# A New Ternary Arsenide LaNi<sub>5</sub>As<sub>3</sub>: Preparation and Crystal Structure

Volodymyr Babizhetskyy<sup>a,b</sup>, Roland Guérin<sup>a</sup>, and Arndt Simon<sup>b</sup>

<sup>a</sup> Laboratoire de Chimie du Solide et Inorganique Moléculaire, UMR CNRS 6511, Institut de Chimie,

Campus de Beaulieu, Avenue du Général Leclerc, F-35042 Rennes Cedex, France

<sup>b</sup> Max-Planck-Institut für Festkörperforschung, Heisenbergstraße 1, Postfach 800665,

D-70569 Stuttgart, Germany

Reprint requests to Prof. Dr. R. Guérin. Fax: +33-2 23 23 67 99. E-mail: roland.guerin@univ-rennes1.fr

Z. Naturforsch. 59b, 1103-1108 (2004); received June 2, 2004

The title compound was prepared from the elements by arc-melting followed by annealing in silica tubes at 1070 K for 3 months. The orthorhombic structure of LaNi<sub>5</sub>As<sub>3</sub> was solved and refined from X-ray single crystal data: space group *Pnma*, a = 11.179(2), b = 3.9133(5), c = 24.443(3) Å, Z = 8, R = 0.040,  $R_w = 0.039$  for 1045 unique reflections with  $I > 2\sigma(I)$  and 110 parameters. It is a new structure type, which can be described by condensed units of trigonal prisms around the arsenic atoms linked together to infinite zigzag chains. The structural features of LaNi<sub>5</sub>As<sub>3</sub> are discussed in comparison with some representatives of a large family of structures with a metal/non-metal ratio equal or close to 2:1.

Key words: Ternary Arsenides, Crystal Structure, Rare-Earth Compound

# Introduction

Recently, we reported on the solid-state phase equilibria in the ternary RE-Ni-As phase diagrams for RE = La and Ce [1]. During this study, several ternary phases were found among which are the compounds RENi<sub>5</sub>As<sub>3</sub>. The compositions were deduced from microprobe analyses on bulk samples. For example, LaNi<sub>5</sub>As<sub>3</sub> was observed through the backscattered electron image of a sample with a nominal atomic composition La/Ni/As = 10/65/25. The compound LaNi<sub>5</sub>As<sub>3</sub> is formed by peritectic reaction between La<sub>6</sub>Ni<sub>20</sub>As<sub>13</sub> and the Ni<sub>1-x</sub>As<sub>x</sub>-rich liquid. Unfortunately, owing to the absence of single crystals and the complexity of the X-ray diffraction patterns, no structure determination could be performed at that time. However, recent attempts to improve the crystal growth by arc melting were successful and small single crystals of LaNi5As3 could be isolated in the form of shiny black platelets.

The present paper describes the synthesis and the crystal structure of this ternary arsenide. In addition, the main crystal chemical features of this new structural type are compared with some related ones, like  $LaCo_5P_3$ ,  $YCo_5P_3$ ,  $CeNi_{5-x}P_3$  and  $YNi_5Si_3$ , which belong to the same family of ternary compounds with a metal/non-metal ratio equal or close to 2:1.

Table 1. Crystal data, intensity collection and structure refinement for LaNi<sub>5</sub>As<sub>3</sub>.

Empirical formula	LaNi <sub>5</sub> As <sub>3</sub>
Crystal system	orthorhombic
Space group	Pnma
a [Å]	11.179(2)
<i>b</i> [Å]	3.9133(5)
c [Å]	24.443(3)
V [Å <sup>3</sup> ]	1069(1)
Z, calculated density [g/cm <sup>3</sup> ]	1, 8.16
Crystal size [mm <sup>3</sup> ]	$0.127\times0.020\times0.014$
Linear absorption coeff. [mm <sup>-1</sup> ]	43.32
Absorption coefficient $[T_{\min}, T_{\max}]$	0.137, 0.546
θ Limits [°]	$2 < \theta < 32$
Data collected	-16 < h < 16
	-5 < k < 5
	-36 < l < 35
Measured reflections	11914
Unique reflections, R <sub>int</sub>	$1139[I > 2\sigma(I)]; 0.079$
Reflections in refinement	$1045[I > 2\sigma(I)]$
Refinement based on F	F
Unweighed R factor	0.040
Weighted $R_w$ factor	0.039
$[w^{-1} = \sigma^2 (F_0)^2 + 0.00053 F_0^2]$	
Extinction correction (Gaussian isotropic)	0.032(3)
Scale factor	0.0799(3)
Goodness of fit	0.99
Max.min [e/Å <sup>3</sup> ]	2.07, -2.46

0932–0776 / 04 / 1000–1103 \$ 06.00 © 2004 Verlag der Zeitschrift für Naturforschung, Tübingen · http://znaturforsch.com

Atom	Site	x/a	y/b	z/c	$U_{11}$	$U_{22}$	$U_{33}$	$U_{13}$	$U_{\rm eq}$ (Å <sup>2</sup> ) *
La1	4c	0.9120(2)	3/4	0.33840(4)	0.0106(5)	0.0099(6)	0.0135(5)	-0.0004(5)	0.0113(3)
La2	4c	0.4137(2)	1/4	0.39571(4)	0.0096(4)	0.0098(5)	0.0117(5)	-0.0001(4)	0.0104(3)
As1	4c	0.9119(2)	1/4	0.43990(7)	0.0104(8)	0.011(1)	0.0115(9)	0.0004(8)	0.0111(5)
As2	4c	0.9125(2)	3/4	0.20373(8)	0.0184(9)	0.010(1)	0.0128(9)	-0.0013(9)	0.0133(5)
As3	4c	0.7012(2)	1/4	0.31752(9)	0.0101(9)	0.010(1)	0.0139(9)	0.0001(7)	0.0112(6)
As4	4c	0.1228(2)	1/4	0.31468(9)	0.0118(9)	0.011(2)	0.0115(9)	-0.0005(7)	0.0114(6)
As5	4c	0.2281(2)	3/4	0.44293(8)	0.0085(9)	0.012(1)	0.0119(9)	0.0005(7)	0.0110(6)
As6	4c	0.5985(2)	3/4	0.44709(8)	0.011(1)	0.010(1)	0.0116(8)	0.0006(8)	0.0109(5)
Ni1	4c	0.0221(2)	3/4	0.4666(1)	0.011(1)	0.012(2)	0.015(1)	0.0001(9)	0.0127(8)
Ni2	4c	0.2195(3)	3/4	0.3469(1)	0.017(2)	0.013(2)	0.013(1)	-0.004(1)	0.0141(9)
Ni3	4c	0.1173(3)	1/4	0.4106(1)	0.017(2)	0.013(2)	0.013(1)	0.0005(9)	0.0140(9)
Ni4	4c	0.6618(3)	3/4	0.2513(1)	0.020(1)	0.013(1)	0.013(1)	0.0026(9)	0.0155(7)
Ni5	4c	0.6043(3)	3/4	0.3503(1)	0.017(2)	0.014(2)	0.012(1)	0.0027(9)	0.0143(9)
Ni6	4c	0.3049(3)	1/4	0.2666(1)	0.011(1)	0.018(2)	0.017(1)	-0.002(1)	0.0153(8)
Ni7	4c	0.7128(3)	1/4	0.4131(1)	0.010(1)	0.017(2)	0.011(1)	0.0010(9)	0.0130(8)
Ni8	4c	0.5905(3)	1/4	0.5038(1)	0.013(1)	0.014(1)	0.013(1)	-0.001(1)	0.0135(7)
Ni9	4c	0.7931(3)	3/4	0.4892(1)	0.014(2)	0.025(2)	0.016(1)	-0.001(1)	0.0182(9)
Ni10	4c	0.5204(3)	1/4	0.2694(1)	0.014(2)	0.017(2)	0.019(1)	0.002(1)	0.0163(9)

Table 2. Atomic coordinates and anisotropic displacement parameters for LaNi<sub>5</sub>As<sub>3</sub>.  $(U_{12} = U_{23} = 0)$ .

\*  $U_{eq}$  is defined as one-third of the trace of the orthogonalized *U*ij tensor.



Fig. 1. View of the unit cell of the LaNi5As3 structure along [010] and characteristic coordination polyhedra.

# **Experimental Section**

A sample of the title compound was prepared from stoichiometric amounts of the constituents, arsenic and nickel as powders and lanthanum as freshly filed chips. They were mixed together and pressed into a pellet, in an argon-filled glove box. The pellet (about 2 g) was first annealed in an evacuated silica tube at 1070 K for two days and then slowly cooled to room temperature. In a second step, the sample was melted with the help of an arc furnace under a Ti/Zr-gettered argon atmosphere. A small excess of arsenic (1%) was added to compensate for evaporation losses during arc melting. The arc-melted ingot was turned over three times and remelted in order to promote homogeneous mixing. Finally, the sample was wrapped with molybdenum foil, enclosed in an evacuated silica tube, annealed at 1070 K for 3 months, and then quenched in cold water. The sample was recovered as a molten ingot with a metallic luster. Small needle-like single crystals could be extracted from the regulus after crushing. They were unreactive towards air.

Qualitative composition analyses on the bulk sample and single crystals were performed with a scanning electron microscope TESCAN 5130 MM with an Oxford Si detector by energy dispersive X-ray spectroscopy. All measurements led to the same atomic percentage of lanthanum, nickel and arsenic corresponding to La/Ni/As = 11(1)/56(1)/34(1).

#### Structure determination

A single crystal of LaNi<sub>5</sub>As<sub>3</sub> with dimensions  $0.13 \times 0.02 \times 0.015 \text{ mm}^3$  was mounted on a STOE IPDS II (Image Plate Diffraction System). Intensity data were collected at ambient temperature using Mo-K<sub> $\alpha$ </sub> radiation ( $\lambda$  =

<u>11</u>04

La1:	2As3	3.106(2)	Ni2:	1As5	2.350(4)	Ni6:	2Ni2	2.930(3)	As1:	1Ni7	2.320(3)
	2As4	3.118(4)		2As4	2.369(2)		1La2	3.381(3)		1Ni1	2.401(3)
	2As1	3.160(2)		1As2	2.487(4)		2La1	3.443(2)		2Ni1	2.402(3)
	1As2	3.292(2)		1Ni4	2.485(4)	Ni7:	1As1	2.320(3)		1Ni3	2.405(3)
	1Ni1	3.367(3)		2Ni3	2.750(3)		1As3	2.340(3)		2Ni9	2.655(2)
	1Ni2	3.443(3)		2Ni6	2.930(3)		2As6	2.480(2)		2La1	3.160(2)
	2Ni6	3.443(2)		2La2	3.157(2)		1Ni8	2.604(4)	As2:	2Ni10	2.391(2)
	1Ni5	3.452(3)		1La1	3.443(3)		2Ni5	2.767(3)		2Ni6	2.408(2)
	2Ni7	3.482(2)	Ni3:	1As4	2.346(3)		2Ni9	2.845(3)		1Ni2	2.487(4)
	2Ni3	3.495(2)		1As1	2.405(3)		1La2	3.370(3)		1Ni5	2.519(2)
	2Ni10	3.499(3)		2As5	2.447(2)		2La1	3.482(2)		2La2	3.121(2)
	IN14	3.515(4)		2N11	2.615(3)	N18:	2As6	2.399(2)		ILal	3.292(2)
	IN14	3.551(4)		IN19	2.645(4)		IAs5	2.410(4)	As3:	IN110	2.338(3)
	IN19	3.918(4)		2N12	2.750(3)		IAs6	2.430(4)		IN17	2.340(3)
	2LaI	3.913(1)		ILa2	3.333(3)		IN1/	2.604(4)		IN16	2.361(3)
La <sub>2</sub> :	2As5	3.076(2)		2La1	3.495(2)		2N18	2.820(3)		2N15	2.376(2)
	2As6	3.110(2)	N14:	2As4	2.573(2)		2N19	3.014(3)		2N14	2.577(2)
	2As2	3.121(2)		2As3	2.577(2)		2La2	3.141(2)		2La1	3.106(2)
	2N15	3.098(3)		IN12	2.485(4)		ILa2	3.299(3)	As4:	IN13	2.346(3)
	2N18	3.141(2)		IN15	2.504(4)	N19:	IAs6	2.406(4)		1N16	2.349(4)
	2N12	3.157(2)		2N110	2.554(3)		2As5	2.576(2)		INIIO	2.352(4)
	IN18	3.299(3)		2N16	2.565(3)		2As1	2.655(2)		2N12	2.369(2)
	1N110	3.310(3)		ILal	3.515(4)		INII	2.618(4)		2N14	2.573(2)
	IN13	3.333(3)		ILal	3.551(4)		IN13	2.645(4)		2La1	3.118(2)
	IN1/	3.370(3)	N15:	IAs6	2.366(3)		2N1/	2.845(3)	As5:	IN12	2.350(4)
	1N10	3.381(3)		2AS3	2.376(2)		2IN18	3.014(3)		INII	2.376(3)
	1N19 2L - 2	3.641(4)		IASZ	2.519(4)		2N11	3.044(3)		1N18 2N1:2	2.410(4)
NT: 1.	2La2	3.913(1)		11N14 2N1:7	2.504(4)		1La2	3.044(5)		2IN15	2.447(2)
IN11:	1AS5	2.370(3)		2IN1/ 2N1:10	2.707(3)	N: 10.	1La1	3.918(4)		2N19	2.570(2)
	1AS1	2.401(3)		2N110	2.936(3)	N110:	1As3	2.338(3)	A - C.	2La2	3.076(2)
	2ASI 2NUI	2.402(2)		2La2	3.098(3)		1AS4	2.352(4)	Aso:	11N15 2N1:0	2.300(3)
	2IN11 2N1:2	2.594(3)	NIC.	1La1	3.452(3)		ZASZ	2.391(2)		2IN18 1 NEO	2.399(2)
	2IN15	2.015(3)	IN10:	1As4	2.349(3)		11N10	2.411(4)		11N19 1.N.:0	2.400(4)
	IN19	2.018(4)		IAS3	2.361(3)		2N14	2.554(3)		IIN18	2.430(4)
	2N19	3.044(3)		2AS2	2.408(2)		2IN15	2.930(3)		2N1/ 2L a2	2.480(2)
	ILaI	3.367(3)		1IN110	2.411(4)		1La2	3.310(3)		2La2	5.110(2)
				21N14	2.365(3)		2LaI	5.499(3)			

1105

Table 3. Interatomic distances (Å) with their esd's for the LaNi<sub>5</sub>As<sub>3</sub> structure.

0.71069 Å). 180 images were measured in oscillation mode of the crystal around the  $\omega$  axis ( $\Delta \omega = 1.0^{\circ}$ ). A numerical absorption correction was made on the basis of an optimized description of the crystal faces. Experimental details and crystallographic data are summarized in Table 1. From the intensity data set the Laue symmetry *mmm* was determined and according to the systematic extinction conditions *hk0* (*h* = 2*n*) and *0kl* (*k* + *l* = 2*n*) the centrosymmetric space group *Pnma* was determined which was found to be correct during structure determination.

The structure was solved by direct methods (SIR97) [2], which resulted in the positions of all lanthanum atoms and part of the arsenic and nickel atoms. Next, the remaining arsenic and nickel positions 4*c* were localized step by step from successive difference Fourier syntheses. The program package JANA2000 was used for the structure refinement [3]. Anisotropic displacement parameters for all positions were included in the last refinement cycles. The final results are summarized in Table 1. A refinement of the occupancy factors gave no evidence for deviations from full

occupancy. Therefore the formula following from the structure determination is LaNi<sub>5</sub>As<sub>3</sub>, in full agreement with the microprobe analyses. The calculated X-ray powder diffraction pattern was in perfect agreement with the experimental one measured on a STOE STADI P powder diffractometer with Cu-K<sub> $\alpha$ 1</sub> radiation. Atomic coordinates, displacement parameters and selected interatomic distances are given in Tables 2 and 3, respectively. A projection of the LaNi<sub>5</sub>As<sub>3</sub> structure along the [010] direction is shown in Fig. 1.

Further details of the crystal structure investigation are available from the Fachinformationszentrum Karlsruhe, D-76344 Eggenstein-Leopoldhafen (Germany), on quoting the depository number CSD-414080, the name of the authors, and citation of the paper.

## Discussion

The structure of the ternary arsenide LaNi<sub>5</sub>As<sub>3</sub> represents a new structure type in the family of ternary



Fig. 2.  $RM_6X_3$  building units and their arrangements in the structures of LaNi<sub>5</sub>As<sub>3</sub> and CeNi<sub>5-x</sub>P<sub>3</sub>. The units drawn with thick lines are shifted by 1/2 of the translation vector in the projection direction.

rare earth transition metal-pnictides. It belongs to the numerous structures with trigonal prismatic coordination of non metals atoms, having a metal/non-metal ratio equal or close to 2:1 [4].

The main characteristic coordination polyhedra (CP) of the atoms in LaNi<sub>5</sub>As<sub>3</sub> are drawn in Fig. 1. The lanthanum atoms have high coordination numbers (CN); 23 and 20 for the lanthanum atoms La1 and La2, respectively. The La1 atom is surrounded by 23 atoms including Ni9 at a large distance of 3.918 Å, and the two La1 atoms neighbouring in the [010] direction. The basic polyhedron corresponds to a heptagonal prism with seven additional atoms outside the rectangular faces of the prism, that is a 7 face-capped heptagonal prism. Such an environment is also observed for the La atoms in other structures like those of La<sub>2</sub>Ni<sub>12</sub>P<sub>5</sub>, La<sub>3</sub>Ni<sub>12</sub>P<sub>7</sub>, or LaCo<sub>5</sub>P<sub>3</sub> [5-7]. The atom La2 has 20 neighbours in its nearest environment. Without the two La2 atoms at a distance of 3.913 Å, the basic polyhedron corresponds to a 6 face-capped hexagonal prism. This coordination scheme is frequently found in pnictide chemistry for light or heavy rare earths and even zirconium, as for example in the structures types Zr<sub>2</sub>Fe<sub>12</sub>P<sub>7</sub>,  $La_6Rh_{32}P_{17}$ ,  $CeNi_{5-x}P_3$ ,  $Ho_5Ni_{19}P_{12}$ ,  $HoCo_3P_2$ , YCo<sub>5</sub>P<sub>3</sub> [8-13].



Fig. 3. Stacking of  $RM_6X_3$  units in the structures of LaCo<sub>5</sub>P<sub>3</sub>, YCo<sub>5</sub>P<sub>3</sub>, and YNi<sub>5</sub>Si<sub>3</sub>. The units drawn with thick lines are shifted by 1/2 of the translation vector in the projection direction.

From ten different crystallographic sites of nickel, only Ni9 has a larger CN of 15 instead of 12 for all the other Ni atoms. This peculiar CN is due to the presence of a polyhedron (5As + 8Ni + 2La) based on a 5 face-capped pentagonal prism. A similar environment has also been found for transition elements, like Ni or Co, in several other related structures like those of  $CeNi_{5-x}P_3$  [10],  $Ho_5Ni_{19}P_{12}$  [11], and  $YCo_5P_3$  [13]. For the other Ni atoms, the CN is equal to 12 (more or less distorted four face-capped "rectangular" prisms). It is worth noting that the CP of Ni4 is built in a different manner, as Ni4 is surrounded by four As atoms in a "square" planar coordination contrary to the other Ni atoms, except the Ni9 atom, which are all tetrahedrally coordinated by As atoms. The CN of 12 is common



Fig. 4. Stacking modes of the  $RM_6X_3$  units present in the structures of  $Zr_2Fe_{12}P_7$ ,  $Yb_6Co_{30}P_{19}$ , and  $Yb_5Ni_{19}P_{12}$ . The units drawn with thick lines are shifted by 1/2 of the translation vector in the projection direction.

for *3d* elements like Fe, Co and Ni in ternary pnictides with rare earth and transition elements.

Finally, all the arsenic atoms occupy centres of trigonal prisms, which are face-capped by three additional

- V. Babizhetskyy, C. Le Sénéchal, J. Bauer, S. Députier, R. Guérin. J. Alloys Comp. 287, 174 (1999).
- [2] A. Altomare, M. C. Burla, M. Camalli, B. Carrozzini, G. L. Cascarano, C. Giacovazzo, A. Guagliardi, A. G. G. Moliterni, G. Polidori, R. Rizzi, J. Appl. Crystallogr. **32**, 115 (1999).
- [3] V. Petricek, M. Dusek, JANA 2000: Crystallographic Computing System for Ordinary and Modulated Structures, Institute of Physics, ASCR, Praha, Czech Republic.

metal atoms. This trigonal-prismatic environment of metalloïd atoms is typical of the large family of pnictides and even silicides, having a metal/non metal ratio equal or close to 2:1 [4].

The interatomic distances in the structure (Table 3) are slightly shorter than the sum of the respective atomic radii. Especially short distances are observed in the nickel-arsenic network: 2.32 Å<d(As-Ni)<2.42 Å as compared to  $r_{\rm Ni} = 1.24$  Å and  $r_{\rm As} = 1.18$  Å [14, 15].

The structure of LaNi<sub>5</sub>As<sub>3</sub> can be easily described by units of composition  $LaNi_6As_3$  which are connected via common Ni atoms in zigzag chains. These chains extend along the [100] direction (Fig. 2). Two neighbouring chains are separated from each other by b/2. It is worth noting that each  $LaNi_6As_3$  unit within the chain is connected by nickel atoms to a terminal  $LaNi_6As_3$  unit. These terminal units alternate on the left and right side of the chain.

In a general manner, these building units are called  $RM_6X_3$  units in literature (R is a rare earth metal, M a transition metal, X represents the metalloïd atom: P, As, Si) [4]. The structure of CeNi<sub>5-x</sub>P<sub>3</sub> [10] represents another stacking sequence of these units as finite linear chains, with only four units (two up- and two downpointed) per chain (Fig. 2). Similar building units in the form of infinite chains are also present in the structures of LaCo<sub>5</sub>P<sub>3</sub> or LaNi<sub>5</sub>P<sub>3</sub> [7], YCo<sub>5</sub>P<sub>3</sub> [13], and YNi<sub>5</sub>Si<sub>3</sub> [16] with similar composition (Fig. 3). The ternary silicide is the structural example where infinite linear chains are observed, while infinite zigzag chains are present in the structures of LaCo<sub>5</sub>P<sub>3</sub> (tilting after each unit) and  $YCo_5P_3$  (tilting after two units). Numerous structural examples, which exhibit other possible arrangements of  $RM_6X_3$  units but connected in different ways can be found in the literature [4]. Fig. 4 shows the stacking sequence of these units in the structure types Zr<sub>2</sub>Fe<sub>12</sub>P<sub>7</sub> [8], Yb<sub>6</sub>Co<sub>30</sub>P<sub>19</sub> [17], and Yb5Ni19P12 [18].

- [4] Yu. Kuz'ma, S. Chykhrij, Phosphides, in: Handbook on the Physics and Chemistry of Rare Earths, Vol. 23, Chapter 156, p. 285, Elsevier, Amsterdam (1996).
- [5] Yu. Kuz'ma, V. Babizhetskyy, S. Chykhrij, S. Oryshchyn, V. Pecharsky, Z. Anorg. Allg. 619, 587 (1993).
- [6] V. Babizhetskyy, S. Chykhrij, S. Oryshchyn, Yu. Kuz'ma, Dopov. Akad. Nauk Ukr. RSR, 9, 116 (1992).
- [7] V. Davydov, Yu. Kuz'ma, Dopov. Akad. Nauk Ukr. RSR, Ser. A 1, 81 (1981).

- [8] E. Ganglberger, Monatsh. Chem. **99**, 557 (1968).
- [9] J. Y. Pivan, R. Guérin, O. Pena, J. Padiou, M. Sergent, Mater. Res. Bull. 23, 513 (1988).
- [10] V. Babizhetskyy, S. Oryshchyn, S. Chykhrij, Yu. Kuz'ma, Kristallografiya 37, 1024 (1992).
- [11] J. Y. Pivan, R. Guérin, M. Sergent, Inorg. Chim. Acta. 109, 221 (1985).
- [12] W. Jeitschko, U. Jakubowski, J. Less-Common Met. 110, 339 (1985).
- [13] U. Meisen, W. Jeitschko, J. Less-Common Met. 102, 127 (1984).
- [14] F. Laves, Theory of Alloy Phases, ASM, Cleveland, OH (1956).
- [15] L. Pauling, Nature of The Chemical Bond, 3<sup>rd</sup> ed, Cornell University Press, Ithaca, NY (1960).
- [16] L. Aksel'rud, V. Jarovets, O. Bodak, Kristallografiya 21, 383 (1976).
- [17] W. Jeitschko, U. Jakubowski-Ripke, Z. Kristallogr. 207, 69 (1993).
- [18] S. Chykhrij, Yu. Kuz'ma, V. Davydov, S. Budnyk, S. Oryshchyn, Kristallografia 43, 596 (1998); Crystallogr. Rep. 43, 548 (1998).