

Fig. 1. Projection along the *a* axis of the atomic arrangement of $(\text{CH}_3)_3\text{SOCdCl}_3$.

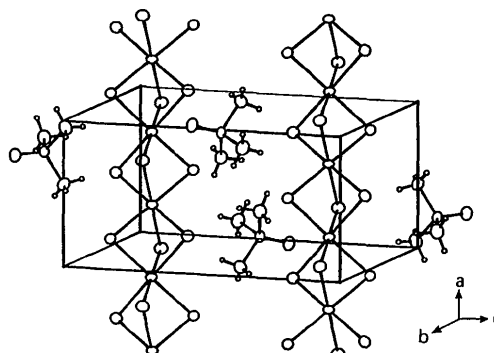


Fig. 2. Atom packing of the unit cell of $(\text{CH}_3)_3\text{SOCdCl}_3$.

in TMCC. The Cd—*X* bond lengths range between 2.731 and 2.803 Å (average 2.767 Å) for the bromide and 2.595 and 2.657 Å (average 2.637 Å) for the chloride (*N.B.* for hexagonal TMCC Cd—Cl = 2.644 Å). These bond lengths are always lower than the ionic radii sums (Shannon & Prewitt, 1969). Cd and *X*(1) atoms are located in the mirror plane (*y* = 0.25 or 0.75), as S, O and C(1) atoms of $(\text{CH}_3)_3\text{SO}^+$. The pyramidal thiocation has only this symmetry element, as found in almost all the other salts studied but it closely approximates to $3m$ symmetry. In the two title compounds, this cation has exactly the same geometrical features.

The projection along the *a* axis of the atomic arrangement of $(\text{CH}_3)_3\text{SOCdCl}_3$ is shown in Fig. 1 and Fig. 2 shows the atom packing of the unit cell.

This work was carried out at the Centre de Diffractométrie de l'Université de Bourgogne.

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Structures of 1,1,1,1,1,2,2,2,2-Decaammine-3,3-diaqua-1,3;2,3-di-(μ -pyrazinato-*N,N'*)-dicobalt(III)silver(I) Nitrate Tetrahydrate and 1,1,1,1,1-Pentaammine-2-(diethylenetriamine-*N,N',N''*)-1,2-(μ -pyrazinato-*N,N'*)-cobalt(III)copper(II) Perchlorate

BY XU BING, CHEN DONG AND TANG WENXIA

Coordination Chemistry Institute, Nanjing University, Nanjing, People's Republic of China

AND YU KAIBEI AND ZHOU ZHONGYUANG

Research Center of Analysis and Measurement, Academia Sinica, Chengdu, People's Republic of China

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Abstract. Complex (I): $[(\text{NH}_3)_5\text{Co}(\mu\text{-C}_4\text{H}_4\text{N}_2)\text{Ag}(\text{H}_2\text{O})_2(\mu\text{-C}_4\text{H}_4\text{N}_2)\text{Co}(\text{NH}_3)_5](\text{NO}_3)_7 \cdot 4\text{H}_2\text{O}$, $M_r = 1098.3$, monoclinic, $C2/c$, $a = 9.431(2)$, $b = 12.374(2)$, $c = 33.716(6)$ Å, $\beta = 92.42(1)^\circ$, $V =$

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3922.6 \AA^3 , $Z = 4$, $D_x = 1.860 \text{ g cm}^{-3}$, $\mu(\text{Mo K}\alpha, \lambda = 0.71070 \text{ \AA}) = 18.40 \text{ cm}^{-1}$, $F(000) = 2248$, $T = 298 \text{ K}$, $R = 0.038$ and $wR = 0.038$ for 2272 significant reflections. The cation has point group symmetry

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C_{2h}. The Ag atom shows square-planar coordination with two water molecules in axial positions and two N atoms in horizontal positions. Two pyrazine groups are connected to two Co atoms and one Ag atom to form a heterotrimeric complex. Each Co atom is coordinated by six N atoms, one from pyrazine and the others from NH₃ groups, forming an octahedral coordination. Complex (II): [(C₄H₁₃N₃)Cu(μ-C₄H₄N₂)Co(NH₃)₅](ClO₄)₅, $M_r = 888.1$, triclinic, $P\bar{1}$, $a = 9.567(4)$, $b = 12.652(5)$, $c = 12.808(4)$ Å, $\alpha = 86.15(3)$, $\beta = 82.70(3)$, $\gamma = 84.58(3)^\circ$, $V = 1528.5$ Å³, $Z = 2$, $D_x = 1.929$ g cm⁻³, $\mu(\text{Mo } K\alpha, \lambda = 0.71070 \text{ \AA}) = 18.40$ cm⁻¹, $F(000) = 902$, $T = 298$ K, $R = 0.059$ and $wR = 0.059$ for 3675 significant reflections. Cu^{II} and Co^{III} are bridged by pyrazine. The Cu atom shows a distorted square coordination by one N atom from pyrazine and three N atoms from diethylenetriamine.

Introduction. A binuclear complex with pyrazine as a bridging ligand and Ru in oxidation state (II) or (III), was first prepared and studied by Creutz & Taube (1969). Since then, a great deal of work has been devoted to this system, in particular to the mixed-valence ion (Lay, Magnuson & Taube, 1988). There are a series of papers on the rates of intramolecular electron transfer in this system. However, the structure of this type of compound has rarely been found so we prepared heterobi- and trinuclear complexes of transition metals with pyrazine as bridging ligands. The present paper reports the structure of a pyrazine-bridged heterobinuclear complex and the first reported structure of a pyrazine-bridged heterotrimeric complex.

Experimental. Complex (I): small orange prismatic crystals from a water solution. Cell dimensions determined on an *R3M/E* four-circle diffractometer by least-squares treatment of the setting angles of 25 reflections in the range $2.5 < \theta < 13^\circ$. Crystal dimensions $0.10 \times 0.20 \times 0.30$ mm; intensities of reflections with indices $h: -10$ to 10 , $k: 0$ to 12 , $l: 0$ to 32 , with $3 \leq 2\theta \leq 45^\circ$ were measured; ω - 2θ scans; scan width $(2.0 + 0.35 \tan \theta)^\circ$, graphite-monochromated Mo $K\alpha$. Three reflections measured every 2 h showed no evidence of crystal decay. No absorption correction. 3011 reflections measured, 2949 unique. 2272 with $I > 3\sigma(I)$ labelled observed and used in structure determination. $R_{\text{int}} = 0.001$. *SHELXTL* version 5.1 (Sheldrick, 1985) for calculations. The Ag atom was located from a Patterson function and the remaining non-H atoms were found *via* the heavy-atom method. After all the coordinates and isotropic and anisotropic thermal parameters for the non-H atoms were refined, the H-atom coordinates were obtained from difference Fourier synthesis and analysis of geometry. Finally, the coordinates, the anisotropic

Table 1. Atomic coordinates ($\times 10^4$) and isotropic thermal parameters ($\times 10^3$) of non-H atoms for complex (I)

$$U_{\text{eq}} = \frac{1}{3}(U_{11} + U_{22} + U_{33}).$$

	x	y	z	U_{eq} (Å ²)
Ag	5000	0	5000	46 (1)
Co	1351 (1)	2719 (1)	6497 (1)	23 (1)
O	6026 (5)	1770 (4)	4720 (1)	63 (2)
O'	2534 (5)	3511 (4)	7548 (2)	64 (2)
O''	8999 (5)	1631 (4)	4659 (2)	66 (2)
O(1)	5848 (5)	4714 (4)	4631 (1)	68 (2)
O(2)	7596 (6)	5720 (5)	4504 (2)	92 (2)
O(3)	7162 (5)	4407 (5)	4128 (2)	86 (2)
O(4)	9479 (6)	4326 (4)	3293 (2)	74 (2)
O(5)	7673 (6)	3754 (6)	2939 (2)	122 (3)
O(6)	9520 (5)	2766 (4)	3018 (2)	71 (2)
O(7)	7844 (4)	1960 (3)	3807 (2)	61 (2)
O(8)	7308 (4)	442 (4)	3530 (2)	59 (2)
O(9)	9399 (4)	688 (4)	3804 (1)	53 (2)
O(10)	0	1578 (4)	7500	50 (2)
O(11)	292 (5)	77 (4)	7193 (1)	53 (2)
N(1)	3836 (5)	1007 (4)	5468 (1)	38 (2)
N(2)	2382 (4)	2064 (3)	6057 (1)	26 (1)
N(3)	2344 (2)	1747 (2)	6873 (1)	31 (1)
N(4)	-147 (3)	1634 (2)	6446 (1)	37 (2)
N(5)	378 (5)	3693 (4)	6118 (1)	33 (2)
N(6)	2816 (5)	3842 (2)	6591 (1)	34 (2)
N(7)	308 (4)	3362 (2)	6926 (1)	34 (2)
N(8)	6867 (5)	4929 (4)	4421 (1)	44 (2)
N(9)	8901 (5)	3648 (4)	3068 (2)	55 (2)
N(10)	8185 (5)	1028 (4)	3709 (1)	39 (2)
N(11)	0	571 (5)	7500	40 (2)
C(1)	2430 (6)	1166 (5)	5438 (2)	42 (2)
C(2)	1725 (6)	1690 (5)	5728 (2)	36 (2)
C(3)	3811 (5)	1932 (4)	6084 (2)	30 (2)
C(4)	4509 (5)	1404 (5)	5789 (2)	34 (2)

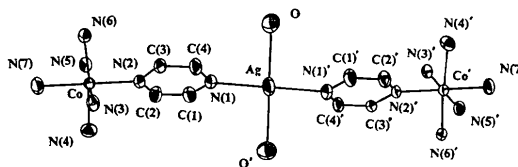


Fig. 1. Perspective view of the cation of complex (I).

thermal parameters for non-H atoms and the isotropic thermal parameters for H atoms were refined by the block-diagonal-matrix least-squares method on F . The final cycle of refinement included 369 variable parameters, $R = 0.038$, $wR = 0.038$, unit weights, goodness of fit = 4.419, maximum shift/e.s.d. = 0.07, density in final difference map ± 1.06 e Å⁻³. Scattering factors and anomalous-dispersion corrections were from *International Tables for X-ray Crystallography* (1974, Vol. IV). Table 1 shows atomic parameters and Table 3* selected interatomic distances and bond angles. Fig. 1 shows the structure of the complex (I) cation.

* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates, and bond lengths and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53926 (48 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Atomic coordinates ($\times 10^4$) and isotropic thermal parameters ($\times 10^3$) of non-H atoms for complex (II)
$$U_{\text{eq}} = \frac{1}{3}(U_{11} + U_{22} + U_{33}).$$

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq} (\AA^2)
Co	9605 (1)	2636 (1)	2371 (1)	23 (1)
Cu	5537 (1)	7333 (1)	2637 (1)	43 (1)
Cl(1)	1612 (2)	1054 (2)	9032 (2)	55 (1)
Cl(2)	7415 (2)	4612 (1)	9680 (1)	43 (1)
Cl(3)	776 (2)	1147 (2)	5826 (2)	51 (1)
Cl(4)	7439 (2)	4406 (2)	5651 (2)	58 (1)
Cl(5)	4345 (2)	1936 (2)	2402 (2)	58 (1)
O(1)	1215 (13)	143 (7)	8776 (7)	146 (5)
O(2)	2437 (8)	1580 (6)	8214 (6)	91 (3)
O(3)	291 (12)	1720 (11)	9144 (10)	198 (7)
O(4)	2010 (13)	1113 (8)	10004 (6)	147 (5)
O(5)	7229 (8)	4815 (6)	8611 (5)	81 (3)
O(6)	8857 (7)	4279 (6)	9749 (5)	86 (3)
O(7)	6571 (7)	3819 (5)	10202 (5)	69 (3)
O(8)	7100 (8)	5573 (5)	10220 (5)	85 (3)
O(9)	1218 (7)	2159 (5)	5983 (6)	77 (3)
O(10)	-460 (10)	955 (7)	6469 (7)	130 (4)
O(11)	560 (8)	1110 (5)	4757 (5)	80 (3)
O(12)	1788 (10)	312 (5)	6070 (7)	111 (4)
O(13)	6660 (8)	3782 (6)	6448 (6)	94 (3)
O(14)	7641 (21)	3901 (9)	4788 (7)	253 (10)
O(15)	6826 (15)	5408 (8)	5485 (11)	199 (7)
O(16)	8724 (13)	4567 (12)	6063 (12)	209 (8)
O(17)	5742 (6)	1640 (7)	2507 (7)	108 (4)
O(18)	3896 (11)	1704 (14)	1515 (9)	211 (8)
O(19)	3452 (7)	1473 (7)	3205 (7)	119 (4)
O(20)	4047 (11)	3023 (8)	2402 (13)	196 (8)
N(1)	10676 (6)	1235 (4)	2347 (5)	36 (2)
N(2)	8300 (6)	2058 (4)	3525 (4)	37 (2)
N(3)	8547 (6)	2164 (4)	1295 (4)	34 (2)
N(4)	10932 (6)	3176 (4)	1216 (4)	36 (2)
N(5)	10715 (6)	3092 (4)	3444 (4)	34 (2)
N(6)	8473 (5)	4034 (4)	2408 (4)	26 (2)
N(7)	6800 (6)	5957 (4)	2552 (4)	38 (2)
N(8)	5003 (7)	7341 (5)	1186 (5)	56 (3)
N(9)	5769 (7)	7527 (5)	4131 (5)	50 (2)
N(10)	4195 (8)	8628 (5)	2764 (6)	66 (3)
C(1)	9050 (7)	4969 (5)	2420 (5)	29 (2)
C(2)	8231 (7)	5921 (5)	2495 (6)	36 (2)
C(3)	6235 (7)	5043 (6)	2532 (6)	36 (2)
C(4)	7057 (7)	4087 (5)	2467 (5)	30 (2)
C(5)	4013 (12)	8266 (7)	986 (7)	77 (4)
C(6)	3490 (15)	8785 (10)	1902 (8)	128 (6)
C(7)	3952 (16)	8895 (9)	3788 (8)	122 (6)
C(8)	4904 (11)	8505 (8)	4474 (7)	73 (4)

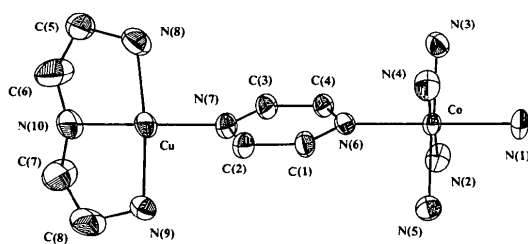


Fig. 2. Perspective view of the cation of complex (II).

Complex (II): purple brown prismatic crystals from a water solution. Data collection and structure determination as for complex (I), unit cell by least squares on 25 reflections, $2.84 < \theta < 9.39^\circ$; crystal dimensions $0.50 \times 0.50 \times 0.40$ mm; intensities of reflections and indices h : 0 to 11, k : -14 to 14, l : -14 to 14, and $3 \leq 2\theta \leq 45^\circ$. 4403 reflections measured, 4291 unique. 3675 with $I > 3\sigma(I)$ labelled observed and used in structure solution and

Table 3. Bond distances (\AA) and bond angles ($^\circ$)

Complex (I)			
Ag—O	2.585 (5)	Ag—N(1)	2.320 (5)
Co—N(2)	1.978 (4)	Co—N(3)	1.957 (2)
Co—N(4)	1.950 (2)	Co—N(5)	1.954 (5)
Co—N(6)	1.974 (3)	Co—N(7)	1.953 (3)
N(1)—C(1)	1.340 (7)	N(1)—C(4)	1.328 (7)
N(2)—C(2)	1.331 (7)	N(2)—C(3)	1.357 (6)
C(1)—C(2)	1.369 (8)	C(3)—C(4)	1.379 (8)
O—Ag—N(1)	89.4 (2)	N(2)—Co—N(3)	89.9 (1)
N(2)—Co—N(4)	91.8 (1)	N(3)—Co—N(4)	87.8 (1)
N(2)—Co—N(5)	89.7 (2)	N(3)—Co—N(5)	179.4 (2)
N(4)—Co—N(5)	92.7 (1)	N(2)—Co—N(6)	92.5 (2)
N(3)—Co—N(6)	90.8 (1)	N(4)—Co—N(6)	175.4 (2)
N(5)—Co—N(6)	88.8 (2)	N(2)—Co—N(7)	179.2 (1)
N(3)—Co—N(7)	90.6 (1)	N(4)—Co—N(7)	87.6 (1)
N(5)—Co—N(7)	89.8 (2)	N(6)—Co—N(7)	88.1 (2)
Ag—N(1)—C(1)	121.5 (4)	Ag—N(1)—C(4)	122.0 (4)
C(1)—N(1)—C(4)	116.4 (5)	Co—N(2)—C(2)	122.7 (4)
Co—N(2)—C(3)	121.1 (3)	C(2)—N(2)—C(3)	116.1 (4)
N(2)—C(2)—C(1)	122.5 (5)	N(2)—C(3)—C(4)	120.8 (5)
N(1)—C(4)—C(3)	122.5 (5)		
Complex (II)			
Co—N(1)	1.962 (5)	Co—N(2)	1.958 (5)
Co—N(3)	1.965 (6)	Co—N(4)	1.955 (5)
Co—N(5)	1.985 (6)	Co—N(6)	1.986 (5)
Cu—N(7)	2.024 (5)	Cu—N(8)	1.988 (7)
Cu—N(9)	1.988 (7)	Cu—N(10)	1.986 (7)
N(6)—C(1)	1.353 (8)	N(6)—C(4)	1.343 (8)
N(7)—C(2)	1.358 (9)	N(7)—C(3)	1.325 (9)
N(8)—C(5)	1.464 (12)	N(9)—C(8)	1.482 (11)
N(10)—C(6)	1.360 (15)	N(10)—C(7)	1.362 (13)
C(1)—C(2)	1.376 (9)	C(3)—C(4)	1.381 (9)
C(5)—C(6)	1.396 (15)	C(7)—C(8)	1.380 (17)
N(1)—Co—N(2)	88.0 (2)	N(1)—Co—N(3)	87.5 (2)
N(2)—Co—N(3)	92.4 (2)	N(1)—Co—N(4)	90.5 (2)
N(2)—Co—N(4)	178.5 (2)	N(3)—Co—N(4)	87.4 (2)
N(1)—Co—N(5)	91.3 (2)	N(2)—Co—N(5)	88.4 (2)
N(3)—Co—N(5)	178.6 (2)	N(4)—Co—N(5)	91.8 (2)
N(1)—Co—N(6)	178.4 (2)	N(2)—Co—N(6)	90.6 (2)
N(3)—Co—N(6)	91.7 (2)	N(4)—Co—N(6)	90.8 (2)
N(5)—Co—N(6)	89.5 (2)	N(7)—Cu—N(8)	95.8 (3)
N(7)—Cu—N(9)	94.4 (2)	N(8)—Cu—N(9)	169.4 (3)
N(7)—Cu—N(10)	176.2 (3)	N(8)—Cu—N(10)	84.5 (3)
N(9)—Cu—N(10)	85.1 (3)	Co—N(6)—C(1)	123.2 (4)
Co—N(6)—C(4)	120.4 (4)	C(1)—N(6)—C(4)	116.4 (5)
Cu—N(7)—C(2)	122.8 (5)	Cu—N(7)—C(3)	119.8 (5)
C(2)—N(7)—C(3)	117.3 (6)	Cu—N(8)—C(5)	110.3 (5)
Cu—N(9)—C(8)	108.7 (5)	Cu—N(10)—C(6)	110.1 (7)
Cu—N(10)—C(7)	109.8 (7)	C(6)—N(10)—C(7)	136.2 (10)
N(7)—C(2)—C(1)	120.9 (6)	N(6)—C(1)—C(2)	121.8 (6)
N(6)—C(4)—C(3)	121.8 (6)	N(7)—C(3)—C(4)	121.8 (6)
N(10)—C(6)—C(5)	119.7 (11)	N(8)—C(5)—C(6)	112.6 (9)
N(9)—C(8)—C(7)	113.6 (8)	N(10)—C(7)—C(8)	119.1 (11)

refinement; $R_{\text{int}} = 0.001$. All the H-atom coordinates were obtained from difference Fourier synthesis and analysis of geometry. Finally the coordinates, the anisotropic thermal parameters for non-H atoms and the isotropic thermal parameters for H atoms were refined by the block-diagonal-matrix least-squares method on F . The final cycle of refinement included 422 variable parameters, $R = 0.059$, $wR = 0.059$, unit weights, goodness of fit = 2.197, maximum shift/e.s.d. = 0.03, density in final difference map $\pm 0.885 \text{ e \AA}^{-3}$. Atom coordinates are given in Table 2, bond distances and angles are in Table 3. Fig. 2 shows the structure of the $[(\text{C}_4\text{H}_{13}\text{N}_3)\text{Cu}(\mu\text{C}_4\text{H}_4\text{N}_2)\text{Co}(\text{NH}_3)_5]^{5+}$ cation.

Discussion. The structure of heterotrimeric complex (I) consists of a $[(\text{NH}_3)_5\text{Co}(\mu\text{-C}_4\text{H}_4\text{N}_2)\text{Ag}(\text{H}_2\text{O})_2(\mu\text{C}_4\text{H}_4\text{N}_2)\text{Co}(\text{NH}_3)_5]^{7+}$ cation, seven NO_3^- anions

and four H₂O molecules. The Ag atom occupies the crystallographic centre of symmetry and also the twofold axis (Fig. 1). One nitrate is located on a twofold axis. In the cation, the Ag atom and the two Co atoms are bridged by two pyrazines. Two O atoms from water molecules also coordinate to the Ag atom. The angles of both N—Ag—N and O—Ag—O are 180.0°. The Ag—O bond distance of 2.585 Å, which is generally considered as a weak bond, is longer than that of Ag—N 2.302 Å, and forms the square-planar coordination. The Co atom is coordinated by six N atoms, five from NH₃ groups and one from pyrazine, forming an octahedral coordination, with the two pyrazine rings being in the same plane. The pyrazine plane is about 86° out of the plane Co—N(3)—N(4)—N(5)—N(6) and 102° out of the plane Ag—N(1)—N(1)—O—O'. The C—C and C—N bond distances of the bridging pyrazines are not significantly different from pyrazine itself (C—N 1.334 Å, C—C 1.378 Å). The distance between the cation and anion is about 3.4–4.0 Å, showing the existence of strong Coulombic forces. Some non-bonded distances for O—O and N—O are less than 3 Å, indicating that there are hydrogen bonds.

The structure of the pyrazine-bridged heterobinuclear complex (II) consists of one [(C₄H₁₃N₃)Cu(μ-C₄H₄N₂)Co(NH₃)₅]⁵⁺ cation and five ClO₄⁻ anions. The pyrazine group bridges the Cu and Co atoms (Fig. 2). The Cu atom shows a distorted square-planar coordination, surrounded by four approximately planar N atoms, N(7) from pyrazine and N(8), N(9) and N(10) from diethylenetriamine. As in Cu,Zn—SOD (superoxide dismutase) (Tainer, Getzoff, Beem, Richardson & Richardson, 1982), the bond length off Cu—N is about 2.0 Å, but there is

no Cu—O bond at the axial position, and the angles of N—Cu—N are different. In Cu,Zn—SOD, N(1)—Cu—N(3) = 160 and N(2)—Cu—N(4) = 130°, and in the cation of complex (II), N(1)—Cu—N(3) = 169.2 and N(2)—Cu—N(4) = 176.8°. The Co atom is coordinated by six N atoms, five from NH₃ groups and one from pyrazine, forming an octahedral coordination. The bond distances and angles around the Co atom are in accord with the corresponding values in the literature (Davis, Dewan & Lippard, 1981).

The pyrazine group shows a good planar conformation. It makes an angle of about 70° with the plane of diethylenetriamine and thus reduces the repulsion force of two planes, forming a stable conformation.

Each ClO₄⁻ anion forms a tetrahedron as usual. The average bond distance of Cl—O is 1.415 Å and O—Cl—O bond angles range from 98.0 to 117.0°. The distance between cations and anions is about 5 Å showing the existence of strong Coulombic forces. Some distances of N...O are less than 3.2 Å, which implies that there are either hydrogen bonds or close contacts.

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Structure of 1,2,3-Tris(dimethylamino)cyclopropenylium Aquatetrachlorooxoniobate(V)

BY H. N. SCHÄFER AND H. BURZLAFF

Institut für Angewandte Physik, Lehrstuhl für Kristallographie, Universität Erlangen–Nürnberg, Bismarckstraße 10, D-8520 Erlangen, Germany

AND A. M. H. GRIMMEISS AND R. WEISS

Institut für Organische Chemie, Lehrstuhl I, Universität Erlangen–Nürnberg, Henkestraße 42, D-8520 Erlangen, Germany

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Abstract. [C₉H₁₈N₃][NbCl₄O(H₂O)], *M*_r = 436.997, triclinic, *P*1, *a* = 12.2716 (9), *b* = 8.9682 (5), *c* = 8.7625 (5) Å, α = 70.203 (9), β = 88.242 (5), γ = 71.299 (5)°, *V* = 856.137 Å³, *Z* = 2, *D*_x = 1.695 g cm⁻³, Mo *K*α, λ = 0.70926 Å, μ = 13.042 cm⁻¹, *F*(000) = 440, room temperature, final

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