

## VIBRATIONAL SPECTRA OF AMMONIUM AMMINE-OXO-DIPEROXOVANADATE\*

Peter SCHWENDT<sup>a</sup> and Miloslav PISÁRČIK<sup>b</sup>

<sup>a</sup> Department of Inorganic Chemistry, 816 31 Bratislava and

<sup>b</sup> Institute of Inorganic Chemistry,  
Slovak Academy of Sciences, 809 34 Bratislava

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Infrared and Raman spectra of solid  $\text{NH}_4[\text{VO}(\text{O}_2)_2\text{NH}_3]$ ,  $\text{ND}_4[\text{VO}(\text{O}_2)_2\text{ND}_3]$ ,  $^{14/15}\text{NH}_4\cdot[\text{VO}(\text{O}_2)_2^{14/15}\text{NH}_3]$  (about 50%  $^{15}\text{N}$ ) and Raman spectrum of solution of  $\text{NH}_4[\text{VO}(\text{O}_2)_2\text{NH}_3]$  have been measured. Interpretation of the spectra was complemented by normal coordinate analysis in the approximation of point mass model ( $\text{NH}_3$ ). The results have shown that there exists coupling of vibrations of two  $\text{V}(\text{O}_2)$  groups, which enables an explanation of differences between spectra of the mono- and diperoxo complexes. The vibrational coupling of VO and OO bonds within one  $\text{V}(\text{O}_2)$  group probably causes small sensitivity of wave number of  $\nu(\text{O}-\text{O})$  band to changes of  $d(\text{O}-\text{O})$  bond length.

In spite of extensive studies by Griffith and coworkers<sup>2,3</sup> there still remain unsolved problems in interpretation of vibrational spectra of vanadium (V) oxo-peroxo complexes, which considerably limits application of vibrational spectroscopy for studies of structure of peroxo complexes in solid phase and in solution. These problems involve assignment of  $\nu(\text{V}-\text{X})$  bands (X means a donor atom of a further ligand lying in the pentagonal plane), vibrational interaction of two  $\text{V}(\text{O}_2)$  groups in the diperoxo complexes, and the question of the extent to which  $\nu(\text{O}-\text{O})$  and  $\nu(\text{V}-\text{O})$  vibrations are characteristic of  $\text{V}(\text{O}_2)$  group. This paper deals with interpretation of vibrational spectra of a model vanadium(V) diperoxo complex containing a further monodentate ligand— $\text{NH}_4[\text{VO}(\text{O}_2)_2-\text{NH}_3]$ , whose structure is known — with the aim to obtain at least approximate answers to the mentioned questions. Incomplete infrared spectrum of this compound was published by Wieghardt and Quilitsch<sup>4</sup>.

### EXPERIMENTAL

*Preparation of the substances.*  $\text{NH}_4[\text{VO}(\text{O}_2)_2\text{NH}_3]$  was prepared according to Drew and Einstein<sup>5</sup>.  $\text{ND}_4[\text{VO}(\text{O}_2)_2\text{NH}_3]$ : 0.91 g  $\text{V}_2\text{O}_5$  was dissolved in 25 ml 5% solution of  $\text{D}_2\text{O}_2$  in  $\text{D}_2\text{O}$  with constant cooling. After addition of 0.2 g  $\text{ND}_4\text{NO}_3$  the cold solution (0°C) was treated with excess gaseous  $\text{ND}_3$  prepared by reaction of  $\text{ND}_4\text{NO}_3$  with KOD. After standing for 24 h in refrigerator the crystallized complex was separated, washed with ethanol and dried in air.

\* Part IV in the series Vibrational Spectra of Vanadium(V) Compounds; Part III see ref.<sup>1</sup>.

$^{14/15}\text{NH}_4[\text{VO}(\text{O}_2)_2\ ^{14/15}\text{NH}_3]$ : 0.091 g  $\text{V}_2\text{O}_5$  was dissolved in 5 ml 3% solution of  $\text{H}_2\text{O}_2$ . The cold solution ( $0^\circ\text{C}$ ) was treated with gaseous  $^{14/15}\text{NH}_3$  prepared by reaction of 5 g ( $^{14/15}\text{NH}_4$ ) $_2\text{SO}_4$  (50%  $^{15}\text{N}$ , Chemie Berlin) with excess KOH solution. The complex was precipitated with ethanol and dried in air.

Analyses of  $\text{NH}_4[\text{VO}(\text{O}_2)_2\text{NH}_3]$  and  $\text{ND}_4[\text{VO}(\text{O}_2)_2\text{ND}_3]$  corresponded to the given composition. Purity of  $^{14/15}\text{NH}_4[\text{VO}(\text{O}_2)_2\ ^{14/15}\text{NH}_3]$  was only checked by infrared spectrum.

*Measurements of the vibrational spectra.* The infrared spectra were measured in Nujol suspension using a Perkin Elmer 576 and a Spectromaster (Grubb Parsons) apparatus. Polystyrene was used for calibration. The Raman spectra were measured with a JEOL JRS 1 apparatus with He—Ne laser (monochromatic radiation output in sample 12 mW). The Raman spectrum of  $\text{NH}_4[\text{VO}(\text{O}_2)_2\text{NH}_3]$  solution in 15%  $\text{NH}_3$  (to prevent decomposition of the complex in aqueous solution) was measured with  $\text{Ar}^+$  laser (ILA 120, Zeiss Jena, 514 nm, 40 mW).

### Normal Coordinate Analysis

The normal coordinate analysis of  $[\text{VO}(\text{O}_2)_2\text{NH}_3]^-$  anion was carried out in approximation of point mass model ( $\text{NH}_3$ ,  $\text{ND}_3$ ) using internal valence coordinates. Notation of the internal coordinates (Fig. 1): Stretching:  $r(\text{VO}_1)$ ,  $r(\text{VO}_n)$  ( $n = 2, 3, 4, 5$ ),  $r(\text{VN})$ ,  $r(\text{O}_2\text{O}_3)$ ,  $r(\text{O}_4\text{O}_5)$ . Deformation:  $\alpha(\text{O}_1\text{VO}_n)$  ( $n = 2, 3, 4, 5$ ),  $\alpha(\text{O}_1\text{VN})$ ,  $\alpha(\text{O}_3\text{VN})$ ,  $\alpha(\text{O}_4\text{VN})$ ,  $\alpha(\text{O}_2\text{VO}_5)$ . The  $\mathbf{G}$  matrix calculation was carried out with the use of non-modified structure of the anion<sup>5</sup>, as the calculated wave numbers appeared to be considerably dependent on geometry of the ion, especially on the angles  $\text{O}_1\text{—V—O}_n$ .

The following internal valence force constants were used in the calculation:  $a) f(\text{V}=\text{O}_1) = 640 \text{ N/m}$ . This value was determined from empirical dependence between bond length and

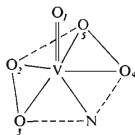


FIG. 1  
Model of  $[\text{VO}(\text{O}_2)_2\text{NH}_3]^-$  anion

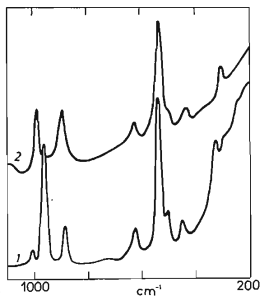


FIG. 2  
Raman spectra of  $\text{NH}_4[\text{VO}(\text{O}_2)_2\text{NH}_3]$ : 1 the solid complex; 2 the complex in 15% ammonia solution

stretching force constants  $f(\text{VO})$  (ref.<sup>6</sup>). *b*) Dependence between the bond length  $d(\text{M}-\text{N})$ , sum of covalent radii  $\sum r_c$ , and the force constant  $f(\text{M}-\text{N})$  of various ammine complexes was studied by Schmidt and Müller<sup>7</sup>. The  $\text{V}-\text{NH}_3$  bond in  $\text{NH}_4[\text{VO}(\text{O}_2)_2\text{NH}_3]$  resembles in its properties (*i.e.*  $d(\text{V}-\text{N}) = 211$  pm,  $\sum r_c = 197$  pm, and  $d(\text{V}-\text{N}) - \sum r_c = 14$  pm) the  $\text{Cr}-\text{NH}_3$  bond in the complex  $[\text{Cr}(\text{NH}_3)_6][\text{CuCl}_5]$  ( $d(\text{Cr}-\text{N}) = 206.4$  pm,  $\sum r_c = 193$  pm,  $d(\text{M}-\text{N}) - \sum r_c = 13.4$  pm). The force constant found for the chromium complex was  $f(\text{Cr}-\text{N}) = 166$  N/m, and therefore we accepted the value 170 N/m for the force constant  $f(\text{V}-\text{N})$ . *c*) The force constant  $f(\text{O}-\text{O})$  can be calculated without difficulties for the isolated ion  $\text{O}_2^-$ . The Raman spectrum of this ion exhibits a band at  $802\text{ cm}^{-1}$  (ref.<sup>8</sup>) corresponding to  $f(\text{O}-\text{O}) = 303$  N/m. As the values of  $d(\text{O}-\text{O})$  in  $\text{NH}_4\text{VO}(\text{O}_2)_2\text{NH}_3$  complex and in  $\text{O}_2^-$  ion differ but slightly (147.2 pm and 149 pm, respectively), the value  $f(\text{O}-\text{O}) = 300$  N/m was used in the calculation. *d*) The known structures<sup>5,9-11</sup> of oxo-peroxo complexes of vanadium(V) indicate that geometry of  $\text{V} \begin{array}{c} \text{O} \\ | \\ \text{O} \end{array}$  group is relatively stable and little affected by the rest of the coordination sphere. With the use of: (*i*) the geometry of the  $\text{V}(\text{O}_2)$  group in  $\text{NH}_4[\text{VO}(\text{O}_2)_2\text{NH}_3]$ , (*ii*) the wave numbers

TABLE I  
Vibrational spectra of  $\text{NH}_4[\text{VO}(\text{O}_2)_2\text{NH}_3]$

IR	Raman		Assignment
	solid complex	complex in solution	
3 340 s			$\nu(\text{NH}_3)$
3 240 sh			
3 190 vs			$\nu_3(\text{NH}_4^+)$
3 132 vs			
1 660 w	1 661 vw		$\nu_2(\text{NH}_4^+)$
1 621 m			$\delta_D(\text{NH}_3)$
1 403 s	1 392 vw		$\nu_4(\text{NH}_4^+)$
1 237 s	1 226 vw		$\delta_S(\text{NH}_3)$
988 s	960 s	1 000 m	$\nu(\text{V}=\text{O})$
877 vs	884 s	957 vs	$\nu(\text{O}-\text{O}) + \nu(\text{V}-\text{O})$
711 m	723 vw	984 s	$\rho_r(\text{NH}_3)$
635 s		888 s	$\nu(\text{V}-\text{O}) + \nu(\text{O}-\text{O})$
623 s	623 m	627 m	$\nu(\text{V}-\text{O})$
531 m	533 vs	537 vs	
500 sh	503 m	496 sh	
449 vw	435 vw	432 m	$\nu(\text{V}-^{14}\text{N})$
442 vw	430 vw		$\nu(\text{V}-^{15}\text{N})$
321 m	322 s	300 s	$\delta(\text{O}=\text{V}-\text{O})$
296 m	287 m		$\delta(\text{O}=\text{V}-\text{N}) + \delta(\text{O}=\text{V}-\text{O})$
230 s	230 w		$\delta(\text{O}-\text{V}-\text{O}) + \delta(\text{O}=\text{V}-\text{O})$

$\tilde{\nu}_1 = 878 \text{ cm}^{-1}$ ,  $\tilde{\nu}_2 = 560 \text{ cm}^{-1}$ , and  $\tilde{\nu}_3 = 560 \text{ cm}^{-1}$  which were calculated as mean values from spectra of five monoperoxo complexes<sup>12-14</sup> and Raman spectrum of  $[\text{VO}(\text{O}_2)(\text{H}_2\text{O})_x]^+$  ion in solution<sup>2</sup>, (iii) the force constant  $f(\text{O}-\text{O}) = 300 \text{ N/m}$ , we determined the force constants  $f_r(\text{V}-\text{O})$ ,  $f_{rr}(\text{VO}-\text{VO})$ ,  $f_{rr}(\text{VO}-\text{OO})$  for a hypothetical isolated  $\text{V}(\text{O}_2)$  group and used them in further calculation.

The bending force constants were chosen to reflect the experimental wave numbers due to bending vibrations. These values represent one solution out of the set of solutions of the inverse spectral problem.

The used set of force constants (the non-zero values only): Stretching force constants (N/m):  $f_r(\text{VO}_1) = 640$ ,  $f_r(\text{VO}_n) = 270$  ( $n = 2, 3, 4, 5$ ),  $f_r(\text{OO}) = 300$ ,  $f_r(\text{VN}) = 170$ . Stretch-stretch interaction force constants (N/m):  $f_{rr}(\text{VO}_2-\text{O}_2\text{O}_3) = f_{rr}(\text{VO}_3-\text{O}_2\text{O}_3) = f_{rr}(\text{VO}_4-\text{O}_4\text{O}_5) = f_{rr}(\text{VO}_5-\text{O}_4\text{O}_5) = 10$ . Bending force constants ( $\text{N} \cdot \text{m}/\text{rad}^2$ ):  $f_\alpha(\text{O}_1\text{VO}_2) = f_\alpha(\text{O}_1\text{VO}_3) = f_\alpha(\text{O}_1\text{VO}_4) = f_\alpha(\text{O}_1\text{VO}_5) = 58$ ,  $f_\alpha(\text{O}_1\text{VN}) = 80$ ,  $f_\alpha(\text{O}_2\text{VO}_5) = 90$ ,  $f_\alpha(\text{O}_3\text{VN}) = f_\alpha(\text{O}_4\text{VN}) = 30$ .

## RESULTS AND DISCUSSION

$\text{NH}_4[\text{VO}(\text{O}_2)_2\text{NH}_3]$  crystallizes in orthorhombic system, space group  $D_{2h}^{16}$ ,  $Z = 4$  (ref.<sup>5</sup>). The anion  $[\text{VO}(\text{O}_2)_2\text{NH}_3]^-$  has an approximately pentagonal bipyramidal

TABLE II  
Vibrational spectra of  $\text{ND}_4[\text{VO}(\text{O}_2)_2\text{ND}_3]$

IR		Raman		Assignment
2 475 s				$\nu(\text{ND}_3)$
2 410 sh				
2 380 s				$\nu_3(\text{ND}_4^+)$
2 330 s				
1 249 w				$\nu_2(\text{ND}_4^+)$
1 194 m				$\delta_D(\text{ND}_3)$
1 074 s				$\nu_4(\text{ND}_4^+)$
987 s	960 s	1 000 m	958 vs	$\nu(\text{V}=\text{O})$
970 s				$\delta_3(\text{ND}_3)$
876 s		882 s		$\nu(\text{O}-\text{O}) + \nu(\text{V}-\text{O})$
635 vs		636 m		$\nu(\text{V}-\text{O}) + \nu(\text{O}-\text{O})$
543 m				$\rho_r(\text{ND}_3)$
531 m		533 vs		$\nu(\text{V}-\text{O})$
500 sh		503 m		
423 vw	410 vw	428 m		$\nu(\text{V}-\text{N})$
325		322 s		$\delta(\text{O}=\text{V}-\text{O})$
294 m		287 m		$\delta(\text{O}=\text{V}-\text{O}) + \delta(\text{O}=\text{V}-\text{N})$
226 s		230 m		$\delta(\text{O}-\text{V}-\text{O}) + \delta(\text{O}=\text{V}-\text{O})$

structure with two bidentate peroxo groups and the nitrogen atom of  $\text{NH}_3$  group in the pentagonal plane (Fig. 1). Site symmetry of the anion in the point mass model approximation is  $C_s$ , and number of normal modes of vibration in the different species of the  $C_s$  group is  $n(A') = 9$  (IR, R) and  $n(A'') = 6$  (IR, R). The correlation between the species of the  $C_s$  group and the species of the  $D_{2h}$  factor group are:  $A' \rightarrow A_g(\text{R}) + B_{2g}(\text{R}) + B_{2u}(\text{IR}) + B_{3u}(\text{IR})$ ,  $A'' \rightarrow B_{1g}(\text{R}) + B_{3g}(\text{R}) + A_u(-) + B_{2u}(\text{IR})$ . The infrared and Raman spectra in the region of stretching vibrations correspond substantially to the site symmetry, only splitting of the band at  $\sim 880 \text{ cm}^{-1}$  is not observed, and one band in the Raman spectrum (at about  $630 \text{ cm}^{-1}$ ) is absent. The correlation splitting is found with the  $\nu(\text{V}-\text{N})$  band in the infrared spectrum ( $A' \rightarrow B_{2u} + B_{3u}$ , Tables I, II) and with the  $\nu(\text{V}=\text{O})$  band.

The infrared spectrum of  $\text{NH}_4[\text{VO}(\text{O}_2)_2\text{NH}_3]$  (Table I) is identical (except for small deviations) with the published spectrum<sup>4</sup> which, however, lacks the  $\nu(\text{V}-\text{N})$  bands and the region of bending vibrations. The spectrum contains all characteristic

TABLE III

Calculated wave numbers of bands in spectrum of  $\text{NH}_4[\text{VO}(\text{O}_2)_2\text{NH}_3]$  and potential energy distribution

$E^a$ $\text{cm}^{-1}$	Calculated wave numbers $\text{cm}^{-1}$	Potential energy distribution <sup>b</sup> %
994, 957	958	93( $\text{VO}_1$ ) <sup>c</sup>
880	899	30(OO), 30(OO)', 19(VO), 19(VO)'
	868	38(OO), 38(OO)', 7(VO), 7(VO)'
635	643 } $\nu_2$	28(VO), 28(VO)', 19(OO), 19(OO)'
623	621 }	38(VO), 38(VO)', 16(VN)
532	533 } $\nu_3$	49(VO), 49(VO)'
502	499 }	40(VO), 40(VO)', 6(O), 6(OO)'
444	450 <sup>d</sup>	73(VN), 11(VO), 11(VO)'
322	322	94( $\text{O}_1\text{VO}$ )
292	292	56( $\text{O}_1\text{VN}$ ), 36( $\text{O}_1\text{VO}$ )
230	234	67( $\text{O}_2\text{VO}_4$ ), 25( $\text{O}_1\text{VO}$ )

<sup>a</sup> Mean value from infrared and Raman spectra; <sup>b</sup> only the contributions greater than 5%; <sup>c</sup> designation of the internal coordinates: ( $\text{VO}_1$ ) =  $r(\text{VO}_1)$ , (VO) =  $r(\text{VO}_2) + r(\text{VO}_3)$ , (VO)' =  $r(\text{VO}_4) + r(\text{VO}_5)$ , (OO) =  $r(\text{O}_2\text{O}_3)$ , (OO)' =  $r(\text{O}_4\text{O}_5)$ , (VN) =  $r(\text{VN})$ , ( $\text{O}_1\text{VN}$ ) =  $\alpha(\text{O}_1\text{VN})$ , ( $\text{O}_1\text{VO}$ ) =  $\alpha(\text{O}_1\text{VO}_2) + \alpha(\text{O}_1\text{VO}_3) + \alpha(\text{O}_1\text{VO}_4) + \alpha(\text{O}_1\text{VO}_5)$ , ( $\text{O}_2\text{VO}_4$ ) =  $\alpha(\text{O}_2\text{VO}_4)$ ; <sup>d</sup> the calculated wave number of the  $\nu(\text{V}-\text{N})$  band for  $\text{ND}_4[\text{VO}(\text{O}_2)_2\text{ND}_3]$  was  $427 \text{ cm}^{-1}$  when using the same **F** matrix.

bands of a coordinated  $\text{NH}_3$ . The intensity of the bands at  $449$  and  $435\text{ cm}^{-1}$  is so low, that these bands were not considered fundamental originally. However, both these bands shift to lower wave numbers not only on deuteration of the complex but also on substitution  $^{14}\text{N} \rightarrow ^{15}\text{N}$ , and, therefore, they can be assigned to  $\nu(\text{V}-\text{N})$ .

The Raman spectra are more suitable for investigation of vibration of the  $\text{VO}(\text{O}_2)_2$  group due to relatively low intensity of bands corresponding to deformation vibrations of the coordinated ammonia (Tables I, II, Fig. 2). In contrast to the infrared spectrum, the  $\nu(\text{V}-\text{N})$  band in the Raman spectrum has a medium intensity. On the basis of analogy of the Raman spectra it is possible to expect a similar structure of  $[\text{VO}(\text{O}_2)_2\text{NH}_3]^-$  ion in solution and in solid phase. Spectrum of  $\text{NH}_4[\text{VO}(\text{O}_2)_2\text{NH}_3]$  in solution exhibits only one band which can be assigned to stretching vibration of terminal  $\text{V}=\text{O}$  group, which supports the presumption that the bands at  $1\,000\text{ cm}^{-1}$  and  $957\text{ cm}^{-1}$  are due to correlation splitting of the  $\nu(\text{V}=\text{O})$  band.

With respect to low symmetry of  $[\text{VO}(\text{O}_2)_2\text{NH}_3]^-$  ion it is impossible to determine the force constants on the basis of the obtained spectral data. Therefore, we chose the so called zero-th order procedure<sup>15</sup>. The force constants were assessed on the basis of those of cognate compounds (groups) and on the basis of presumption of correlation between stretching force constants and bond lengths of the corresponding bonds (see Experimental). The force constants obtained in this way were used for normal coordinate analysis without any further modification.

The calculated wave numbers of spectral bands of  $\text{NH}_4[\text{VO}(\text{O}_2)_2\text{NH}_3]$  and  $\text{ND}_4[\text{VO}(\text{O}_2)_2\text{ND}_3]$  do not substantially differ from the experimental ones (Table III). The potential energy distribution (Table III) agrees with the experimentally found shift of wave numbers of bands connected with the substitutions  $\text{H} \rightarrow \text{D}$  and/or  $^{14}\text{N} \rightarrow ^{15}\text{N}$ , because the shift is observed with the bands corresponding to those vibrations which involve a non-negligible contribution from the internal coordinate  $r(\text{VN})$ .\* Hence, the potential energy distribution can be considered to give a substantially real description of the normal modes of the  $[\text{VO}(\text{O}_2)_2\text{NH}_3]^-$  ion.

The potential energy distribution indicates vibrational coupling of the two  $\text{V}(\text{O}_2)$  groups. With respect to similar structure of  $\text{V}(\text{O}_2)$  groups in mono- and diperoxo complexes it is impossible to expect great differences between stretching force constants of the  $\text{V}(\text{O}_2)$  groups in the two types of complexes. The difference of wave numbers of the  $\nu_2$  and  $\nu_3$  bands (which correspond predominantly to  $\nu(\text{V}-\text{O})$  stretching vibrations; about  $100\text{ cm}^{-1}$ , Table III) can be explained as a result of kine-

\* The point mass model approximation does not allow to explain the shift of the band at  $623\text{ cm}^{-1}$  to higher wave numbers due to deuteration. Shifts to higher wave numbers caused by deuteration of adjacent bonds can sometimes be observed with organic molecules. Thus *e.g.* bands  $\nu(\text{C}-\text{C})$  in  $(\text{CH}_3)_2\text{CO}$  and  $(\text{CD}_3)_2\text{CO}$  or bands  $\nu(\text{C}-\text{O})$  in  $\text{HCOOH}$  and  $\text{DCOOD}$  are shifted in this way<sup>16</sup>.

matic interaction of two  $V(O_2)$  groups which is considerable as it can be seen from numerical values of elements of  $G$  matrix. In contrast to diperoxo complexes, the distance of the  $\nu_2$  and  $\nu_3$  bands of monoperoxo complexes is small (up to  $10\text{ cm}^{-1}$ ) (refs.<sup>1,12,13</sup>). The mentioned difference of wave numbers depends also on coordination number of vanadium atom in the complex<sup>1</sup>.

Coupling of the vibrations  $\nu(V-O)$  and  $\nu(O-O)$  makes itself felt within one  $V(O_2)$  group. This result accounts for a known fact that internuclear distance  $d(O-O)$  cannot be correlated with wave number of the band at about  $880\text{ cm}^{-1}$  (which is usually assigned to  $\nu(O-O)$  in the case of the bound ligand  $O_2$ ).

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