isotypic with its caesium homologue, $CsGd(PO_3)_4$ (Naïli & Mhiri, 2005).

The asymmetric unit of the title compound contains one Gd, one Rb, four P and 12 O atoms (Fig. 1). The basic structural features are two helical polyphosphate chains, formed by corner-sharing of $PO_4$ tetrahedra, which extend along the [101] direction with a period of eight tetrahedra (Fig. 2). Both

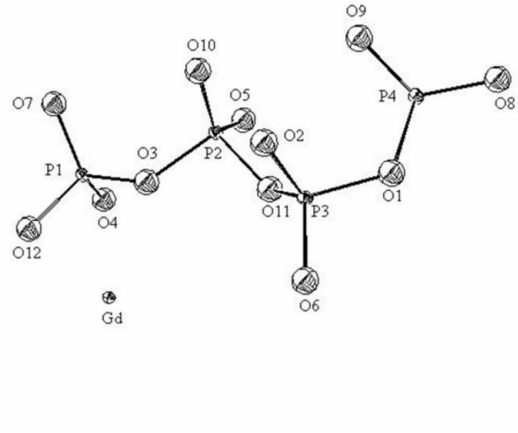


**Figure 1**
The asymmetric unit of the title compound, with anisotropic displacement parameters drawn at the 50% probability level.

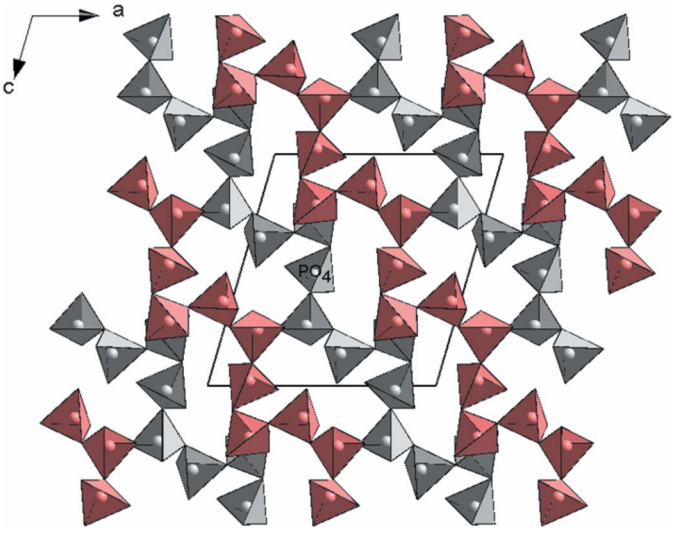


**Figure 2**
The two helical polyphosphate chains in the structure of $RbGd(PO_3)_4$.

chains are related by $2_1$ symmetry and are interconnected by $GdO_8$ dodecahedra, leading to a three-dimensional framework structure with tunnels in which the $Rb^+$ cations are located (Fig. 3). The $GdO_8$ dodecahedra are considerably distorted and isolated from each other in the sense that they do not share a common O atom, in contrast to the related structures of $KGd(PO_3)_4$ (Rekik *et al.*, 2004) or $KGdP_4O_{12}$ (Ettis *et al.*, 2003). The shortest Gd···Gd distance in the title compound is 5.737 (3) Å. The $RbO_{11}$ polyhedra are very irregular, as shown by the Rb—O distances (Table 1). They share two O atoms to form dimers which are arranged in rows parallel to the [001] direction.

## Experimental

The preparation of single crystals of $RbGd(PO_3)_4$ was achieved by a polyphosphate flux prepared by heating a stoichiometric mixture of 1.86 g $H_3PO_4$ (85%$_{wt}$, Merck, pA), 0.26 g $Gd_2O_3$ (99.99%, Merck) and 2.14 g $RbH_2PO_4$. The latter was obtained from an aqueous solution containing $Rb_2CO_3$ (98.9%, Merck) and $H_3PO_4$ (85%$_{wt}$, Merck) in an Rb:*P* molar ratio of 1:2. Slow evaporation of water under ambient conditions yielded crystals after 3 to 5 d. Their composition was confirmed by X-ray powder diffraction. The reaction mixture was heated in a Pt crucible at a temperature of 473 K for 4 h. The temperature was then increased progressively at the rate of 2 K $min^{-1}$ up to 823 K, kept there for 2 d and then cooled to 323 K at a rate of 40 K $d^{-1}$. The resulting crystals were washed with warm water and nitric acid to dissolve the remaining $Gd_2O_3$. Colourless and transparent crystals with a truncated hexagonal pyramidal habit were obtained.

*Crystal data*

$RbGd(PO_3)_4$
$M_r$ = 558.60
Monoclinic, $P2_1/n$
$a$ = 10.385 (2) Å
$b$ = 8.976 (2) Å
$c$ = 11.008 (2) Å
$\beta$ = 106.2 (1)°
$V$ = 985.4 (6) Å$^3$
$Z$ = 4
$D_x$ = 3.765 Mg m$^{-3}$
Mo $K\alpha$ radiation
$\mu$ = 12.35 mm$^{-1}$
$T$ = 298 (2) K
Truncated hexagonal pyramid, colourless
0.18 × 0.18 × 0.14 mm

*Data collection*

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: $\psi$ scan (North *et al.*, 1968)
$T_{min}$ = 0.129, $T_{max}$ = 0.178
2537 measured reflections
2137 independent reflections
1878 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.024
$\theta_{max}$ = 27.0°
2 standard reflections
frequency: 60 min
intensity decay: 1.3%

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)]$ = 0.023
$wR(F^2)$ = 0.056
$S$ = 1.07
2137 reflections
164 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0237P)^2 + 1.2148P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.79 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.78 e Å$^{-3}$
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0152 (4)

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| Gd—O9$^{i}$ | 2.325 (3) | Rb—O12$^{i}$ | 3.485 (4) |
| Gd—O4 | 2.368 (3) | P1—O7 | 1.481 (4) |
| Gd—O10$^{i}$ | 2.379 (3) | P1—O4 | 1.491 (4) |
| Gd—O5$^{ii}$ | 2.401 (3) | P1—O12 | 1.589 (3) |
| Gd—O7$^{iii}$ | 2.416 (3) | P1—O3 | 1.611 (4) |
| Gd—O6$^{iv}$ | 2.417 (3) | P2—O10 | 1.485 (4) |
| Gd—O8$^{v}$ | 2.430 (3) | P2—O5 | 1.493 (4) |
| Gd—O2$^{i}$ | 2.473 (3) | P2—O3 | 1.605 (4) |
| Rb—O4$^{iii}$ | 2.925 (3) | P2—O11 | 1.611 (3) |
| Rb—O5$^{vi}$ | 2.971 (4) | P3—O6 | 1.487 (3) |
| Rb—O6$^{vii}$ | 2.986 (3) | P3—O2 | 1.493 (3) |
| Rb—O2$^{vii}$ | 3.016 (3) | P3—O1 | 1.610 (3) |
| Rb—O10$^{i}$ | 3.139 (3) | P3—O11 | 1.616 (3) |
| Rb—O3$^{i}$ | 3.212 (4) | P4—O9 | 1.488 (4) |
| Rb—O7$^{iii}$ | 3.287 (4) | P4—O8 | 1.494 (4) |
| Rb—O11$^{vi}$ | 3.330 (3) | P4—O1 | 1.608 (3) |
| Rb—O8$^{v}$ | 3.458 (4) | P4—O12$^{viii}$ | 1.616 (4) |
| Rb—O7$^{i}$ | 3.462 (4) | | |
| O7—P1—O4 | 118.4 (2) | O2—P3—O1 | 111.54 (19) |
| O7—P1—O12 | 109.3 (2) | O6—P3—O11 | 108.84 (19) |
| O4—P1—O12 | 110.5 (2) | O2—P3—O11 | 108.66 (19) |
| O7—P1—O3 | 108.2 (2) | O1—P3—O11 | 102.32 (18) |
| O4—P1—O3 | 110.2 (2) | O9—P4—O8 | 118.6 (2) |
| O12—P1—O3 | 98.29 (19) | O9—P4—O1 | 107.94 (19) |
| O10—P2—O5 | 121.5 (2) | O8—P4—O1 | 110.36 (19) |
| O10—P2—O3 | 107.79 (19) | O9—P4—O12$^{viii}$ | 109.3 (2) |
| O5—P2—O3 | 109.6 (2) | O8—P4—O12$^{viii}$ | 110.4 (2) |
| O10—P2—O11 | 111.13 (19) | O1—P4—O12$^{viii}$ | 98.33 (19) |
| O5—P2—O11 | 105.99 (19) | P3—O1—P4 | 125.1 (2) |
| O3—P2—O11 | 98.42 (18) | P2—O3—P1 | 129.7 (2) |
| O6—P3—O2 | 117.1 (2) | P2—O11—P3 | 131.7 (2) |
| O6—P3—O1 | 107.32 (19) | P1—O12—P4$^{v}$ | 134.4 (2) |

Symmetry codes: (i) $x, y+1, z$; (ii) $-x+1, -y, -z$; (iii) $-x+\frac{3}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$; (iv) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$; (v) $x+\frac{1}{2}, -y-\frac{1}{2}, z+\frac{1}{2}$; (vi) $x+\frac{1}{2}, -y+\frac{1}{2}, z+\frac{1}{2}$; (vii) $-x+1, -y, -z+1$; (viii) $x-\frac{1}{2}, -y-\frac{1}{2}, z-\frac{1}{2}$.

For better comparison with the isotypic $CsGd(PO_3)_4$ (Naïli & Mhiri, 2005), the final atomic coordinates and the atomic labels were converted to correspond to those of the Cs compound.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *MolEN* (Fair, 1990); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg & Berndt, 2001) and *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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## References


Brandenburg, K. & Berndt, M. (2001). *DIAMOND*. Release 2.1e. Crystal Impact GbR, Bonn, Germany.

El Masloumi, M., Imaz, I., Chaminade, J. P., Videau, J. J., Couzi, M., Mesnaoui, M. & Maazaz, M. (2005). *J. Solid State Chem.* **178**, 3581–3588.

Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.

Ettis, H., Naïli, H. & Mhiri, T. (2003). *Cryst. Growth Des.* **3**, 599–602.

Fair, C. K. (1990). *MolEN*. Enraf–Nonius, Delft, The Netherlands.

Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.

Ferid, M., Ariguib, N. K. & Trabelsi, M. (1987). *J. Solid State Chem.* **69**, 1–9.

Hong, H. Y. (1975*a*). *Mater. Res. Bull.* **10**, 635–640.

Hong, H. Y. (1975*b*). *Mater. Res. Bull.* **10**, 1105–1110.

Jaouadi, K., Naïli, H., Zouari, N., Mhiri, T. & Daoud, A. (2003). *J. Alloys Compds*, **354**, 104–114.

Jaouadi, K., Zouari, N., Mhiri, T. & Pierrot, M. (2005). *J. Cryst. Growth*, **273**, 638–645.

Koizumi, H. (1976). *Acta Cryst.* B**32**, 2254–2256.

Masse, R., Guitel, J.-C. & Durif, A. (1977). *Acta Cryst.* B**33**, 630–632.

Naïli, H. & Mhiri, T. (2005). *Acta Cryst.* E**61**, i204–i207.

North, A. C. T., Philips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.

Palkina, K. K., Maksimova, S. I., Chudinova, N. N., Vinogradova, N. V. & Chibiskova, N. T. (1979). *Izv. Akad. Nauk. SSSR, Neorg. Mater.* **17**, 110–117. (In Russian.)


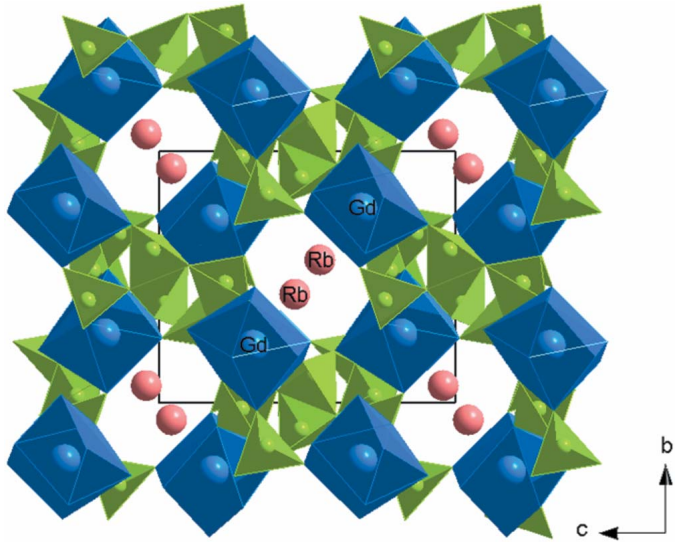


**Figure 3**
The $RbGd(PO_3)_4$ crystal structure in a projection on the *bc* plane. The polyphosphate chains are shown as green polyhedra.


Rekik, W., Naïli, H. & Mhiri, T. (2004). *Acta Cryst.* C**60**, i50–i52.

Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.