

Tableau 2. Distances (Å) et angles interatomiques (°) avec les écarts types entre parenthèses

Groupements $\text{PO}_3\text{H}^{2-}$			
P(1)—O(11)	1,501 (9)	P(2)—O(21)	1,542 (7)
P(1)—O(12)	1,560 (9)	P(2)—O(22)	1,502 (7)
P(1)—O(13)	1,495 (7)	P(2)—O(23)	1,516 (8)
P(1)—H(1)	1,41	P(2)—H(2)	1,41
O(11)—P(1)—O(12)	111,7 (5)	O(21)—P(2)—O(22)	106,1 (4)
O(11)—P(1)—O(13)	116,1 (5)	O(21)—P(2)—O(23)	112,0 (4)
O(12)—P(1)—O(13)	105,8 (4)	O(22)—P(2)—O(23)	114,3 (5)
O(11)—P(1)—H(1)	107,5	O(21)—P(2)—H(2)	108,0
O(12)—P(1)—H(1)	107,7	O(22)—P(2)—H(2)	108,0
O(13)—P(1)—H(1)	107,6	O(23)—P(2)—H(2)	108,0
Groupement $\text{LaO}_8$			
La—O(11)	2,471 (8)	La—O(13)	2,475 (7)
La—O(13)	2,568 (7)	La—O(21)	2,485 (7)
La—O(22)	2,677 (7)	La—O(22)	2,492 (7)
La—O(23)	2,429 (7)	La—W(3)	2,561 (8)
Molécules d'eau			
W(1)···W(3)	2,82 (1)	W(2)···O(12)	2,59 (1)
W(1)···W(3)	2,85 (1)	W(2)···O(13)	2,75 (1)
W(1)···W(2)	2,85 (1)	W(2)···O(23)	2,90 (1)
W(3)···O(11)	2,82 (1)		

Les molécules d'eau assurent avec le cation  $\text{La}^{3+}$ , la cohésion de l'édifice structural. Les atomes d'hydrogène de ces molécules n'ont pas été positionnés et les plus courtes distances  $\text{W} \cdots \text{O}$  ne permet-

tent pas de prédire la direction de ces liaisons (Tableau 2c). On observe pour  $W(1)$  trois distances longues (2,82 et 2,85 Å) révélatrices d'un réseau complexe de liaisons hydrogène faibles. Pour  $W(2)$  mise à part la distance courte  $W(2) \cdots \text{O}(12)$ , deux atomes d'oxygène se trouvent à des distances de 2,75 et 2,90 Å. Alors que  $W(3)$  se trouve à une distance de 2,82 Å de O(11). Ici aussi ces liaisons hydrogènes sont faibles.

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## Structure Refinement of Monoclinic 12-Layer $\text{TaNi}_3$ with $\beta\text{-NbPt}_3$ Type. New Crystallographic Descriptions of this Type and of the $\text{Nb}_3\text{Rh}_5$ Type Based on Smaller Unit Cells

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**Abstract.** The atom arrangements in the  $\beta\text{-NbPt}_3$  and  $\text{Nb}_3\text{Rh}_5$  [ $=(\text{Nb}_{0.75}\text{Rh}_{0.25})\text{Rh}$ ] structures can be described with unit cells having only one third of the original volumes.  $\beta\text{-NbPt}_3$ :  $mP16$ ,  $P2_1/m$ ,  $a = 4.870$ ,  $b = 5.537$ ,  $c = 9.268$  Å,  $\beta = 100.62^\circ$ .  $\text{Nb}_3\text{Rh}_5$ :  $mP6$ ,  $P2/m$ ,  $a = 4.772$ ,  $b = 2.806$ ,  $c = 6.949$  Å,  $\beta = 103.76^\circ$ . According to the new description ( $\text{Nb}_{0.75}\text{Rh}_{0.25})\text{Rh}$  is isotypic to  $\text{LiSn}$ . Structure refinement of 12-layer  $\text{TaNi}_3$  with  $\beta\text{-NbPt}_3$  type,  $M_r = 357.078$ ,  $mP16$ ,  $P2_1/m - f^2e^4$ ,  $a = 4.5319$  (6),  $b = 5.1253$  (8),  $c = 8.632$  (1) Å,  $\beta = 100.79$  (1)°,  $V = 196.96$  (4) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 12.038$  Mg m<sup>-3</sup>,  $\lambda(\text{Mo K}\alpha) = 0.71073$  Å,  $\mu$

$= 82.661$  mm<sup>-1</sup>,  $F(000) = 628$ ,  $T = 300$  K,  $R = 0.068$ ,  $wR = 0.062$  for 309 contributing unique reflections. The new description of the structure type is confirmed.

**Introduction.** The  $\beta\text{-NbPt}_3$  and  $\text{Nb}_3\text{Rh}_5$  structures are substitution variants of close-packed structures with stackings  $hhcc$  and  $hhc$ , respectively (Giessen & Grant, 1964). The results of the application of *MISSYM* (Le Page, 1988) to the  $\beta\text{-NbPt}_3$  and  $\text{Nb}_3\text{Rh}_5$  data suggest a non-space-group translation in both structures. This means that the unit cells in

the original paper do not correspond to the smallest possible ones and consequently the crystallographic descriptions of these structures should be changed.

#### The $\beta$ -NbPt<sub>3</sub> type and isotypes

An intensity calculation of  $\beta$ -NbPt<sub>3</sub> based on the published structure data, transformed to standard space group setting  $P12_1/m1$ , indicates that all reflections  $hkl$ , where  $l$  is not  $3n + h$ , have zero intensity. This is an indication that the chosen unit cell is too large.

The  $\beta$ -NbPt<sub>3</sub> structure can be described with a unit cell having only one third of the original volume without changing the space group  $P2_1/m$ . Performing a unit-cell transformation based on

$$\mathbf{a}' = \mathbf{b}, \mathbf{b}' = -\mathbf{a} \text{ and } \mathbf{c}' = -\frac{1}{3}\mathbf{b} + \frac{1}{3}\mathbf{c} \quad (1)$$

and standardizing the results with the *STRUCTURE-TIDY* program (Gelato & Parthé, 1987), the original structure data can be transformed into the data shown in Table 1. Note that (1) also includes a change from  $P2_1/m11$  to standard setting  $P12_1/m1$ . The transformation can be made without any approximations since there is a perfect equivalence of the different atom positions which must coincide with the description using the small unit cell. Since the original atom coordinates had been given as fractions, the transformed data are presented to two decimal places. It seems probable that Giessen & Grant (1964), for a comparison with other close-packed structures, selected a large unit cell where the monoclinic angle is close to 90°; however, this is not the correct crystallographic description.

The  $\beta$ -NbPt<sub>3</sub> type has also been reported for  $\beta$ -TaPt<sub>3</sub> (Giessen & Grant, 1964) and 12-layer TaNi<sub>3</sub>, stable between 1128 and 1473 K (Ruhl, Giessen, Cohen & Grant, 1967). According to a later paper (Giessen & Grant, 1967) the 12-layer TaNi<sub>3</sub>, also denoted as TaNi<sub>3</sub>(12)S, is the equilibrium structure of TaNi<sub>3</sub> up to the melting point. For the reported isotypic phases the following new smaller unit-cell parameters can be calculated:

#### $\beta$ -TaPt<sub>3</sub>:

$$a = 4.869, b = 5.537, c = 9.269 \text{ \AA}, \beta = 100.62^\circ, Z = 4.$$

#### 12-layer TaNi<sub>3</sub>:

$$a = 4.523, b = 5.126, c = 8.612 \text{ \AA}, \beta = 100.91^\circ, Z = 4.$$

In the original papers on  $\beta$ -NbPt<sub>3</sub> and isotypes no error limits were given for the lattice constants and the adjustable positional atom coordinates were approximated by simple fractions, thus we considered it worthwhile re-examining this structure type using single-crystal diffraction methods. The isotypic 12-layer TaNi<sub>3</sub> structure was chosen to confirm the new description of this structure type.

Table 1. *New standardized description of the  $\beta$ -NbPt<sub>3</sub> type with smaller unit cell*

Data transformed from the original ones published by Giessen & Grant (1964).

$$mP16, P12_1/m1, a = 4.870, b = 5.537, c = 9.268 \text{ \AA}, \beta = 100.62^\circ.$$

			$x$	$y$	$z$
Pt(1)	in	4(f)	$\sim 0.13$	$\sim 0.0$	$\sim 0.38$
Pt(2)	in	4(f)	$\sim 0.71$	$\sim 0.0$	$\sim 0.13$
Pt(3)	in	2(e)	$\sim 0.21$	$\frac{1}{4}$	$\sim 0.13$
Pt(4)	in	2(e)	$\sim 0.38$	$\frac{1}{4}$	$\sim 0.63$
Nb(1)	in	2(e)	$\sim 0.63$	$\frac{1}{4}$	$\sim 0.38$
Nb(2)	in	2(e)	$\sim 0.79$	$\frac{1}{4}$	$\sim 0.88$

#### Experimental.

*Refinement of 12-layer TaNi<sub>3</sub>.* A sample of nominal composition TaNi<sub>3</sub> was prepared by arc melting under argon atmosphere (Ta 99.9 and Ni 99.99%). The weight loss was 0.2%. The sample was annealed at 1373 K for 14 d in a silica tube under  $4.00 \times 10$  Pa argon atmosphere. A single crystal with an irregular shape (approximate mean radius: 0.024 mm), obtained from the annealed sample, was mounted on a Philips PW 1100 automatic four-circle diffractometer, Mo  $K\alpha$  radiation with graphite monochromator. The small unit cell ( $a = 4.532, b = 5.125, c = 8.632 \text{ \AA}, \beta \approx 101^\circ$ ) was first found and then transformed into the large unit cell ( $a = 4.532, b = 5.125, c = 25.44 \text{ \AA}, \beta \approx 90^\circ$ ). A small set of data was collected based on the large cell to test the correctness of the unit-cell transformation. It was found that, in addition to the reflections  $hkl$  with  $l = 3n + h$  which were expected, some weak reflection  $hkl$  with  $l = 3n - h$  occur, all other reflections  $hkl$  having zero intensity. The positions of the reflections with  $l = 3n - h$  were measured and indexed also by the small unit cell, but with a different orientation matrix. This led us to assume that the crystal contains two domains of different volumes which are twins. The twin plane was found to be (001) which is parallel to the  $AB_3$  layers in the structure. If there is twinning in the structure this would be the plane which one would expect to be the twin plane. The mutual orientations of the twin domains are such that reflections  $hkl$  with  $h$  not being  $3n$  (small and large cell) come from only one or the other twin domain. When  $h = 3n$  the observed intensity has contributions from both domains.

The unit cell was transformed again into the small one and the orientation fixed on the large domain to make a new data collection. The unit-cell parameters given in the *Abstract* were refined from  $2\theta$  values of 35 reflections (Mo  $K\alpha, \lambda = 0.71073 \text{ \AA}, 18 < 2\theta < 40^\circ$ ) using the program *LATCON* (Schwarzenbach, 1966). They are close to the calculated values given above. 1352 reflections were collected out to  $(\sin\theta)/\lambda = 0.702 \text{ \AA}^{-1}$  ( $0 \leq h \leq 6, 0 \leq k \leq 7, -12 \leq l \leq 12$  and corresponding anti-reflections) in the  $\omega$ - $2\theta$  scan

mode, yielding 636 unique reflections ( $R_{\text{int}} = 0.10$ ). Two standard reflections ( $1\bar{2}\bar{2}$  and  $004$ ) were measured with maximum intensity variations 2.2 and 1.8% respectively. The spherical absorption correction was made by using the program *ABSORB* with max. and min. transmission factors of 0.0917 and 0.0646. The anomalous-dispersion factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). The extinction of  $0k0$  with  $k = 2n + 1$  led to two possible space groups:  $P2_1$  and  $P2_1/m$ . The space group  $P2_1/m$  in standard setting (*International Tables for Crystallography*, 1983, Vol. A) and the atomic positional parameters of the transformed data for the  $\beta$ -NbPt<sub>3</sub> structure in Table 1 were used as starting data for the structure refinement. The refinement was based on  $|F|$  values using anisotropic atomic displacement parameters and the program *CRYLSQ* (Olthof-Hazekamp, 1989). 43 variables refined to  $R = 0.070$  and  $wR = 0.066$  [ $w = 1/\sigma^2(|F_{\text{rel}}|)$ ,  $S = 2.071$ ] considering 375 contributing unique reflections with  $|F| \geq 3\sigma(F)$ . The max. shift/e.s.d. in the last cycle is 0.00007.\* The final residual electron density is  $-9.7\text{--}12.1 \text{ e } \text{\AA}^{-3}$ . The programs used to refine the structure are from the *XTAL2.6* system (Hall & Stewart, 1989).

A second refinement was carried out excluding the coincident reflections  $hkl$  with  $h = 3n$ . 43 variables refined to  $R = 0.068$  and  $wR = 0.062$  [ $w = 1/\sigma^2(|F_{\text{rel}}|)$ ,  $S = 1.723$ ] considering 309 contributing unique reflections with  $|F| \geq 3\sigma(F)$ . The max. shift/e.s.d. in the last cycle is 0.00006.\* The final residual electron density is  $-7.8\text{--}6.3 \text{ e } \text{\AA}^{-3}$ . The positions of the largest peak and hole are around the heavy Ta atoms (distances less than 0.8 Å). The atomic positional and displacement parameters obtained from the second refinement are given in Table 2 and interatomic distances up to 3.5 Å in Table 3. A comparison of the two refinements shows that the atomic positional parameters and the interatomic distances do not differ significantly but the  $R$  values and the residual electron densities are lower in the refinement excluding the reflections  $hkl$  with  $h = 3n$ .

The relatively high  $R$  values and residual electron density are due to the quality of the crystal. Since the sample is not brittle it is very difficult to break it without producing stacking faults near the crystal boundary. It is very difficult to obtain a mono-domain crystal and the twinning of the crystal may be produced simply by moderate force. Also Giessen & Grant (1967) had noted that cold work will pro-

Table 2. Atomic positional and displacement parameters for the 12-layer TaNi<sub>3</sub> structure (space group  $P12_1/m1$ ) (refinement excluding the reflections  $hkl$  with  $h = 3n$ )

The equivalent atomic displacement factors are expressed as  $U_{\text{eq}} = (1/3)\sum_i U_{ij}a_i^*a_j^*a_j$ . E.s.d.'s are given in parentheses.

	Wyckoff position	x	y	z	$U_{\text{eq}}(\text{\AA}^2 \times 10^2)$
Ni(1)	in 4(f)	0.1130 (13)	0.0025 (9)	0.3736 (5)	0.6 (2)
Ni(2)	in 4(f)	0.7044 (13)	0.0017 (7)	0.1235 (6)	0.8 (2)
Ni(3)	in 2(e)	0.2011 (20)	$\frac{1}{4}$	0.1262 (9)	0.6 (3)
Ni(4)	in 2(e)	0.3675 (18)	$\frac{1}{4}$	0.6234 (7)	0.6 (3)
Ta(1)	in 2(e)	0.6115 (6)	$\frac{1}{4}$	0.3781 (2)	0.4 (1)
Ta(2)	in 2(e)	0.7907 (6)	$\frac{1}{4}$	0.8722 (3)	0.41 (9)

Table 3. Interatomic distances up to 3.5 Å in 12-layer TaNi<sub>3</sub> (refinement excluding the reflections  $hkl$  with  $h = 3n$ )

Ta(1)—Ni(4)	2.5645 (5)	Ni(2)—Ni(2)	2.546 (5)
Ni(4)	2.566 (8)	Ni(2)	2.550 (7)
Ni(3)	2.583 (8)	Ni(1)	2.568 (7)
2Ni(1)	2.585 (6)	Ni(2)	2.580 (5)
2Ni(1)	2.609 (6)	Ni(3)	2.583 (10)
2Ni(1)	2.613 (5)	Ta(2)	2.594 (6)
2Ni(2)	2.640 (5)	Ta(2)	2.607 (5)
Ta(2)—2Ni(3)	2.5629 (4)	Ni(4)	2.610 (8)
2Ni(1)	2.591 (5)	Ni(3)	2.615 (9)
2Ni(2)	2.594 (6)	Ni(3)	2.616 (10)
Ni(3)	2.596 (8)	Ta(2)	2.621 (6)
Ni(4)	2.598 (7)	Ta(1)	2.640 (5)
2Ni(2)	2.607 (5)	Ni(3)—2Ta(2)	2.5629 (4)
2Ni(2)	2.621 (6)	2Ni(1)	2.579 (8)
Ni(1)—Ni(1)	2.537 (7)	2Ni(2)	2.583 (10)
Ni(4)	2.538 (9)	Ta(1)	2.583 (8)
Ni(2)	2.568 (7)	Ta(2)	2.596 (8)
Ni(3)	2.579 (8)	2Ni(2)	2.615 (9)
Ni(1)	2.579 (7)	2Ni(2)	2.616 (10)
Ni(4)	2.580 (7)	Ni(4)—2Ni(1)	2.538 (9)
Ta(1)	2.585 (6)	2Ta(1)	2.5645 (5)
Ni(1)	2.588 (7)	Ta(1)	2.566 (8)
Ta(2)	2.591 (5)	2Ni(1)	2.580 (7)
Ta(1)	2.609 (6)	Ta(2)	2.598 (7)
Ta(1)	2.613 (5)	2Ni(2)	2.610 (8)
Ni(4)	2.682 (9)	2Ni(1)	2.682 (9)

voke a change of the 12-layer TaNi<sub>3</sub> structure to the 3-layer TaNi<sub>3</sub> modification with TiAl<sub>3</sub> type. Therefore, whenever the crystal is broken by force, stacking faults occur in the region near the surface which can lead to particular diffraction effects. The stacking disorder does not, however, influence the atom ordering inside the layers.

## Discussion.

*The  $\beta$ -NbPt<sub>3</sub> type.* The basic construction element of the  $\beta$ -NbPt<sub>3</sub> type is a close-packed atom layer of composition  $AB_3$ . There are only two ways to arrange, in an ordered fashion,  $A$  and  $B$  atoms in the ratio 1:3 on a plane if two conditions are to be met: i.e. (i) there must be no contacts between the minority atoms and (ii) coordination circles of all minority atoms must be identical and non-rotated.

\* Lists of observed and calculated structure factors, atomic parameters and interatomic distances for both refinements (excluding and including reflections  $hkl$  with  $h = 3n$ ) have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53538 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

As a result one finds a hexagonal  $AB_3$  and a rectangular  $AB_3$  layer with plane group  $p6mm$  and  $p2mm$ , respectively, shown on the upper part of Fig. 1. These layers will be stacked in a close packed manner with the restriction that there should be no contacts between minority atoms of different planes. The stacking is conveniently characterized by the Jagodzinski-Wyckoff stacking symbols. Ten  $AB_3$  structure types are known which can be interpreted as different stackings of the hexagonal  $AB_3$  layer:  $\text{Ni}_3\text{Sn}$  LT  $[(h)_2]$ ,  $\text{Ba}(\text{Pb}_{0.8}\text{Ti}_{0.2})_3$   $[(hhchhc)_2]$ ,  $\text{BaPb}_3$   $[(hhc)_3]$ ,  $\gamma\text{-Ta}(\text{Pd}_{0.67}\text{Rh}_{0.33})_3$   $[(hhchc)_2]$ ,  $\text{TiNi}_3$   $[(hc)_2]$ ,  $\text{PuGa}_3$  HT  $[(hcc)_3]$ ,  $\text{HoAl}_3$   $[(hcchc)_3]$ ,  $\text{PuAl}_3$  HT  $[(hcc)_2]$ ,  $\text{Ti}(\text{Pt}_{0.89}\text{Ni}_{0.11})_3$   $[hccccchc]$ ,  $\text{Cu}_3\text{Au}$   $[(c)_3]$ . Four other  $AB_3$  structure types represent stackings of the rectangular  $AB_3$  layer:  $\beta\text{-TiCu}_3$  LT  $[(h)_2]$ ,  $\beta\text{-NbPt}_3$   $[(hhcc)_3]$ ,  $\beta\text{-NbPd}_3$   $[(hcc)_2]$ ,  $\text{TiAl}_3$   $[(c)_3]$ . Thus the  $\beta\text{-NbPt}_3$  structure, as mentioned under the last group, is built up of 12 rectangular  $AB_3$  layers in  $(hhcc)_3$  stacking.

From Table 3 one can see that in 12-layer  $\text{TaNi}_3$  the distances around the central atom to the 12 nearest neighbouring atoms are similar. There are no significant differences between Ta–Ni and Ni–Ni distances, although the sums of the atomic radii are different (Ta–Ni: 2.713 and Ni–Ni: 2.492 Å). This contraction of the Ta–Ni distances is observed also

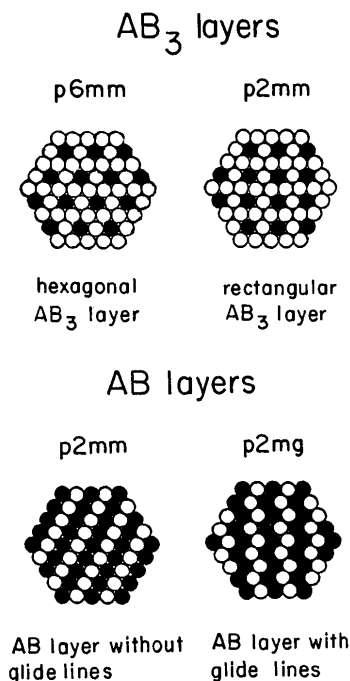


Fig. 1. The two kinds of close packed  $AB_3$  and  $AB$  layers where the number of the homonuclear contacts is a minimum. In each  $AB_3$  layer coordination circles of all the minority atoms are identical and non-rotated. In each  $AB$  layer the planar coordinations of all atoms are equal.

Table 4. New standardized description of the  $\text{Nb}_3\text{Rh}_5$   $[(\text{Nb}_{0.75}\text{Rh}_{0.25})\text{Rh}]$  type with smaller unit cell

Data transformed from the original ones published by Giessen & Grant (1964). The mixed sites are denoted by  $X$ .

$mP6$ ,  $P12/m1$ ,  $a = 4.772$ ,  $b = 2.806$ ,  $c = 6.949$  Å,  $\beta = 103.76^\circ$ .

			$x$	$y$	$z$
$X(1)$	in	$2(n)$	$\sim 0.28$	$\frac{1}{2}$	$\sim 0.33$
$\text{Rh}(1)$	in	$2(m)$	$\sim 0.22$	0	$\sim 0.67$
$X(2)$	in	$1(e)$	$\frac{1}{2}$	$\frac{1}{2}$	0
$\text{Rh}(2)$	in	$1(a)$	0	0	0

The values reported in *Structure Reports* Vol. 29, p. 71 for the  $2(m)$  sites of  $\text{Nb}_3\text{Rh}_5$  are those belonging to the  $2(n)$  sites and vice versa.

for the other two close-packed  $\text{TaNi}_3$  structures, i.e. the 2-layer  $\text{TaNi}_3$  structure ( $\beta\text{-TiCu}_3$  LT type) (Pylaeva, Gladyshevskii & Kripyakevich, 1958) and the 3-layer  $\text{TaNi}_3$  structure ( $\text{TiAl}_3$  type) (Nowotny & Oesterreicher, 1964). In the 2-layer  $\text{TaNi}_3$  structure the Ta–Ni distances are 2.549, 2.557, 2.606, 2.626 Å and in the 3-layer structure 2.565, 2.601 Å respectively. In both compounds the Ni–Ni distances have the same values. The space fillings\* of 12-layer  $\text{TaNi}_3$ , 2-layer  $\text{TaNi}_3$  and 3-layer  $\text{TaNi}_3$  are 76.2, 76.0 and 76.5%, respectively, if we assume the hard sphere model ( $r_{\text{Ta}} = 1.467$  Å and  $r_{\text{Ni}} = 1.246$  Å). These values, which are slightly larger than that of the ideal close packed structure (0.74), may be expected because of the strong interactions between Ta and Ni atoms.

The  $\text{Nb}_3\text{Rh}_5$   $[(\text{Nb}_{0.75}\text{Rh}_{0.25})\text{Rh}]$  type. The  $\text{Nb}_3\text{Rh}_5$  structure can also be described with a unit cell having only one third of the original volume without changing the space group, in this case  $P2/m$ . Using the same unit-cell transformation as in (1) and structure standardization, the original structure data are transformed into the new data, shown in Table 4.

Comparing this new crystallographic description of the  $\text{Nb}_3\text{Rh}_5$  structure with known data of other structure types, we find that this compound is isotypic to  $\text{LiSn}$  (Müller & Schäfer, 1973) for which the standardized data are reported in Table 5. The fact that  $\text{LiSn}$  has been determined by single-crystal diffraction methods makes us feel confident that the new description of the Nb–Rh alloy structure is correct, even without experimentally re-examining it. We shall henceforth refer to this atom arrangement as the  $\text{LiSn}$  type.

The basic construction element of the  $\text{LiSn}$  type is also a close-packed atom layer but here of composition  $AB$ . If one admits that (i) the number of homonuclear contacts must be as small as possible and (ii) all atoms must have the same planar coordination, there are only two ways to arrange, in an orderly fashion, the  $A$  and  $B$  atoms on a plane. Both layers,

\* Space filling of  $\text{TaNi}_3 = [(4/3\pi(r_{\text{Ta}}^3 + 3r_{\text{Ni}}^3)]Z/V_{\text{measured}}$ .

Table 5. *The standardized structure data for LiSn based on the values given by Müller & Schäfer (1973)*

$mP6$ ,  $P12/m1$ ,  $a = 5.17 \pm 0.02$ ,  $b = 3.18 \pm 0.02$ ,  $c = 7.74 \pm 0.02$  Å,  
 $\beta = 104.5 \pm 0.3^\circ$ .

			$x$	$y$	$z$
Li(1)	in	2(n)	0.263	$\frac{1}{2}$	0.336
Sn(1)	in	2(m)	0.234	0	0.660
Li(2)	in	1(e)	$\frac{1}{2}$	$\frac{1}{2}$	0
Sn(2)	in	1(a)	0	0	0

Note that LiSn had to be transformed from setting  $P112/m$  to standard setting  $P12/m1$ . This was obviously also intended in *Structure Reports* Vol. 39A, p. 82, but while the atom coordinates were transformed to the setting with  $b$  axis unique, the cell parameters given correspond to the original setting with  $c$  axis unique.

shown in the lower part of Fig. 1, are rectangular and have plane groups  $p2mm$  and  $p2mg$ , respectively. Four  $AB$  structure types can be interpreted as different stackings of the rectangular  $p2mm$  layer: MgCd  $[(h)_2]$ , LiSn  $[(hhc)_3]$ ,  $\alpha$ -TaRh  $[(hcc)_2]$  and TiAl  $[(c)_3]$ . The UPb  $[(c)_3]$  type is the only  $AB$  type known where  $p2mg$  layers are stacked. The LiSn type is built up of 9 rectangular  $AB$  layers without a glide line which are stacked in the sequence  $(hhc)_3$ .

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## Structure Determination of Ba(OD)Br.2D<sub>2</sub>O by Neutron Powder Diffraction

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**Abstract.** Barium hydroxide bromide dihydrate ( $d$ -5), Ba(OD)Br.2D<sub>2</sub>O,  $M_r = 275.32$ , tetragonal,  $P4/nmm$ ,  $Z = 2$ , neutron radiation,  $\lambda = 1.09$  Å,  $\mu_{\text{calc}} = 0.065 \text{ cm}^{-1}$ ,  $F(000) = 126.62 \text{ fm}$ ,  $[(\sin\theta)/\lambda]_{\text{max}} = 0.632 \text{ Å}^{-1}$ ,  $T_1 = 300 \text{ K}$ ,  $a = 4.5825(4)$ ,  $c = 11.791(2) \text{ Å}$ ,  $V = 247.60(3) \text{ Å}^3$ ,  $D_x = 3.693 \text{ Mg m}^{-3}$ ,  $R_{\text{profile}} = 0.0208$ ;  $T_2 = 16 \text{ K}$ ,  $a = 4.5567(2)$ ,  $c = 11.718(1) \text{ Å}$ ,  $V = 243.30(1) \text{ Å}^3$ ,  $D_x = 3.758 \text{ Mg m}^{-3}$ ,  $R_{\text{profile}} = 0.0209$ . The structure is related to the PbFCl type with ninefold coordination of the Ba atoms. The OD<sup>−</sup> ions are not coordinated to the Ba

atoms, but are acceptors of four very strong hydrogen bonds. The OD distances are 0.992(5) [0.994(4)] Å for D<sub>2</sub>O, 0.956(13) [0.951(9)] Å for OD<sup>−</sup> and 1.639(5) [1.622(4)] Å for DOD...OD<sup>−</sup>.

**Introduction.** Recently we reported the crystal structure of Ba(OH)Cl.2H<sub>2</sub>O, which is the first representative of a new structure type (Lutz, Kellersohn & Beckenkamp, 1989). From vibrational spectra and X-ray powder photographs it was inferred that Ba(OH)Br.2H<sub>2</sub>O is isotypic. Because single crystals of sufficient quality for an X-ray structure determination could not be grown, we performed a neu-

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