# [Ag(NH$_3$)$_2$]Ag(OsO$_3$N)$_2$: a new nitridoosmate(VIII)

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## Abstract

Dark brown single crystals of [Ag(NH$_3$)$_2$]Ag(OsO$_3$N)$_2$ were obtained from the reaction of Ag$_2$CO$_3$, OsO$_4$, and NH$_3$ in aqueous solution. The crystal structure was solved in the monoclinic space group $C2/m$, with the following unit-cell dimensions: $a = 1962.5(3)$, $b = 633.1(1)$, $c = 812.6(1)$ pm, $\beta = 96.71(1)°$. The final reliability factor was $R = 0.0256$ for 1034 reflections with $I > 2\sigma(I)$. Linear [Ag(NH$_3$)$_2$]$^+$ ions are present oriented perpendicular to the [010] direction, leading to short Ag$^+$–Ag$^+$ distances of 316 pm. A second type of Ag$^+$ ions in the crystal structure present coordination number "6 + 1" and are surrounded by oxygen and nitrogen atoms of the nitridoosmate groups. Within the first of the two crystallographically distinguishable anions one can clearly differentiate between oxygen and nitrogen atoms while the second one exhibits a N/O disorder over two positions. The infrared spectrum of [Ag(NH$_3$)$_2$]Ag(OsO$_3$N)$_2$ shows the typical absorptions which can be attributed to the complex anions and the NH$_3$ ligands.

## 1. Introduction

The nitridoosmate ion has been subject of several investigations including vibrational [1–6] and electronic spectroscopy [6–8] as well as structure solutions [3–5,9–15] and chemical behavior [16–18]. According to these findings it is believed that the tetrahedral ion contains an Os–N triple bond what is displayed by the distances within the tetrahedron which are about 175 pm for the Os–O, and 168 pm for the Os–N bond, respectively. The different bonding situation of oxygen and nitrogen atoms in OsO$_3$N$^-$ is also reflected by the IR spectra in which the Os–N stretching vibration at about 1021 cm$^{-1}$ can be clearly distinguished from the respective Os–O vibrations in the range from 870 to 890 cm$^{-1}$ [1–6]. Unfortunately, the crystal structures of nitridoosmates which are known up to now do not clearly show these bond length differences in any case. As an example KOsO$_3$N may serve which crystallizes with the scheelite structure (tetragonal, $I4_1/a$) and shows only one distance within the tetrahedron [10,13]. This is explained by the assumption that the OsO$_3$N$^-$ anion is disordered in a

way that each vertex is occupied by oxygen and nitrogen atoms in a ratio of 3:1. Thus, the observed distances are average values of the "real" Os–O and Os–N distances. A similar situation is found for RbOsO$_3$N, although it has orthorhombic symmetry [14]. In CsOsO$_3$N, however, one can distinguish between the Os–N (167 pm) and Os–O (174 pm) bonds [15]. At present, the number of structurally characterized nitridoosmates is very limited and further investigations are obviously necessary. In this respect the crystal structure of [Ag(NH$_3$)$_2$]Ag(OsO$_3$N)$_2$ seems to be interesting because at least one OsO$_3$N$^-$ ion is not disordered in the structure and allows the inspection of the different distances. Besides these general aspects our interest for the preparation of silvernitridoosmates arises in particular from its potential as a starting material for the synthesis of nitridoosmates of higher valent metals, especially of the rare earth elements. Since a couple of years we are interested in lanthanide compounds with complex oxo-anions [19]. In the case of tetrahedral anions we investigated perchlorates, hydrogensulfates, amidosulfates and methanesulfonates and started recently to extend our efforts towards the synthesis of lanthanoide compounds containing *transition metal oxo-anions*. For the respective monovalent species the present knowledge

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is mainly restricted to perrhenates [20–26], so we thought the nitridoosmate ion, $OsO_3N^-$, to be another interesting tetrahedral and monovalent anion. A simple synthetic approach for lanthanoide nitridoosmates seemed to be the metathetical reaction of rare earth trihalides with $Ag(OsO_3N)$. The preparation of the latter and several other nitridoosmates has been described some 10 years ago [3]. To our surprise, however, accurate structural data of $Ag(OsO_3N)$ are not available. Because the knowledge of our starting materials is of fundamental importance, we decided to characterize $Ag(OsO_3N)$ by means of single-crystal diffraction. However, our attempts to obtain appropriate single crystals of the silver compound led now to a new, hitherto unknown silver nitridosmate, $[Ag(NH_3)_2]Ag(OsO_3N)_2$. The interesting structural features of $[Ag(NH_3)_2]Ag(OsO_3N)_2$ forced us to present this unique compound in this paper.

## 2. Experimental section

### 2.1. Synthesis

To a suspension of 300 mg of $Ag_2CO_3$ (Merck, p.a.) in 5 mL distilled water 100 mg of $OsO_4$ (degussa, p.a.) were given. After addition of 3 mL aqueous $NH_3$ the solution turned clear and was stirred for 1 h at 40°C. The solution was kept at room temperature and dark brown, nearly black crystals separated out within a few days. They were recovered by suction for further investigations.

### 2.2. X-ray crystallography

Several crystals were selected with the help of a microscope and sealed in glass capillaries. For all of them the same monoclinic C-centered unit cell was determined with the help of an image plate diffractometer (STOE IPDS II). For the respective best specimen reflection intensities were collected using the same diffractometer. The structure solution was successful assuming the space group $C2/m$ using direct methods provided by the program SHELXS-86 [27]. A least square refinement was carried out with SHELXL-97 [28], and for all atoms anisotropic thermal displacement parameters were introduced. Finally, a numerical absorption correction was carried out using the programs X-SHAPE [29] and X-RED [30]. For one crystallographic site a mixed occupation with oxygen and nitrogen must be assumed (see Results and discussion). Details of the data acquisition and the crystallographic data are summarized in Tables 1–3. Additionally, the data have been deposited with the Fachinformationszentrum Karlsruhe, D-76344 Eggenstein-Leopoldshafen (crysdata@FIZ-Karlsruhe.de) and

Table 1
$[Ag(NH_3)_2]Ag(OsO_3N)_2$: crystallographic data

| | |
|---|---|
| Formula weight (g/mol) | 748.18 |
| Unit-cell parameters | $a = 1962.5(3)$ pm |
| | $b = 633.1(1)$ pm |
| | $c = 812.6(1)$ pm |
| | $\beta = 96.71(1)°$ |
| Cell volume | 1002.7(3) Å$^3$ |
| Z | 4 |
| Crystal shape | Prismatic |
| Crystal size | $0.1 \times 0.15 \times 0.20$ mm |
| Crystal system | Monoclinic |
| Space group | $C2/m$ (No. 12) |
| Measuring device | Stoe IPDS II |
| Radiation | Mo$K\alpha$ (graphite monochromator; $\lambda = 71.07$ pm) |
| Temperature | 170 K |
| $2\theta$ range | $5° < 2\theta < 54°$ |
| Index range | $-24 \leqslant h \geqslant 24$ |
| | $-8 \leqslant k \geqslant 8$ |
| | $-9 \leqslant l \geqslant 10$ |
| $\varphi$-range; $\varphi$-increment | $0° < \omega < 180°$; 2° (2 runs at $\varphi = 0°, 90°$) |
| Number of exposures | 180 |
| Irradiation/exposure | 6 min |
| Detector distance | 80 mm |
| Absorption correction | Numerical, after crystal shape optimization [9,10] |
| Absorption coefficient | 29.12 mm$^{-1}$ |
| $T_{min}/T_{max}$ | 0.0420/0.1464 |
| Measured reflections | 5178 |
| Unique reflections, | 1196 |
| Observed with $I > 2\sigma(I)$ | 1034 |
| $R_{int}/R_\sigma$ | 0.0581/0.0357 |
| Structure determination | SHELXS-86 [27], SHELXL-97 [28] |
| Scattering factors | Intern. Tables. Vol. C [42] |
| Goodness of fit | 1.053 |
| $R_1$; w$R_2(I > 2\sigma(I))$ | 0.0256; 0.0606 |
| $R_1$; w$R_2$ (all data) | 0.0320; 0.0624 |
| Extinction coefficient | 0.00057(6) |
| Depositiory no. | CSD413434 |

are available on quoting the deposition number given in Table 1.

### 2.3. IR spectrometry

The IR spectrum of $[Ag(NH_3)_2]Ag(OsO_3N)_2$ was measured using the KBr technique with the help of the spectrometer IFS66v/s (BRUKER) in the range from 4000 to 400 cm$^{-1}$. The observed bands and their assignment were given in Table 4.

## 3. Results and discussion

### 3.1. Crystal structure

$[Ag(NH_3)_2]Ag(OsO_3N)_2$ crystallizes monoclinically in the monoclinic space group $C2/m$. The silver atoms of

Table 2
Atomic coordinates, Wykoff notation and equivalent isotropic displacement parameters for $[Ag(NH_3)_2]Ag(OsO_3N)_2$

| Atom | Wyckoff | $x/a$ | $y/a$ | $z/a$ | $U_{eq}$ ($10^{-1}/pm^2$) |
|---|---|---|---|---|---|
| Os1 | $4i$ | 0.11117(2) | 0 | 0.00793(5) | 22.1(1) |
| Os2 | $4i$ | 0.29998(2) | 0 | 0.32268(5) | 20.7(1) |
| Ag1 | $2d$ | 1/2 | 0 | 1/2 | 25.8(3) |
| Ag2 | $2c$ | 1/2 | 1/2 | 1/2 | 30.0(3) |
| Ag3 | $4i$ | 0.30408(4) | 0 | 0.83808(10) | 25.9(2) |
| O10 | $4i$ | 0.0490(6) | 0 | 0.8366(10) | 50(3) |
| O11 | $8j$ | 0.8950(3) | 0.7734(9) | 0.8704(7) | 31(1) |
| O21 | $4i$ | 0.3569(4) | 0 | 0.5051(9) | 32(2) |
| O22 | $4i$ | 0.3453(4) | 0 | 0.1494(10) | 29(2) |
| N1 | $4i$ | 0.5049(5) | 0 | 0.7635(11) | 27(2) |
| N2 | $4i$ | 0.1078(5) | 0 | 0.4760(13) | 32(2) |
| N10 | $4i$ | 0.8109(6) | 0 | 0.0597(15) | 41(3) |
| N/O(20)[a] | $8j$ | 0.2482(3) | 0.2191(11) | 0.3191(8) | 43(3) |

*Note*: $U_{eq} = 1/3[U_{22} + 1/\sin^2\beta (U_{11} + U_{33} + 2U_{13}\cos\beta)]$.
[a] Occupation ratio fixed to be 1:1.

Table 3
Selected distances (pm) and Angles (deg) for $[Ag(NH_3)_2]Ag(OsO_3N)_2$

| | | | |
|---|---|---|---|
| Ag1–N1 (2×) | 213.2(9) | Os1–N10 | 168.5(11) |
| Ag1–O21 (2×) | 281.4(8) | Os1–O10 | 174.0(9) |
| | | Os1–O11 (2×) | 175.4(6) |
| | | | |
| Ag2–N2 (2×) | 214.6(10) | | |
| Ag2–O10 (2×) | 279.2(8) | Os2–(N/O)20 (2×) | 171.5(6) |
| | | Os2–O21 | 175.1(8) |
| Ag1–Ag2 (2×) | 316.56(6) | Os2–O22 | 175.3(7) |
| | | | |
| Ag3–(N/O)20 (2×) | 235.4(7) | | |
| Ag3–O11 (2×) | 247.8(6) | | |
| Ag3–N10 | 249.3(10) | | |
| Ag3–O22 | 256.5(8) | | |
| Ag3–O21 | 300.9(7) | | |
| | | | |
| N10–Os1–O10 | 108.5(5) | (N/O)20–Os2–N/O(20) | 108.0(5) |
| N10–Os1–O11 (2×) | 108.0(3) | N10–Os2–O21 (2×) | 109.4(3) |
| O10–Os1–O11 (2×) | 111.3(3) | (N/O)20–Os2–O22 (2×) | 109.9(3) |
| O11–Os1–O11 | 109.7(4) | O21–Os2–O22 | 110.1(4) |

Table 4
IR data ($cm^{-1}$) for $[Ag(NH_3)_2]Ag(OsO_3N)_2$

| $OsO_3N^-$ | | | $[Ag(NH_3)]^+$ | |
|---|---|---|---|---|
| Assignment for $C_{3v}$ symmetry | Frequencies for the "free" ion | Observed frequencies | | |
| $v_4(E)$, $v_{as}(OsO)$ | 871 | 870 vs | $v_s(NH)$ | 3440 vs |
| $v_2(A_1)$, $v_s(OsO)$ | 890 | 892 s | $\delta_{as}(NH_3)$ | 1631 s |
| $v_1(A_1)$, $v(OsN)$ | 1021 | 1026 s | $\delta_s(NH_3)$ | 1196 m |

vs = very strong, s = strong, m = medium.

the complex $[Ag(NH_3)_2]^+$ ions, Ag(1) and Ag(2), are located on special sites ($2c$ and $2d$) of this space group and linearly coordinated by $NH_3$ ligands with the distances Ag–N being 213.2 and 214.6 pm, respectively (Table 3). If the next nearest oxygen atoms (O21, O10), which belong to the $OsO_3N^-$ ions, are taken into account, the coordination spheres of these silver atoms are completed to distorted square planar arrangements. The respective distances Ag–O are, however, quite long (281.4 and 279.2 pm). The third silver atom, Ag(3), is surrounded by six monodentate $OsO_3N^-$ ions with a trigonal prismatic coordination polyhedron. The distances Ag–O and Ag–N cover the range from 235.4 up to 256.5 pm. The shortest distances of 235.4 pm are observed from Ag(3) to the corner of the $OsO_3N^-$ tetrahedron with mixed N/O occupation (see below). The nitridoosmate ions link the Ag(3)$^+$ ions to infinite chains $^1_\infty[Ag(3)(OsO_3N)_{6/3}]$ which extend in the [010] direction (Fig. 1). The chains are further connected to layers by weak Ag–O bonds ($d(Ag(3)–O21) = 300.9$ pm) (Fig. 2), and this increases the coordinatioin number up



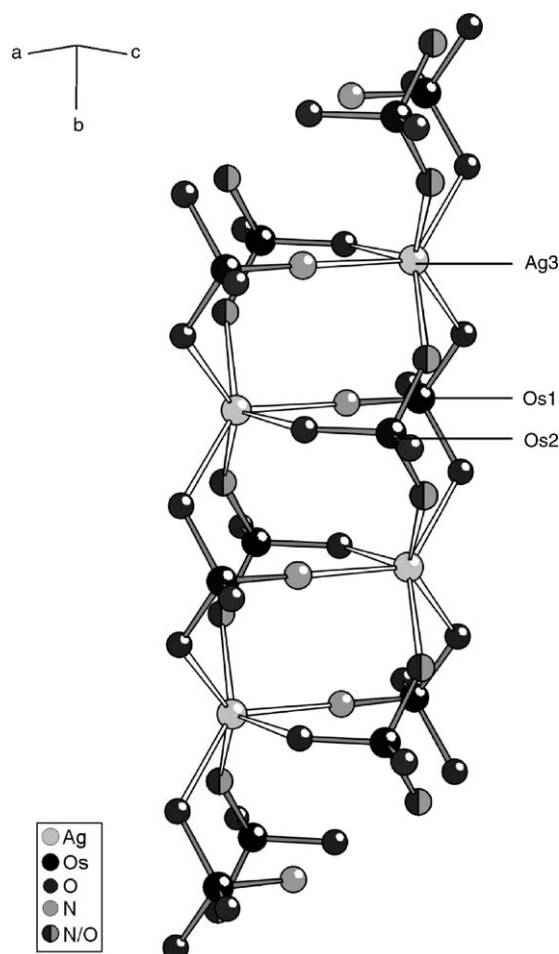Fig. 1. Linkage of the Ag(3)$^+$ ions in the crystal structure of $[Ag(NH_3)_2]Ag(OsO_3N)_2$. Double chains $^1_\infty[Ag(3)(OsO_3N)_{6/3}]$ are formed which are oriented in the [010] direction. The Ag(3)$^+$ ions are in trigonal prismatic coordination of oxygen and nitrogen atoms.

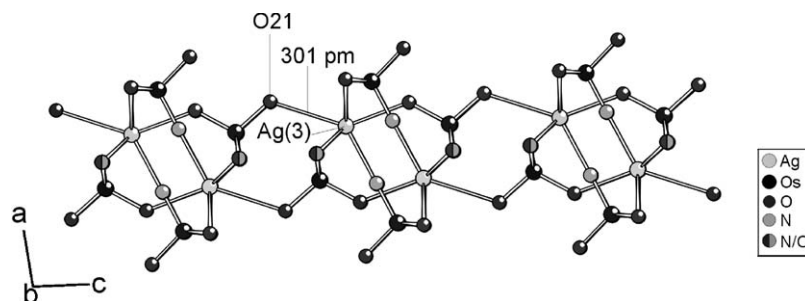Fig. 2. Connection of the chains $^1_\infty[Ag(3)(OsO_3N)_{6/3}]$ to layers via weak Ag–O contacts (301 pm).



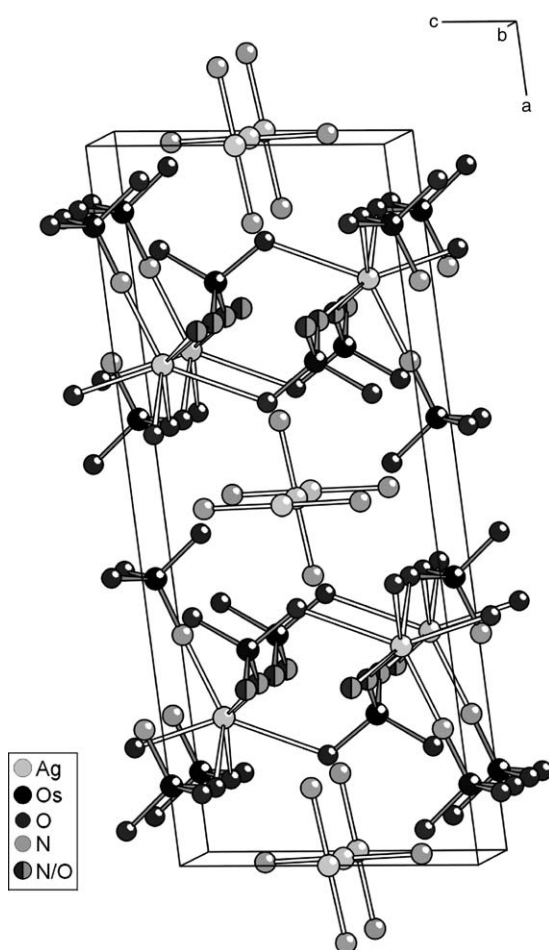Fig. 3. Crystal structure of $[Ag(NH_3)_2)]Ag(OsO_3N)_2$. The layers depicted in Fig. 2 are separated by the complex $[Ag(NH_3)_2)]^+$ ions.
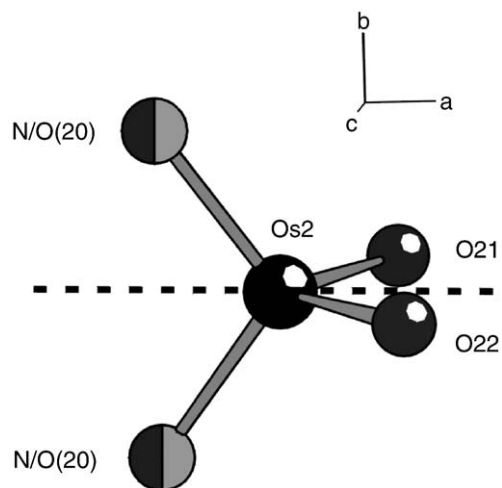


Fig. 4. The $Os(2)O_3N^-$ tetrahedron shows a mixed occupation of two corners by nitrogen and oxygen atoms. These two sites are related crystallographically by a mirror plane (hatched line).

$Os(1)O_3N^-$ is completely ordered and shows the expected values of 168.5 pm for the Os–N bond and around 175 pm for the Os–O bonds (Table 3). In $Os(2)O_3N^-$ two distances are found which can be assigned to Os–O bonds (175.1 and 175.3 pm), while two identical further distances of 171.5 pm are too long compared to the typical Os–N bond, but also shorter than the expected value for an Os–O bond. The distance of 171.5 pm is almost exactly the average value between the typical Os–N and Os–O bond lengths and it can be assumed that the two respective vertices of the tetrahedron are occupied by oxygen and nitrogen atoms in a ratio of 1:1. As mentioned in the introduction such disorder is often observed for nitridoosmates [10,13,14]. Unfortunately, the scattering factors of nitrogen and oxygen atoms are too similar to allow the refinement of individual occupation factors for N20 and O20, respectively. Therefore, the atomic coordinates of this position (8j) have been refined assuming an 1:1 occupation ratio (Table 2). A detailed look on the $Os(2)O_3N^-$ tetrahedron (Fig. 4) shows that the two sites under discussion are related by a mirror plane because
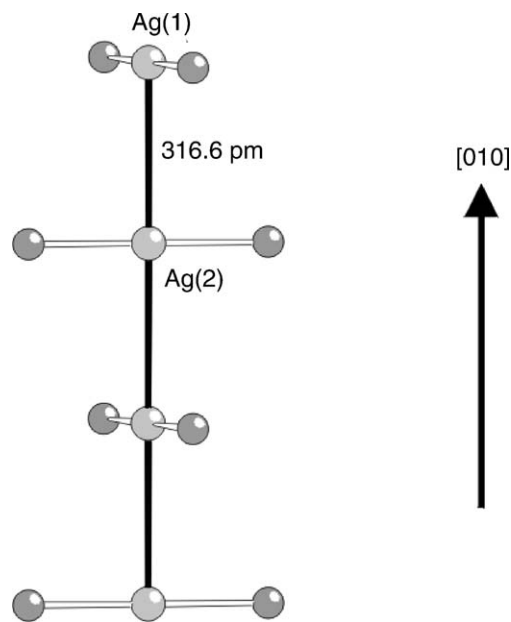
to seven. Coordination numbers between six and eight are frequently found for Ag(I) compounds with complex oxo-anions and $Ag(ClO_4)$ as well as $Ag(NO_3)$ may serve as examples [31,32]. Along the [100] direction these layers are stacked alternatingly with the $[Ag(NH_3)_2]^+$ ions (Fig. 3).

The nitridosmate ions belong to two crystallographically different species, $Os(1)O_3N^-$ and $Os(2)O_3N^-$. With respect to the distances within the tetrahedron,

Fig. 5. The $[Ag(NH_3)_2)]^+$ ions are stacked in the [010] direction leading to short $Ag^+$–$Ag^+$ distances of 316.6 pm.

the Os(2) atom (4*i*) has the site symmetry *m*. Thus, one possibility to avoid the disorder within the tetrahedron is to remove the mirror plane by lowering the space group symmetry up to *C*2 (No. 5). However, attempts to refine a more reliable structure model assuming this non-centrosymmetric space group failed due to strong correlation effects. Furthermore, no second harmonic generation effect was observed when a sample of the compound was irradiated with the fundamental of a Nd:YAG laser (1064 nm) providing severe evidence that the compound is centrosymmetric. A second possibility to overcome the problem is to find a crystallographic superstructure, which allows an ordering of the tetrahedra. Because we were not able to detect any superstructure reflections even on long time exposures, we ruled out that option and we believe that we are in fact dealing with a disorder problem.

Another interesting feature of the crystal structure of $[Ag(NH_3)_2]Ag(OsO_3N)_2$ is related to the arrangement of the linear $[Ag(NH_3)_2]^+$ ions. They are oriented perpendicular to the *b*-axis as can be seen from Fig. 5. In this way short $Ag^+$–$Ag^+$ distances result in the [010] direction which are found to be 316.6 pm. This is longer than the shortest $Ag^+$–$Ag^+$ distances which range from 280 to 300 pm [33–35], but significantly shorter than the van-der-Waals distance of 340 pm [36]. A very similar arrangement of $[Ag(NH_3)_2]^+$ ions is found in the two modifications of $[Ag(NH_3)_2](ClO_4)$ [37]. The latter also exhibits short $Ag^+$–$Ag^+$ distances (around 305 pm). Such distances are frequently observed in silver compounds and were discussed in terms of $d^{10}-d^{10}$ interactions [38,39]. Although the dark color of silver-

rich compounds is sometimes said to be related to these interactions, it would be doubtful to make them accountable for the observed color of $[Ag(NH_3)_2]Ag(OsO_3N)_2$, especially if one takes into account that the above mentioned perchlorate is colorless. It is more reliable to attribute the dark color of the nitridoosmate to interactions of the $OsO_3N^-$ anions with the $Ag^+$ ions. Investigations of the optical properties of $[Ag(NH_3)_2]Ag(OsO_3N)_2$ are currently in progress.

### 3.2. IR spectrum

The undisturbed $OsO_3N^-$ ion has $C3_v$ symmetry and thus can show six normal modes in the vibration spectrum which may be classified according to $\Gamma = 3A_1 + 3E$. The bands are usually observed around 1021, 890, 871, 370, 320, and 309 cm$^{-1}$ [1–6]. The latter three bands do not fall in the region of the measurement. Although the nitridoosmate anions have only $C_s$ symmetry in the present case, no splitting of the bands is observed. Furthermore, also factor group splitting seems to be negligible so that only three bands are observed. They fit very well to the expected values (Table 4). Additionally, the stretching and bending vibrations of the $NH_3$ molecules could be located in the typical regions [40,41]. The stretching vibrations are located around 3300 cm$^{-1}$ and are noticeably obscured by $H_2O$ impurities of the KBr pellet. The deformation modes are found at 1630 and 1196 cm$^{-1}$, respectively.

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