

octaédriques et prismatiques des atomes de thulium, ces derniers occupant des sites prismatiques propres. Ce comportement se retrouve avec les composés KLS_2 ($L = La$ à Yb) (Ballestracci, 1965) dans lesquels le potassium occupe des sites octaédriques distincts de ceux des atomes de terres rares.

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Structure of Tetrapotassium Trisilver Heptanitrite and Potassium Silver Dinitrite Hemihydrate

BY YUKO OKAMURA, MAKOTO ISHIHARA, SHIGERU OHBA AND YOSHIHIKO SAITO

Department of Chemistry, Faculty of Science and Technology, Keio University, Hiyoshi 3, Kohoku-ku, Yokohama 223, Japan

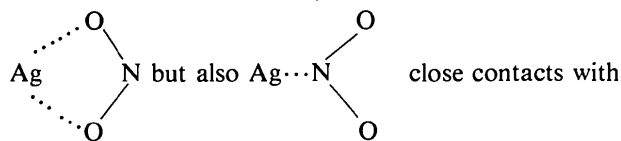
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Abstract. $Ag_3K_4(NO_2)_7$ (I), $M_r = 802.0$, orthorhombic, $Pnma$, $a = 11.370$ (4), $b = 11.010$ (3), $c = 14.091$ (3) Å, $V = 1764.0$ (8) Å³, $Z = 4$, $D_x = 3.02$ Mg m⁻³, $\lambda(Mo K\alpha) = 0.71073$ Å, $\mu = 4.29$ mm⁻¹, $F(000) = 1512$, $T = 297$ (1) K, final $R = 0.088$ for 1555 observed unique reflections. $AgK(NO_2)_2 \cdot \frac{1}{2}H_2O$ (II), $M_r = 248.0$, orthorhombic, $Cmca$, $a = 25.715$ (6), $b = 12.857$ (3), $c = 6.787$ (2) Å, $V = 2243.9$ (9) Å³, $Z = 16$, $D_m = 2.91$ (1), $D_x = 2.94$ Mg m⁻³, $\mu = 4.24$ mm⁻¹, $F(000) = 1872$, $T = 298$ (1) K, final $R = 0.071$ for 683 observed unique reflections. Rather high R values are attributable to the disordered structures. In (I) Ag^+ ions are disordered and in (I) and (II) NO_2^- ions exhibit novel positional disorder, where the positions of the O atoms are fixed and the N atom has two alternative positions.

The preference of a chelate ring formation of NO_2^- with Ag^+ was confirmed, suggesting that $Ag \cdots O$ attractive interaction is stronger than $Ag \cdots N$. One of the nitrite ions in (I) bridges among three Ag^+ cations. K^+ ions are coordinated by seven to eight O atoms.

Introduction. The pale yellow color of $AgNO_2$, $Ag_2Li(NO_2)_3$ and $AgNa(NO_2)_2$ crystals is due to the perturbation of NO_2^- by Ag^+ ions (Yamashita &

Azumi, 1984). In these crystals there exist not only



the Ag^+ ion lying nearly on the NO_2 plane. The temperature dependence of the structure of $AgNa(NO_2)_2$ (Ishihara, Ohba, Saito & Shiozaki, 1987) showed that $Ag \cdots O_2N$ attraction is stronger than $Ag \cdots NO_2$.

The structures of the title double salts were determined to study further the interactions between NO_2^- and Ag^+ ions. The unit cell constants and systematic absences of (II) have already been reported by Nardelli, Cavalca & Braibanti (1952).

Experimental. Crystals of (I) and (II) were grown from aqueous solution in different concentrations. (I): Pale yellow plate-like crystals grown from *ca* 22% KNO_2 aqueous solution saturated with $AgNO_2$, slightly hygroscopic. Spherically ground crystal of diameter 0.44 (2) mm. Rigaku AFC-5 four-circle diffractometer, graphite monochromator. Laue group mmm , systematic absences $0kl$ with k odd and $h0l$ with $h+l$

Table 1. Positional parameters (×10⁴) and equivalent isotropic temperature factors (Hamilton, 1959)

Compound (I)	x	y	z	B/B _{eq} (Å ² ×10)
Ag(11)*	3217 (2)	7500	2979 (2)	30
Ag(12)*	3723 (3)	7500	2938 (2)	39
Ag(21)*	2995 (2)	7500	917 (2)	30
Ag(22)*	3334 (3)	7500	971 (2)	37
Ag(3)	3588 (2)	2500	1982 (2)	71
K(1)	1655 (2)	5045 (2)	4188 (2)	28
K(2)	475 (2)	119 (3)	1225 (2)	30
N(11)*	1500	2500	2650	49
O(1)	1004 (9)	1544 (8)	2739 (6)	40
N(2)	1315 (15)	2500	96 (10)	37
O(2)	1335 (8)	1539 (8)	-389 (6)	36
N(3)	4573 (15)	2500	217 (15)	50
O(3)	5040 (10)	1546 (9)	-29 (9)	53
N(41)*	600	7500	2500	57
O(4)	1047 (11)	6551 (10)	2701 (9)	63
N(51)*	3800	2500	4300	43
O(5)	3551 (9)	1547 (9)	4674 (9)	52
N(6)	3320 (12)	4736 (15)	1863 (10)	57
O(61)	2951 (11)	5268 (15)	1213 (9)	71
O(62)	3546 (9)	5248 (9)	2604 (8)	47
N(12)*	500	2500	2700	39
N(42)*	1600	7500	2700	39
N(52)*	3100	2500	4800	34

Compound (II)	x	y	z	B/B _{eq} (Å ² ×10)
Ag	1537 (1)	3018 (1)	4064 (3)	64
K(1)	0	1449 (3)	891 (7)	35
K(2)	2119 (1)	0	5000	35
O(31)	681 (4)	2130 (8)	3938 (15)	45
O(32)	1298 (4)	1338 (8)	5230 (17)	49
N(3)	830 (4)	1320 (9)	4784 (19)	41
O(W)	0	4535 (10)	2337 (17)	32
O(11)†	970	5000	5000	57
O(12)†	1780	5000	5000	96
N(1)†	1375	4550	5000	71
O(21)†	2500	1800	2500	52
O(22)†	2500	3400	2500	56
N(2)†	2270	2600	2650	45

* Population parameters of the disordered atoms: N(11), 0.70; N(12), 0.30; N(41), 0.80; N(42), 0.20; N(51), 0.80; N(52), 0.20; Ag⁺ ions 0.50. As the positional parameters for N were fixed, there are no standard deviations.

† Population parameters of the disordered NO₂⁻ ion 0.5. As the positional parameters were fixed, there are no standard deviations.

odd [*ba*c̄ setting of *Pna*2₁ (No. 33) or *cab* setting of *Pnma* (No. 62)]; cell parameters refined by least squares for 20 2θ values (20° < 2θ < 30°), intensity measurement performed to 2θ_{max} = 55° (2θ ≤ 40°, *h* -10→10, *k* -10→10, *l* 0→13; 40 < 2θ ≤ 55°, *h* -14→14, *k* 0→14, *l* 0→18), θ-2θ scan, scan speed 6° min⁻¹ in θ. Variation of |F_o| of five standard reflections, 0.99 < Σ(|F_o|/|F_o|_{initial})/5 < 1.00, 5636 reflections measured, 4063 observed reflections with |F_o| > 3σ(|F_o|), 1555 unique (R_{int} = 0.022). Correction for absorption (μ_r = 0.94, 0.19 < A < 0.21). At first, the space group *Pnma* was assumed and the indices were changed to be *klh*. The *v* coordinates of the higher peaks in the Patterson function, P(*u,v,w*) converged into the ranges 0.00-0.04 and 0.46-0.50, suggesting that Ag atoms lie on mirror planes at *y* = ¼ or ¾. Two possible arrangements of three independent Ag atoms were deduced. One of them could be refined successfully and the remaining atoms were located on a Fourier synthesis. There are six independent NO₂⁻ ions, the N atoms of five of these lie on the mirror plane. By introducing anisotropy in thermal parameters, the R

Table 2. Interatomic distances (Å) and angles (°)

Compound (I)	Distance (Å)	Angle (°)	
N(11)-O(1)	1.20*	Ag(22)-O(61)	2.519 (16)
O(1)-N(11)-O(1)	122*	Ag(12)-O(62)	2.532 (10)
N(12)-O(1)	1.20*	Ag(3)-N(11)	2.554 (2)
O(1)-N(12)-O(1)	123*	Ag(11)-O(62)	2.563 (10)
N(2)-O(2)	1.260 (12)	Ag(11)-O(2 ^{vi})	2.582 (9)
O(2)-N(2)-O(2)	114.3 (12)	Ag(12)-O(2 ^{vi})	2.585 (9)
N(3)-O(3)	1.227 (14)	Ag(21)-O(5 ^{vii})	2.694 (12)
O(3)-N(3)-O(3)	117.8 (16)	Ag(11)-O(4)	2.708 (13)
O(41)-O(4)	1.20*	Ag(3)-N(3)	2.728 (21)
O(4)-N(41)-O(4 ⁱⁱ)	122*	Ag(21)-O(3 ^v)	2.768 (12)
N(42)-O(4)	1.22*	Ag(11)-N(41 ⁱⁱⁱⁱ)	2.792 (2)
O(4)-N(42)-O(4 ⁱⁱ)	118*	Ag(22)-N(3 ^v)	2.910 (19)
N(51)-O(5)	1.21*	Ag(3)-O(1 ⁱⁱⁱⁱ)	2.968 (9)
O(5)-N(51)-O(5)	121*	Ag(21)-N(42)	2.971 (3)
N(52)-O(5)	1.18*	Ag(12)-O(4 ^{viii})	2.981 (13)
O(5)-N(52)-O(5)	125*	K(2)-O(1)	2.716 (9)
N(6)-O(61)	1.165 (20)	K(1)-O(4)	2.760 (12)
N(6)-O(62)	1.214 (18)	K(1)-O(3 ^v)	2.768 (11)
O(61)-N(6)-O(62)	121.2 (16)	K(2)-O(62)	2.774 (11)
Ag(12)-Ag(22)	2.807 (4)	K(1)-O(1)	2.789 (9)
Ag(11)-Ag(22)	2.833 (4)	K(1)-O(3 ⁱⁱⁱ)	2.801 (11)
Ag(11)-Ag(21)	2.917 (4)	K(2)-O(61 ^v)	2.847 (13)
Ag(12)-Ag(21)	2.966 (4)	K(2)-O(4)	2.851 (12)
Ag(11)-N(42)	1.880 (2)†	K(1)-O(5 ^v)	2.862 (11)
Ag(21)-N(52 ^{vii})	2.007 (3)†	K(1)-O(2 ^{vi})	2.878 (9)
Ag(3)-N(12 ^{viii})	2.220 (2)	K(1)-O(61 ^v)	2.909 (13)
Ag(12)-N(41 ^{viii})	2.222 (3)	K(2)-O(2)	2.928 (9)
Ag(22)-N(52 ^{vii})	2.320 (3)	K(2)-O(5 ⁱⁱⁱ)	2.977 (11)
Ag(12)-N(42)	2.437 (4)	K(2)-O(2 ⁱⁱⁱ)	2.993 (9)
Ag(3)-N(6)	2.486 (16)	K(2)-O(5 ⁱⁱⁱⁱ)	3.061 (12)
Ag(21)-O(61)	2.493 (16)	K(1)-O(62)	3.107 (11)
Ag(22)-O(3 ^v)	2.507 (12)		

Symmetry code: (i) *x*, -*y*+½, *z*; (ii) *x*, -*y*+¾, *z*; (iii) *x*-½, *y*, *z*+½; (iv) -*x*+½, *y*-½, *z*+½; (v) -*x*+½, *y*-½, *z*-½; (vi) -*x*+1, -*y*+1, -*z*; (vii) -*x*+½, *y*+½, *z*-½; (viii) *x*+½, *y*, -*z*+½; (ix) -*x*+½, *y*+½, *z*+½; (x) -*x*+1, *y*+½, -*z*; (xi) -*x*+½, -*y*+1, *z*+½; (xii) -*x*, -*y*, -*z*; (xiii) -*x*+½, -*y*, *z*-½; (xiv) *x*-½, -*y*+½, -*z*+½.

Compound (II)	Distance (Å)	Angle (°)	
N(1)-O(11)	1.19*	Ag-O(11)	3.004 (2)
N(1)-O(12)	1.19*	K(2)-O(32)	2.728 (10)
O(11)-N(1)-O(12)	122*	K(2)-O(32 ⁱⁱ)	2.728 (10)
N(2)-O(21)	1.19*	K(1)-O(W ⁱⁱ)	2.739 (13)
N(2)-O(22)	1.19*	K(1)-O(W)	2.739 (13)
O(21)-N(2)-O(22)	120*	K(2)-O(12 ^{vi})	2.831 (3)
N(3)-O(31)	1.249 (16)	K(2)-O(22 ^v)	2.841 (1)
N(3)-O(32)	1.241 (15)	K(2)-O(22 ^{vii})	2.841 (1)
O(31)-N(3)-O(32)	113.2 (11)	K(1)-O(31)	2.848 (11)
Ag-N(1)	2.111 (1)	K(1)-O(31 ⁱⁱⁱⁱ)	2.848 (11)
Ag-N(2)	2.182 (2)	K(1)-O(31 ^{vii})	2.857 (11)
Ag-O(32)	2.381 (11)	K(1)-O(31 ^{vi})	2.857 (11)
Ag-O(31)	2.481 (11)	K(2)-O(21)	3.032 (1)
Ag-O(12)	2.700 (2)	K(2)-O(21 ^v)	3.032 (1)
Ag-O(22)	2.739 (3)	K(1)-O(11 ^v)	3.171 (2)
Ag-O(32 ⁱⁱⁱ)	2.799 (12)	K(1)-O(11 ^{vii})	3.171 (2)
Ag-N(3)	2.883 (11)		

Symmetry code: (1) *x*, -*y*+1, -*z*+1; (ii) -*x*+½, *y*, -*z*+½; (iii) *x*, -*y*+½, *z*-½; (iv) *x*, -*y*, -*z*+1; (v) -*x*, *y*-½, -*z*+½; (vi) -*x*+½, *y*-½, *z*; (vii) *x*, -*y*+½, *z*+½; (viii) -*x*, *y*, *z*; (ix) -*x*, -*y*+½, *z*+½.

* As the positional parameters were fixed, there are no standard deviations.

† These short distances are unrealistic (see text).

value was reduced from 0.203 to 0.103. No more atoms could be found on difference synthesis. The alternative space group, *Pna*2₁, was rejected because of a larger *R* value, 0.128.

The vacant space in the unit cell showed that there is no room for water of crystallization to be accommodated and that the Ag and N atoms on the mirror plane are loosely packed. Fourier synthesis suggested that the positions of Ag(1), Ag(2), N(1), N(4) and N(5) are disordered. They were divided into two possible positions according to the peak shape, and the split

atomic coordinates for N were fixed in the refinement. Their population parameters were estimated by the trial and error method with a restriction that the isotropic thermal parameters remain almost equal. Except for the N atoms, which have smaller occupation factors, anisotropic thermal parameters were introduced. $\sum w|F_o| - |F_c|^2$ minimized, $w^{-1} = \sigma^2(|F_o|) + (0.015|F_o|)^2$, final $R = 0.088$, $wR = 0.109$, $S = 5.33$ for 1555 unique reflections.* The large R value may be attributed to the disorder of the Ag and N atoms. Complex neutral atom scattering factors from *International Tables for X-ray Crystallography* (1974). Reflection/parameter ratio 8.0, $\Delta/\sigma < 0.82$, $-3.6 < \Delta\rho < 2.2$ e \AA^{-3} , UNICSIII program system (Sakurai & Kobayashi, 1979), FACOM M-380R computer of this university.

(II): Density measured by flotation method. Pale lemon yellow plate-like crystals grown from ca 82% KNO_2 aqueous solution saturated with AgNO_2 , hygro-

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51441 (25 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

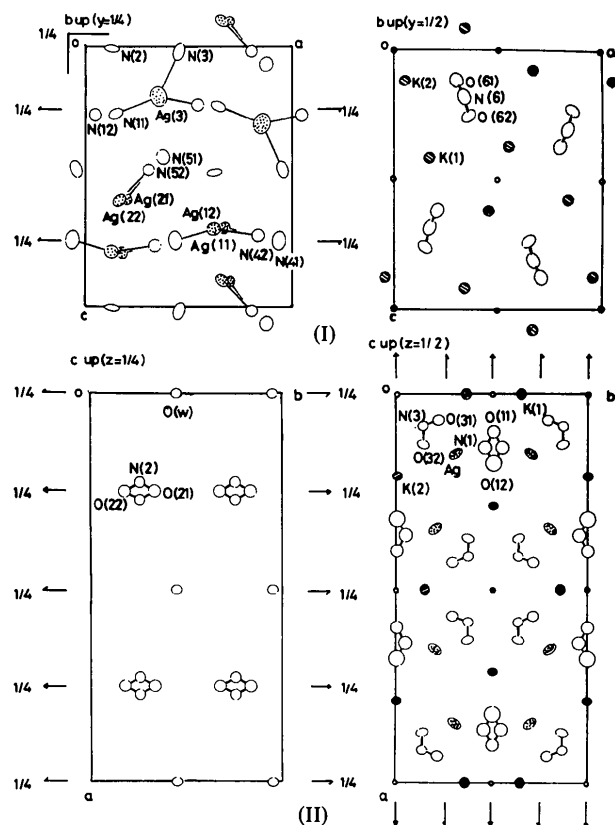


Fig. 1. Partial projections of the crystal structure of (I) along b near $y = \frac{1}{4}$ and $\frac{1}{2}$, and (II) along c near $z = \frac{1}{4}$ and $\frac{1}{2}$.

scopic. Spherically ground crystal of diameter 0.35 (2) mm. Laue group mmm , systematic absences hkl with $h+k$ odd, $0kl$ with k odd, $h0l$ with h odd or l odd and $hk0$ with h odd or k odd [$C2cb$, the $\bar{c}ba$ setting of $Aba2$ (No. 41) or $Cmca$ (No. 64)]; cell parameters refined by least squares for 20 2θ values ($20 < 2\theta < 30^\circ$), $2\theta_{\max} = 55^\circ$ ($h -33 \rightarrow 33$, $k 0 \rightarrow 16$, $l -8 \rightarrow 8$), five standard reflections, with $0.97 < \sum(|F_o|/|F_o|_{\text{initial}})/5 < 1.07$, 4944 reflections measured, 2387

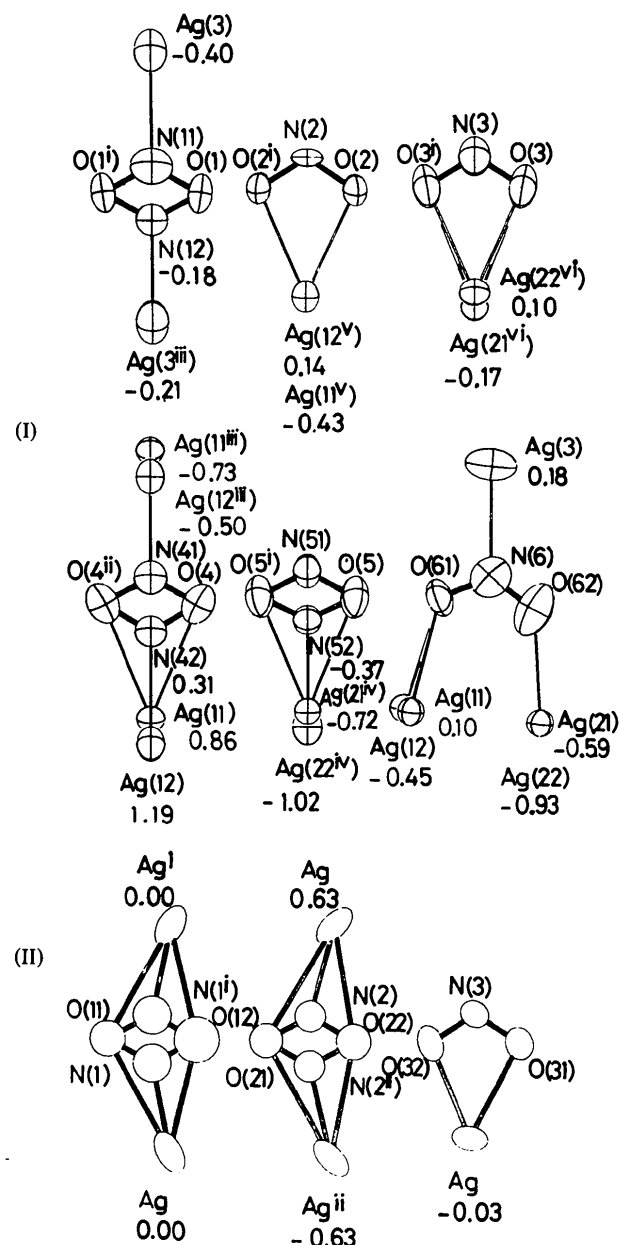


Fig. 2. Arrangements of Ag^+ ions around the NO_2^- ions in (I) and (II) with distances from the NO_2 plane less than 1.0 \AA , indicated by numbers near the atom labels. The symmetry code is given in Table 2. Distances in \AA .

observed, 683 unique ($R_{\text{int}} = 0.032$); $\mu r = 0.74$, $0.34 < A < 0.36$. With the assumption of space group *Cmca*, the structure could be solved by Patterson and Fourier methods. There are three independent nitrite ions, and two of these are disordered around the twofold axis. Positions of these disordered NO₂ ions were deduced from Fourier synthesis and fixed with isotropic thermal parameters and with weight $\frac{1}{2}$. Water H atoms could not be found on difference synthesis. Final $R = 0.071$, $wR = 0.083$, $S = 3.61$ for 683 unique reflections. Reflection/parameter ratio 7.7, $\Delta/\sigma < 0.19$, $-5.5 < \Delta\rho < 3.2 \text{ e } \text{Å}^{-3}$. Possibility of the space group *C2cb* was also carefully checked. However, every attempt to refine the structure based on this space group did not succeed ($R = 0.079$).

Discussion. Final atomic coordinates and interatomic distances and bond angles are presented in Tables 1 and 2. The crystal structures are shown in Fig. 1. Fig. 2 illustrates the arrangement of Ag⁺ cations around the nitrite ions, with shifts from the NO₂ plane less than 1 Å. Three of the six independent nitrite ions in (I) and two of three in (II) are disordered, where the positions of the two O atoms are almost fixed and the N atom takes two alternative positions, whereas in Ag₂Li(NO₂)₃ (Ohba, Matsumoto, Ishihara & Saito, 1986) and AgNa(NO₂)₂ (Ishihara *et al.*, 1987), the center of gravity of the disordered NO₂⁻ ion is fixed. For each ordered nitrite ion, except for NO₂⁻ (6) in (I), the chelated position between the two O atoms is occupied by an Ag⁺ ion and there is no Ag...N close contact. This feature indicates that Ag...O₂ attraction is stronger than Ag...N as expected from the temperature dependence of the disorder of nitrite ions in AgNa(NO₂)₂ (Ishihara *et al.*, 1987). The NO₂⁻ (6) ion of (I) is surrounded by five K⁺ ions nearly at $y = \frac{1}{2}$ as shown in Fig. 1. It acts as a bridge among three Ag⁺ ions at $y = \frac{1}{4}$ or $\frac{3}{4}$ as seen from Fig. 2. As far as we know, this is the

first example of this form of bridging, although three other forms are found in polynuclear metal complexes (Finney, Hitchman, Raston, Rowbottom & White, 1981). The distances between [Ag(11), Ag(12)] and [Ag(21), Ag(22)] range from 2.807 (4) to 2.966 (4) Å, which are nearly equal to 2.88 Å, the distance between nearest neighbors in metallic silver. The distances Ag(11)...N(42), 1.880 (2) Å, and Ag(21^{iv})...N(52), 2.007 (3) Å, in (I) are much shorter than the normal Ag...N distances observed in AgNO₂ (Ohba & Saito, 1981), Ag₂Li(NO₂)₃ and AgNa(NO₂)₂ crystals, ranging from 2.154 (8) to 2.327 (4) Å. In view of such unrealistic Ag...N distances, Ag(11) must be vacant if N(42) is occupied and *vice versa*. The same is true for the Ag(21) and N(52) positions. K⁺ ions are coordinated by seven to eight O atoms with distances from 2.716 (9) to 3.171 (2) Å. The crystal structure determinations suffered from the ambiguity between a centrosymmetric and a noncentrosymmetric space group. This problem was discussed by Marsh (1986).

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Structure de Na(Al_{1,5}As_{0,5})(As₂O₇)₂

PAR AHMED DRISS ET TAHAR JOUINI

Département de Chimie, Faculté des Sciences, Campus Universitaire, 1060 Tunis, Tunisie

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Abstract. $M_r = 624.60$, triclinic, $P\bar{1}$, $a = 7.727$ (4), $b = 7.118$ (2), $c = 4.839$ (2) Å, $\alpha = 104.43$ (3), $\beta = 93.71$ (3), $\gamma = 90.07$ (4)°, $V = 257.2$ Å³, $Z = 1$, D_m (in bromobenzene) = 4.02, $D_x = 4.03 \text{ Mg m}^{-3}$, $\lambda(\text{Ag K}\alpha) = 0.5608$ Å, $\mu = 8.23 \text{ mm}^{-1}$, $F(000) = 291$, final R

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= 0.038 and $wR = 0.038$ for 1102 independent reflections. This structure provides the first example of the partial replacement of aluminium by arsenic giving rise to mixed $X(1)\text{O}_6$ and $X(2)\text{O}_6$ octahedra containing different amounts of Al^{III} and As^V. The preferential

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