$S = 1.169$
496 reflections
41 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0181P)^2 + 0.3376P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.885$ e Å$^{-3}$
$\Delta\rho_{\min} = -0.627$ e Å$^{-3}$
Extinction coefficient: 0.0141 (10)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

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

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

| | $x$ | $y$ | $z$ | $U_{\rm eq}$ |
|---|---|---|---|---|
| As1 | 0.40949 (5) | 0.03079 (5) | 1/4 | 0.0068 (2) |
| Ca1 | 1/3 | 2/3 | 0.0050 (2) | 0.0111 (2) |
| Ca2 | 0.26988 (10) | 0.24976 (10) | 1/4 | 0.0105 (2) |
| Cl1† | 0 | 0 | 0.1263 (5) | 0.0218 (6) |
| O1 | 0.1559 (4) | 0.5081 (4) | 0.2500 | 0.0144 (7) |
| O2 | 0.5347 (4) | 0.3966 (3) | 0.2500 | 0.0116 (6) |
| O3 | 0.3511 (3) | 0.0892 (3) | 0.0535 (4) | 0.0174 (5) |

† Site occupancy = 0.5.

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

| | | | |
|---|---|---|---|
| As1—O1$^{\rm i}$ | 1.669 (3) | Ca1—O3$^{\rm vi}$ | 2.915 (2) |
| As1—O3$^{\rm ii}$ | 1.681 (2) | Ca1—O3$^{\rm viii}$ | 2.915 (2) |
| As1—O3 | 1.681 (2) | Ca1—O3$^{\rm vii}$ | 2.915 (2) |
| As1—O2$^{\rm iii}$ | 1.696 (3) | Ca2—O2 | 2.316 (3) |
| Ca1—O1$^{\rm iv}$ | 2.382 (2) | Ca2—O3$^{\rm ix}$ | 2.324 (2) |
| Ca1—O1 | 2.382 (2) | Ca2—O3$^{\rm vii}$ | 2.324 (2) |
| Ca1—O1$^{\rm v}$ | 2.382 (2) | Ca2—O3 | 2.530 (2) |
| Ca1—O2$^{\rm vi}$ | 2.456 (2) | Ca2—O3$^{\rm ii}$ | 2.530 (2) |
| Ca1—O2$^{\rm vii}$ | 2.456 (2) | Ca2—Cl1 | 2.7557 (14) |
| Ca1—O2$^{\rm viii}$ | 2.456 (2) | Ca2—Cl1$^{\rm ii}$ | 2.7557 (14) |

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

The intensities were corrected for the anisotropic part of the absorption using a $\psi$ scan, however, for logistical reasons, it was impossible to apply the spherical absorption correction subsequently requested by the editor.

Data collection: *TEXSAN* (Molecular Structure Corporation, 1989). Cell refinement: *TEXSAN*. Data reduction: *TEXSAN*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1991). Software used to prepare material for publication: Microsoft *WORD*5.1a.

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Lists of structure factors, anisotropic displacement parameters and complete geometry have been deposited with the IUCr (Reference: BR1116). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

## References

Elliott, J. C. (1994). In *Structure and Chemistry of the Apatites and Other Calcium Orthophosphates*. Amsterdam: Elsevier.

Kay, I. M., Young, R. A. & Posner, A. S. (1964). *Nature*, **204**, 1050–1052.

Kreidler, E. R. & Hummel, F. A. (1970). *Am. Mineral.* **55**, 170–184.

Mackie, P. E., Elliott, J. C. & Young, R. A. (1972). *Acta Cryst.* B**28**, 1840–1848.

Molecular Structure Corporation (1989). *TEXSAN. Single Crystal Structure Analysis Software*. Version 5.0. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Prener, J. S. (1967). *J. Electrochem. Soc.* **114**, 77–83.

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.

Sheldrick, G. M. (1991). *SHELXTL-Plus*. Release 4.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.

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# A Mixed Nickel–Lithium Pyrophosphate, $LiNi_{1.5}P_2O_7$

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## Abstract

The title compound, dilithium trinickel bis(diphosphate), is composed of $[P_2O_7]$ layers parallel to the crystallographic plane (001), separated by chains of $[NiO_6]$ octahedra. The coordination polyhedra of the $Li^+$ ions link the $[P_2O_7]$ groups and the $[NiO_6]$ octahedra, and so ensure the cohesion of the structure.

## Comment

Work on mixed pyrophosphates $ABP_2O_7$ has shown that the structural type of these compounds depends upon the size of the $A$ and $B$ cations and the conformation of the $[P_2O_7]$ groups. With two cations of different size, particularly an alkaline earth cation and a transition metal cation, mixed pyrophosphates crystallize in one of three types of structure: cage (Riou, Labbe & Goreaud, 1988*b*), sheet (Riou, Labbe & Goreaud, 1988*a*; Moqine, Boukhari & Holt, 1991; Alaoui El Belghiti, Boukhari & Holt, 1991; Riou, Leligny, Pham, Labbe & Raveau, 1991) or tunnel (Riou & Goreaud, 1990; Murashova, Velikodnyi & Trunov, 1991; Riou & Raveau, 1991). When two different 3$d$ transition elements are present, the structure is composed of a succession of metal planes separated by layers of $[P_2O_7]$ groups. The conformation of the $P_2O_7$ groups is staggered and the

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structure is of the thortveitite type (Benkhouja, Zahir, Sadel, Drillon & Bernier, 1991).

Following previous work on mixed pyrophosphates of transition elements (*MM'*$P_2O_7$; Benkhouja, Sadel, Zahir, Legoll & Drillon, 1992; Benkhouja, Zahir, Sadel, Handizi, Boukhari, Holt, Aride & Drillon, 1995; Bettach, Benkhouja, Sadel, Zahir & Drillon, 1994), we present here the partial substitution of a transition element by an alkali metal (lithium) in order to discuss the crystallographic array of the cations in compounds of this type. These compounds are rarely mentioned in the literature (Erragh, Boukhari, Elouadi & Holt, 1991; Spirlet, Rebizant & Liegeois-Duyckaerts, 1993).

A projection of the structure on the (010) plane is shown in Fig. 1. The structure can be described as a framework composed of [$P_2O_7$] layers parallel to the crystallographic plane (001) and [$NiO_6$] octahedra grouped in trimers. The coordination polyhedra of the $Li^+$ ions link the [$P_2O_7$] groups to the [$NiO_6$] octahedra and so ensure the cohesion of the structure.

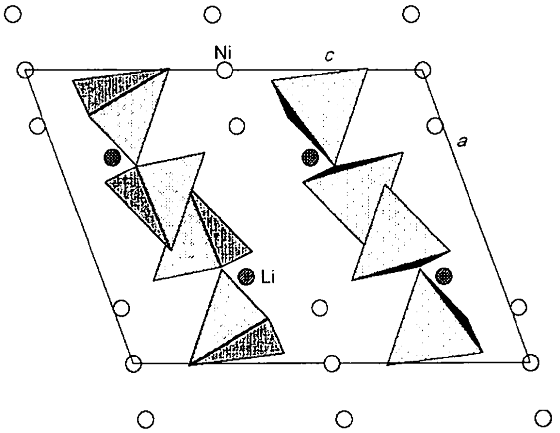

Fig. 1. A projection of the structure onto the (010) plane.

The conformation of the [$P_2O_7$] groups is staggered, justifying the attribution of these compounds to the thortveitite family (Cruickshank, Lynton & Barclay, 1962). The P—O—P angle is 134.10 (9)°, comparable with that found for the mixed nickel pyrophosphates $Sr_{0.5}Ni_{1.5}P_2O_7$ (134°) and $Pb_{0.5}Ni_{1.5}P_2O_7$ (134.4°) (El Bali, 1990; Krasnikov, Constant & Bel'skii, 1985). The O—P—O angles lie between 101.13 (8) and 115.68 (8)°. The lengths of the terminal P—O bonds are between 1.494 (1) and 1.537 (1) Å, and the average length of the P—O bridging bonds is 1.589 (2) Å. These distances are close to those given in the literature (Bauer, 1974).

The $Ni^{2+}$ ions display coordination number 6 and occupy two types of coordination site. The Ni1—O distances vary from 2.036 (1) to 2.237 (2) Å, while the Ni2—O distances lie between 2.030 (2) and 2.118 (1) Å, values comparable to those obtained for $\alpha$-$Ni_2P_2O_7$ (Lukaszewicz, 1967). The [$NiO_6$] octahedra, which share a common edge, form trimer chains in a zigzag arrangement running parallel to the *b* axis (Fig. 2).

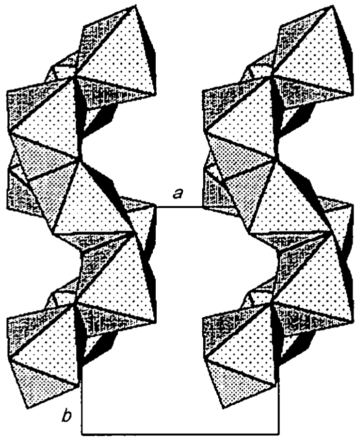

Fig. 2. Zigzag chains of trimers of [$NiO_6$] octahedra.

The lithium polyhedron is formed by four O atoms at distances between 1.910 (5) and 2.274 (5) Å. Two other O atoms, O6 and O2, are at greater distances: 2.491 (6) and 2.703 (4) Å, respectively. The maximum length of the Li—O bonds obtained using the Donnay & Allmann (1970) procedure and the radii given by Shannon (1976) is 2.67 Å. We can therefore consider as an initial approximation that lithium has fivefold rather than sixfold coordination.

The structure of $LiNi_{1.5}P_2O_7$ is related to that of $\alpha$-$Ni_2P_2O_7$ by replacement of the five-coordinate nickel site by lithium. The appearance of nickel in a special position may be explained by the formal substitution of half an $Ni^{2+}$ ion by one $Li^+$ ion. We may conclude that the sequence of $Ni^{2+}$ atoms in $\alpha$-$Ni_2P_2O_7$ (a planar system) has been formally disrupted by $Li^+$ polyhedra to give rise to trimers formed of zigzag strips of [$NiO_6$] octahedra (a linear system).

## Experimental

Crystals were prepared from NiO, $Li_2CO_3$ and $(NH_4)_2HPO_4$ taken in adequate proportions and heated progressively to 773 K. After decomposition of the carbonate and the phosphate, the mixture was melted at 1173 K and maintained at this temperature for 4 h. It was then cooled to 773K at a rate of about 2 K $h^{-1}$ and then to 473 K at 10 K $h^{-1}$. Below this temperature the oven was turned off.

*Crystal data*

$LiNi_{1.5}P_2O_7$
$M_r = 269.0$
Monoclinic
$P2_1/c$
$a = 7.189$ (1) Å
$b = 7.744$ (2) Å
$c = 9.331$ (2) Å
$\beta = 110.32$ (4)°

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 18$–22°
$\mu = 6.51$ $mm^{-1}$
$T = 298$ K
Sphere

$V = 487.2$ (1) Å$^3$
$Z = 4$
$D_x = 3.68$ Mg m$^{-3}$

0.08 mm (radius)
Yellow

*Data collection*

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: spherical
$T_{min} = 0.243$, $T_{max} = 0.272$
2384 measured reflections
2168 independent reflections

1917 observed reflections $[I > 3\sigma(I)]$
$R_{int} = 0.039$
$\theta_{max} = 30°$
$h = -11 \rightarrow 11$
$k = 0 \rightarrow 12$
$l = 0 \rightarrow 15$
3 standard reflections
frequency: 60 min
intensity decay: 1.2%

*Refinement*

Refinement on $F$
$R = 0.028$
$wR = 0.040$
$S = 1.521$
1917 reflections
110 parameters
$w = 4F_o^2/[\sigma^2(F_o^2) + 0.0016F_o^4]$
$(\Delta/\sigma)_{max} = 0.092$
$\Delta\rho_{max} = 0.62$ e Å$^{-3}$
$\Delta\rho_{min} = -0.56$ e Å$^{-3}$

Extinction correction: isotropic (Zachariasen, 1963)
Extinction coefficient: $0.86 \times 10^{-5}$
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

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

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

| | $x$ | $y$ | $z$ | $U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.3252 (2) | 0.1355 (2) | 0.8099 (2) | 0.0069 (3) |
| P1 | 0.11853 (6) | 0.20644 (6) | 0.80626 (5) | 0.00372 (8) |
| P2 | 0.40526 (6) | −0.05139 (6) | 0.78457 (5) | 0.00417 (8) |
| O2 | 0.0336 (2) | 0.0788 (2) | 0.8916 (2) | 0.0069 (3) |
| O3 | −0.0088 (2) | 0.2069 (2) | 0.6364 (2) | 0.0067 (3) |
| O4 | 0.6178 (2) | −0.0098 (2) | 0.8016 (2) | 0.0071 (3) |
| O5 | 0.3793 (2) | −0.1703 (2) | 0.9045 (2) | 0.0075 (3) |
| O6 | 0.2778 (2) | −0.1129 (2) | 0.6238 (2) | 0.0082 (3) |
| O7 | 0.1530 (2) | 0.3789 (2) | 0.8828 (2) | 0.0072 (3) |
| Ni1 | −0.18779 (3) | 0.12630 (3) | 0.98014 (3) | 0.00527 (5) |
| Ni2 | 0 | 0 | 1/2 | 0.00461 (6) |
| Li1 | −0.2942 (7) | 0.0810 (6) | 0.6371 (5) | 0.021 (1) |

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

| | | | |
|---|---|---|---|
| O1—P1 | 1.573 (2) | Ni1—O6$^{iv}$ | 2.237 (2) |
| O1—P2 | 1.605 (2) | Ni2—O3 | 2.061 (2) |
| P1—O2 | 1.524 (2) | Ni2—O3$^{v}$ | 2.061 (2) |
| P1—O3 | 1.530 (1) | Ni2—O6 | 2.118 (1) |
| P1—O7 | 1.494 (1) | Ni2—O6$^{v}$ | 2.118 (1) |
| P2—O4 | 1.515 (2) | Ni2—O7$^{vi}$ | 2.030 (2) |
| P2—O5 | 1.510 (2) | Ni2—O7$^{vii}$ | 2.030 (2) |
| P2—O6 | 1.537 (1) | Li1—O2 | 2.703 (4) |
| Ni1—O2 | 2.065 (2) | Li1—O3 | 2.274 (5) |
| Ni1—O2$^{i}$ | 2.065 (1) | Li1—O4$^{iii}$ | 1.981 (6) |
| Ni1—O3$^{ii}$ | 2.036 (1) | Li1—O5$^{iv}$ | 2.017 (5) |
| Ni1—O4$^{iii}$ | 2.055 (1) | Li1—O6$^{v}$ | 2.491 (6) |
| Ni1—O5$^{i}$ | 2.049 (2) | Li1—O7$^{vi}$ | 1.910 (5) |
| P1—O1—P2 | 134.10 (9) | O3—Ni2—O3$^{v}$ | 180 |
| O1—P1—O2 | 108.00 (8) | O3—Ni2—O6 | 102.09 (5) |
| O1—P1—O3 | 104.11 (9) | O3—Ni2—O6$^{v}$ | 77.91 (5) |
| O1—P1—O7 | 107.65 (8) | O3—Ni2—O7$^{vi}$ | 83.57 (6) |
| O2—P1—O3 | 109.80 (8) | O3—Ni2—O7$^{vii}$ | 96.43 (6) |
| O2—P1—O7 | 111.09 (9) | O3$^{v}$—Ni2—O6 | 77.91 (5) |
| O3—P1—O7 | 115.68 (8) | O3$^{v}$—Ni2—O6$^{v}$ | 102.09 (5) |
| O1—P2—O4 | 101.13 (8) | O3$^{v}$—Ni2—O7$^{vi}$ | 96.43 (6) |
| O1—P2—O5 | 107.33 (9) | O3$^{v}$—Ni2—O7$^{vii}$ | 83.57 (6) |
| O1—P2—O6 | 107.48 (7) | O6—Ni2—O6$^{v}$ | 180 |
| O4—P2—O5 | 115.59 (8) | O6—Ni2—O7$^{vi}$ | 95.74 (6) |
| O4—P2—O6 | 113.95 (9) | O6—Ni2—O7$^{vii}$ | 84.26 (6) |
| O5—P2—O6 | 110.42 (8) | O6$^{v}$—Ni2—O7$^{vi}$ | 84.26 (6) |
| O2—Ni1—O2$^{i}$ | 76.19 (6) | O6$^{v}$—Ni2—O7$^{vii}$ | 95.74 (6) |
| O2—Ni1—O3$^{ii}$ | 90.93 (6) | O7$^{vi}$—Ni2—O7$^{vii}$ | 180 |
| O2—Ni1—O4$^{iii}$ | 89.19 (6) | O3—Li1—O4$^{iii}$ | 133.30 (2) |
| O2—Ni1—O5$^{i}$ | 172.52 (5) | O3—Li1—O5$^{iv}$ | 78.68 (2) |
| O2—Ni1—O6$^{iv}$ | 97.72 (6) | O3—Li1—O6$^{v}$ | 66.75 (2) |
| O2$^{i}$—Ni1—O3$^{ii}$ | 89.68 (5) | O3—Li1—O7$^{vi}$ | 80.81 (2) |
| O2$^{i}$—Ni1—O4$^{iii}$ | 98.77 (5) | O4$^{iii}$—Li1—O5$^{iv}$ | 109.34 (3) |
| O2$^{i}$—Ni1—O5$^{i}$ | 98.45 (6) | O4$^{iii}$—Li1—O6$^{v}$ | 158.26 (2) |
| O3$^{ii}$—Ni1—O5$^{i}$ | 83.75 (6) | O4$^{iii}$—Li1—O7$^{vi}$ | 96.20 (2) |
| O3$^{ii}$—Ni1—O6$^{iv}$ | 75.72 (5) | O5$^{iv}$—Li1—O6$^{v}$ | 80.29 (2) |
| O4$^{iii}$—Ni1—O5$^{i}$ | 96.86 (6) | O5$^{iv}$—Li1—O7$^{vi}$ | 154.12 (3) |
| O4$^{iii}$—Ni1—O6$^{iv}$ | 95.66 (5) | O6$^{v}$—Li1—O7$^{vi}$ | 77.20 (2) |
| O2$^{i}$—Ni1—O6$^{iv}$ | 164.21 (5) | O2—Li1—O3 | 59.54 (1) |
| O3$^{ii}$—Ni1—O4$^{iii}$ | 171.32 (6) | O2—Li1—O4$^{iii}$ | 74.49 (1) |
| O5$^{i}$—Ni1—O6$^{iv}$ | 86.09 (6) | | |

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

The structure was solved using the Patterson function. Two Ni atoms were located, one in a general position and the second in the special position 0,0,1/2. Difference Fourier synthesis led to the location of the remaining sites for the P, O and Li atoms. Refinement was carried out by full-matrix least-squares methods. The value of $U_{eq}$ for the $Li^+$ ion [0.021 (1) Å$^2$] is about three times higher than the $U_{eq}$ values of the O atoms, which may indicate slight disorder in the position of the the $Li^+$ ion.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *SDP-Plus* (Frenz, 1985). Data reduction: *SDP-Plus*. Program(s) used to solve structure: *MULTAN*11/82 (Main *et al.*, 1982). Program(s) used to refine structure: *SDP-Plus*. Molecular graphics: *ORTEP*II (Johnson, 1976).

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Lists of structure factors and anisotropic displacement parameters have been deposited with the IUCr (Reference: JZ1112). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

## References

Alaoui El Belghiti, A., Boukhari, A. & Holt, E. M. (1991). *Acta Cryst.* C**47**, 473–477.

Bauer, W. H. (1974). *Acta Cryst.* B**30**, 1195–1215.

Benkhouja, K., Sadel, A., Zahir, M., Legoll, P. & Drillon, M. (1992). *J. Alloys Compd.* **188**, 230–233.

Benkhouja, K., Zahir, A., Sadel, A., Drillon, M. & Bernier, J. C. (1991). *C. R. Acad. Sci. Sér. C*, **313**, 881–884.

Benkhouja, K., Zahir, M., Sadel, A., Handizi, A., Boukhari, A., Holt, E. M., Aride, J. & Drillon, M. (1995). *Mater. Res. Bull.* **30**, 49–55.

Bettach, M., Benkhouja, K., Sadel, A., Zahir, M. & Drillon, M. (1994). *Adv. Mater. Res.* **1–2**, 543–552.

Cruickshank, D. W. J., Lynton, H. & Barclay, G. A. (1962). *Acta Cryst.* **15**, 491–498.

Donnay, G. & Allmann, R. (1970). *Am. Mineral.* **55**, 1003–1015.

El Bali, B. (1990). Thesis, University of Rabat, Morocco.

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

Erragh, F., Boukhari, A., Elouadi, B. & Holt, E. M. (1991). *J. Crystallogr. Spectrosc. Res.* pp. 321–326.

Frenz, B. A. (1985). *Enraf–Nonius SDP-Plus Structure Determination Package*. Version 3.0. Enraf–Nonius, Delft, The Netherlands.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Krasnikov, V. U., Constant, Z. A. & Bel'skii, V. A. (1985). *Izv. Akad. Nauk SSSR Neorg. Mater.* **21**, 1360–1363.
Lukaszewicz, K. (1967). *Bull. Acad. Pol. Sci. Ser. Sci. Chim.* **15**, 47–51.
Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1982). *MULTAN*11/82. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Universities of York, England, and Louvain, Belgium.
Moqine, A., Boukhari, A. & Holt, E. M. (1991). *Acta Cryst.* C**47**, 2294–2297.
Murashova, E. V., Velikodnyi, Yu. A. & Trunov, V. K. (1991). *Zh. Neorg. Khim.* **36**, 847–850.
Riou, D. & Goreaud, M. (1990). *Acta Cryst.* C**46**, 1191–1193.
Riou, D., Labbe, P. & Goreaud, M. (1988*a*). *C. R. Acad. Sci. Sér. C*, **307**, 1751–1756.
Riou, D., Labbe, P. & Goreaud, M. (1988*b*). *C. R. Acad. Sci. Sér. C*, **307**, 903–907.
Riou, D., Leligny, H., Pham, C., Labbe, P. & Raveau, B. (1991). *Acta Cryst.* B**47**, 608–617.
Riou, D. & Raveau, B. (1991). *Acta Cryst.* C**47**, 1708–1709.
Shannon, R. D. (1976). *Acta Cryst.* A**32**, 751–757.
Spirlet, M. R., Rebizant, J. & Liegeois-Duyckaerts, M. (1993). *Acta Cryst.* C**49**, 209–211.
Zachariasen, W. H. (1963). *Acta Cryst.* **16**, 1139–1144.

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*Acta Cryst.* (1996). C**52**, 2963–2964

# Rubidium Indium Antimonide, $Rb_2In_2Sb_3$

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## Abstract

The structure of dirubidium diindium triantimonide, a layered material isotypic with $A_2In_2Sb_3$ ($A$ = Na, K, Cs), is reported.

## Comment

The title compound, $Rb_2In_2Sb_3$, has been structurally characterized and shown to contain discrete layers of $InSb_4$ tetrahedra separated by rubidium. These tetrahedra are connected *via* common corners, edges and short Sb—Sb distances. All distances and angles are as expected [In—Sb distances are in the range 2.8075 (10)–2.9646 (10) Å] and correspond well with the previously reported isotypic structures [*cf.* In—Sb distances in the range 2.804 (4)–2.962 (4) Å for $Cs_2In_2Sb_3$] (Cordier & Ochmann, 1991; Blase, Cordier, Poth & Weil, 1995).

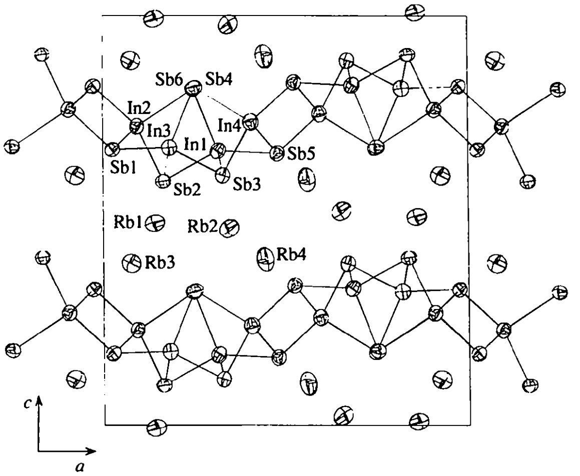

Fig. 1. *ORTEP* (Johnson, 1965) representation of the layered structure of $Rb_2In_2Sb_3$ showing the $In_2Sb_3$ layers separated by rubidium (90% probability level).

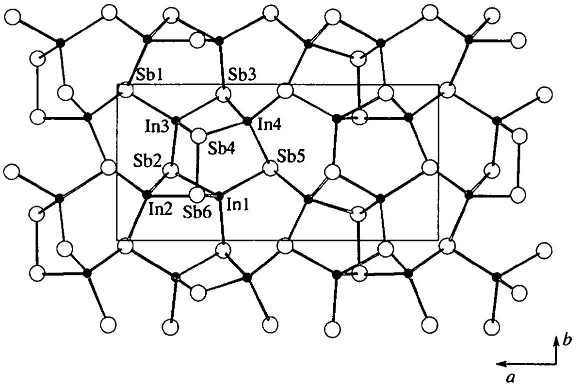

Fig. 2. Cross-section of the $In_2Sb_3$ layer showing corner- and edge-sharing connections of $InSb_4$ tetrahedra and the short Sb—Sb distances.

## Experimental

$Rb_2In_2Sb_3$ was prepared by direct combination of the elements. A dry quartz tube was charged with Rb (0.26 g, 3 mmol), In (0.23 g, 2 mmol) and Sb (0.36 g, 3 mmol). The tube was evacuated for 1.5 h at $5 \times 10^{-3}$ Torr (1 Torr = 133.322 Pa) and sealed under vacuum. The sample was heated at 873 K in an automatic control furnace for 10 h and then cooled to ambient temperature over a 96 h period.

*Crystal data*

$Rb_2In_2Sb_3$
$M_r = 765.83$

Ag $K\alpha$ radiation
$\lambda = 0.56086$ Å

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