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Tris(1-phenyl-3,5-dimethylpyrazole)silver(I) Nitrate

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Abstract. $[Ag(C_{11}H_{12}N_2)_3]NO_3$, $C_{33}H_{36}AgN_7O_3$, trigonal, $R3$, hexagonal axis: $a = 15{\cdot}30\ (2)$, $c = 11{\cdot}87\ (1)$ Å, $\gamma = 120{\cdot}0\ (2)°$, $V = 2406$ Å^3, $Z = 3$, $D_m = 1{\cdot}40$ (flotation), $D_x = 1{\cdot}42$ Mg m^{-3}. The final R was 0·047. The coordination around the Ag^+ corresponds to a trigonal bipyramid with the three N atoms of the ligands in the equatorial plane and NO_3^- groups at the apices.

Introduction. The crystals were prepared by Dr M. Molina and co-workers (Institute of Chemistry of Araraquara, UNESP). They are white and opaque, with prismatic habit. Chemical analysis showed a composition ratio of 1:3 between Ag^+ and the ligands (Molina, Angst, Garcia & Melios, 1972). The structure determination was undertaken in order to study the coordination around Ag^+ and the interaction between Ag^+ and NO_3^-. Cell dimensions were determined by a least-squares fit to settings for 25 reflexions ($\pm hkl$) on a CAD-4 automatic diffractometer. Intensity measurements were carried out up to 30° in θ, with graphite-monochromated Mo $K\alpha$ radiation and a crystal in the form of an elongated block 0·5 × 0·3 × 0·8 mm. Lorentz and polarization corrections were applied but no absorption correction [μ(Mo $K\alpha$) = 0·66 mm^{-1}] was made. 909 unique reflections were collected and after application of the acceptance criterion $F^2 \geq 2\sigma(F^2)$, 898 reflexions were retained for use in the structure analysis.*

The structure was solved by application of direct methods, using the program *MULTAN* (Germain, Main & Woolfson, 1971) and difference Fourier calculations. It was refined by a full-matrix least-squares method by minimization of $\sum w(k|F_o| - |F_c|)^2$ until all the atomic parameter shifts were smaller than a standard deviation. The final unweighted R factor omitting unobserved reflections is 0·047 and including them is 0·049. Anisotropic temperature factors were assigned to all non-hydrogen atoms. Complex neutral-atom scattering factors were employed (*International Tables for X-ray Crystallography*, 1974). Final positional parameters are shown in Table 1.

Discussion. The Ag^+ and the N of the nitrate group lie on the threefold axis.

Table 1. *Fractional atomic coordinates* (×10^4) *with their e.s.d.'s*

	x	y	z
Ag(1)	0	0	0
O(1)	10 (10)	741 (9)	5500 (10)
N(3)	0	0	4906 (62)
N(1)	2281 (3)	1833 (3)	427 (4)
N(2)	1689 (3)	924 (3)	−77 (4)
C(3)	2310 (4)	766 (4)	−731 (5)
C(4)	3301 (4)	1567 (4)	−608 (5)
C(5)	3263 (4)	2233 (4)	128 (5)
C(6)	1839 (4)	2205 (4)	1205 (5)
C(7)	1313 (5)	1628 (6)	2114 (7)
C(8)	858 (6)	1983 (8)	2860 (7)
C(9)	943 (6)	2922 (8)	2664 (9)
C(10)	1480 (7)	3488 (7)	1786 (12)
C(11)	1936 (6)	3151 (5)	1042 (8)
C(12)	1922 (6)	−159 (6)	−1448 (7)
C(13)	4083 (5)	3203 (5)	598 (8)

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33897 (6 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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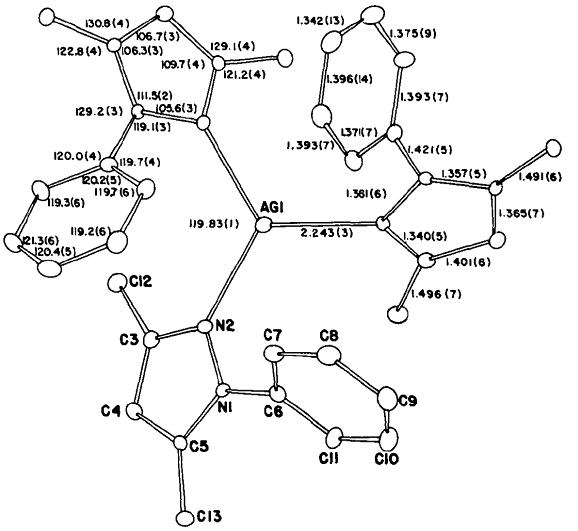

Fig. 1. Diagram of the molecule showing bond lengths (Å) angles (°) and atom numbering.

The Ag^+ ion is coordinated to three ligand molecules, $C_{11}H_{12}N_2$. It is at the center of a triangle formed by the N(2) atoms of the pyrazolic rings (Fig. 1). The nitrate group is at about $c/2$ from the heavy atom (Fig. 2). All relevant distances and angles are shown in Figs. 1 and 2. The structure can be described as non-bonded chains of bipyramids running allong the c direction sharing their axial vertices.

As can be seen from the final values of the thermal parameters of the N and O atoms of NO_3^-, this group exhibits much thermal motion [N(3): $U_{11} = U_{22} = 2U_{12} = 0{\cdot}079$, $U_{33} = 0{\cdot}358$, $U_{23} = U_{13} = 0$ Å^2; O(1): $U_{11} = 0{\cdot}151$, $U_{22} = 0{\cdot}157$, $U_{33} = 0{\cdot}152$, $U_{12} = 0{\cdot}087$, $U_{13} = 0{\cdot}027$, $U_{23} = 0{\cdot}045$ Å^2]. The vibration of the N atom is extremely anisotropic with the greatest amplitude along the c direction. This may be due to the large free space left by the organic ligands. The NO_3^- group is not planar and the distance N(3)–O(1) [1·33(3) Å] is longer than the usual value (Addison, Logan & Wallwork, 1971), but similar to one of the N–O distances in $AgNO_3$: 1·19 (6), 1·32 (6), 1·23 (6) Å (Lindley & Woodward, 1966). The deformations and interactions of the nitrate group cannot be checked by infrared spectroscopy because the NO_3^- characteristic bands are masked by typical bands of the organic ligands. All other bond angles and distances are normal.

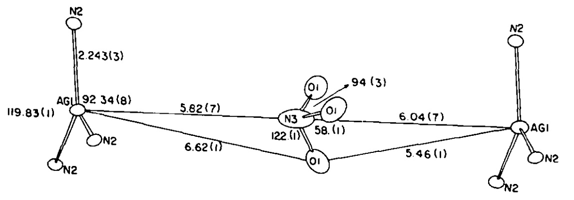

Fig. 2. The relationship of the nitrate group, showing bond distances (Å) and angles (°).

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Isopropylammonium Trichloromanganate(II) Dihydrate

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Abstract. $(CH_3)_2CHNH_3MnCl_3.2H_2O$, $C_3H_{10}Cl_3Mn$-$N.2H_2O$, monoclinic, $P2_1/c$, $a = 14{\cdot}435\ (14)$, $b = 5{\cdot}889\ (2)$, $c = 13{\cdot}281\ (9)$ Å, $\beta = 109{\cdot}60\ (2)$°, $Z = 4$, $D_c = 1{\cdot}607$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å. The structure contains discrete $Mn_2Cl_6(H_2O)_4$ dimers which are hydrogen-bonded together to form a two-dimensional layer in the bc plane. Adjacent layers are separated by the organic cations. Each dimer contains a pair of nearly symmetric Mn–Cl–Mn bridges with a bridging angle of 94·58°. The average Mn–Cl distance is 2·558 Å and the average Mn–O distance is 2·212 Å.

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