Crystal structure of bis(tetramethylammonium) rubidium heptaarsenide–ammonia(1/1), $[\text{N}(\text{CH}_3)_4]_2\text{RbAs}_7(\text{NH}_3)$

N. Korber

Universität Bonn, Institut für Anorganische Chemie, Gerhard-Domagk-Str. 1, D-53121 Bonn, Germany

and H. G. von Schnering

Max-Planck-Institut für Festkörperforschung, Heisenbergstr. 1, D-70569 Stuttgart, Germany

Received August 8, 1996, transferred to 2nd update of database ICSD in 1997, CSD-No. 402583

$\text{C}_8\text{H}_{27}\text{As}_7\text{N}_3\text{Rb}$, monoclinic, $P12_1/c1$ (No. 14), $a = 13.275(3) \text{ \AA}$, $b = 8.813(2) \text{ \AA}$, $c = 19.142(9) \text{ \AA}$, $\beta = 100.94(3)^\circ$, $V = 2198.8 \text{ \AA}^3$, $Z = 4$, $R(F) = 0.063$, $R_w(F^2) = 0.149$.

Table 1. Parameters used for the X-ray data collection

Crystal:	red block, size 0.152 x 0.380 x 0.494 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ :	126.99 cm ⁻¹
Diffractometer:	Stoe STADI4
Scan mode:	variable scan speed ω
$T_{\text{measurement}}$:	200 K
$2\theta_{\text{max}}$:	40°
$N(hkl)_{\text{unique}}$:	1846
Criterion for I_o :	$I_o > 2 \sigma(I_o)$
$N(\text{param})_{\text{refined}}$:	181
Programs:	SHELXS-86, SHELXL-93

Table 2. Final atomic coordinates and displacement parameters (in Å²)

Atom	Site	x	y	z	U_{iso}
H(11A)	4e	0.167(6)	0.386(5)	0.342(3)	0.03(1)
H(11B)	4e	0.052(3)	0.399(6)	0.304(5)	0.03(1)
H(11C)	4e	0.138(8)	0.468(2)	0.268(3)	0.03(1)
H(12A)	4e	0.287(2)	0.23(1)	0.279(1)	0.03(1)
H(12B)	4e	0.242(4)	0.311(8)	0.207(5)	0.03(1)
H(12C)	4e	0.238(4)	0.134(6)	0.213(6)	0.03(1)
H(13A)	4e	0.176(6)	0.118(8)	0.351(3)	0.03(1)
H(13B)	4e	0.132(9)	0.014(3)	0.286(2)	0.03(1)
H(13C)	4e	0.057(4)	0.109(8)	0.323(5)	0.03(1)
H(14A)	4e	0.053(6)	0.127(6)	0.176(4)	0.03(1)
H(14B)	4e	0.057(6)	0.303(9)	0.164(3)	0.03(1)
H(14C)	4e	-0.016(2)	0.24(1)	0.211(2)	0.03(1)
H(21A)	4e	0.425(4)	0.15(1)	0.168(4)	0.03(1)
H(21B)	4e	0.441(5)	0.321(4)	0.189(5)	0.03(1)
H(21C)	4e	0.491(2)	0.19(1)	0.242(2)	0.03(1)
H(22A)	4e	0.513(8)	0.299(5)	0.058(1)	0.03(1)
H(22B)	4e	0.488(7)	0.13(1)	0.069(1)	0.03(1)
H(22C)	4e	0.599(3)	0.18(1)	0.062(2)	0.03(1)
H(23A)	4e	0.569(2)	0.451(4)	0.176(6)	0.03(1)
H(23B)	4e	0.659(7)	0.396(6)	0.140(4)	0.03(1)
H(23C)	4e	0.664(7)	0.369(5)	0.222(3)	0.03(1)
H(24A)	4e	0.609(4)	0.016(3)	0.181(6)	0.03(1)
H(24B)	4e	0.653(7)	0.129(9)	0.243(1)	0.03(1)
H(24C)	4e	0.701(4)	0.123(9)	0.174(5)	0.03(1)

Source of material: $[\text{NMe}_4]_2\text{RbAs}_7(\text{NH}_3)$ was prepared from Rb_3As_7 and NMe_4I (molar ratio 1:2) in liquid ammonia at 233 K by slow diffusion through a G3 glass frit. A dark red, crystalline product began to precipitate after 12 h, the reaction was complete after 5 d. The solvent was removed by slow evaporation and the residue washed three times with liquid ammonia to remove RbI . The total yield was 79%. The product is very sensitive towards air and moisture. It decomposes at room temperature within 3–6 h, depending on the size of the crystals. The H atoms of the NH_3 group (N3) are not determined.

The structure of $[\text{NMe}_4]_2\text{RbAs}_7(\text{NH}_3)$ is isotypic to that of the analogous heptaphosphide $[\text{NMe}_4]_2\text{RbP}_7(\text{NH}_3)$ (see ref. 1). It consists of one-dimensionally infinite $[\text{RbAs}_7]^{2-}$ chains running parallel to the crystallographic b axis. The chains are separated by NMe_4^+ ions and NH_3 molecules. The Rb ions are coordinated by eight As atoms from three different cages.

Table 3. Final atomic coordinates and displacement parameters (in Å²)

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
Rb(1)	4e	0.0673(2)	0.2309(3)	−0.0043(1)	0.027(1)	0.020(1)	0.046(2)	−0.001(1)	−0.003(1)	−0.003(1)
As(1)	4e	−0.2453(2)	0.0502(3)	−0.0786(1)	0.037(2)	0.010(1)	0.040(2)	−0.003(1)	−0.002(1)	−0.004(1)
As(2)	4e	−0.1823(2)	0.3950(3)	0.0206(1)	0.038(2)	0.017(2)	0.032(2)	−0.003(1)	0.002(1)	−0.011(1)
As(3)	4e	−0.3352(2)	0.4332(3)	−0.0771(1)	0.040(2)	0.027(2)	0.043(2)	0.020(1)	0.000(1)	0.003(1)
As(4)	4e	−0.1411(2)	0.1843(3)	−0.1486(1)	0.024(1)	0.035(2)	0.030(2)	0.003(1)	0.006(1)	−0.008(1)
As(5)	4e	−0.4007(2)	0.1919(3)	−0.1058(1)	0.022(1)	0.045(2)	0.030(2)	−0.006(1)	−0.002(1)	−0.003(1)
As(6)	4e	−0.1730(2)	0.1356(3)	0.0400(1)	0.032(1)	0.021(2)	0.028(2)	0.000(1)	−0.003(1)	0.008(1)
As(7)	4e	−0.1595(2)	0.4292(3)	−0.1067(1)	0.041(2)	0.020(2)	0.038(2)	−0.011(1)	0.003(1)	0.008(1)
N(1)	4e	0.130(1)	0.238(2)	0.2595(8)	0.016(4)	0.015(5)	0.016(4)	0.000(2)	0.003(2)	0.000(2)
C(11)	4e	0.121(2)	0.386(2)	0.297(1)	0.018(6)	0.015(6)	0.019(6)	−0.001(4)	0.002(4)	0.001(4)
C(12)	4e	0.234(1)	0.227(3)	0.238(1)	0.022(6)	0.018(6)	0.022(6)	0.001(4)	0.003(4)	0.000(4)
C(13)	4e	0.123(2)	0.108(3)	0.309(1)	0.029(7)	0.028(7)	0.030(7)	−0.001(4)	0.005(4)	0.002(4)
C(14)	4e	0.049(2)	0.225(3)	0.197(1)	0.024(6)	0.026(7)	0.025(7)	−0.003(4)	0.004(4)	0.000(4)
N(2)	4e	0.568(1)	0.228(2)	0.1605(9)	0.019(5)	0.020(5)	0.020(5)	0.000(2)	0.003(2)	−0.001(2)
C(21)	4e	0.472(2)	0.223(3)	0.193(1)	0.031(7)	0.028(7)	0.030(7)	0.000(4)	0.006(4)	0.001(4)
C(22)	4e	0.540(2)	0.206(3)	0.080(1)	0.033(7)	0.034(7)	0.033(7)	0.001(4)	0.006(4)	0.001(4)
C(23)	4e	0.619(2)	0.373(3)	0.176(1)	0.035(7)	0.034(7)	0.035(7)	−0.002(4)	0.007(4)	0.001(4)
C(24)	4e	0.638(2)	0.115(3)	0.192(1)	0.039(7)	0.040(8)	0.041(7)	0.002(4)	0.006(4)	0.000(4)
N(3)	4e	0.332(2)	0.231(3)	0.932(1)	0.046(6)	0.046(6)	0.046(6)	0.001(2)	0.008(2)	−0.001(2)

References

1. Korber, N.; von Schnering, H. G.: Alkali Metal Polyphosphides in low Dimensions: Synthesis and Structures of [(CH₃)₄N]₂RbP₇(NH₃) and [(C₂H₅)₄N]₄Cs₂P₁₁. *Chem. Ber.* **129** (1996) 155–159.
2. Sheldrick, G. M.: SHELXS-86. Program for the solution of crystal structures. University of Göttingen, Germany 1986.
3. Sheldrick, G. M.: SHELXL-93, a program for refining crystal structures. University of Göttingen, Germany 1993.