1.0040 g ($3.01 \times 10^{-3}$ mole) of potassium antimonyl-*d*-tartrate. The solution was allowed to come to room temperature by standing in air and then chilled in an ice bath. An essentially quantitative precipitation of the cobalt(II) complex results. The precipitate was collected on a sintered-glass funnel. The solid was then added to 25 ml of water which had been previously chilled to 8° and saturated with chlorine gas. Chlorine gas was passed into the agitated suspension for 5 min. The suspension was then allowed to warm to room temperature, 10 ml of water was added, chlorine was passed into the system for 1 min, and the mixture was heated to 60°. The small amount of residue remaining at this time was filtered off. The perchlorate salt of the complex was precipitated by addition of 5 ml of a solution of sodium perchlorate and chilling in an ice bath. The yield of tris(1,10-phenanthroline)cobalt(III) perchlorate dihydrate was 0.4405 g (95.5% of theoretical). The specific rotation, calculated from measurements on a 0.100% solution, was +796° $(\text{g/cc})^{-1}\,\text{dm}^{-1}$.

## Discussion

The data clearly indicate that the resolution of tris(1,10-phenanthroline) complexes of tervalent ions into their optical isomers has been achieved for the first time.

The complexes of Co(III) and Cr(III) both show anomalous rotatory dispersions (Figure 1), which are consistent with the absorption bands at 470 and 426 mμ, respectively.

The preparation of $\text{Cr(phen)}_3{}^{3+}$ from the chromium(II) complex has been found to be very sensitive to the mode of oxidation. Thus, dropwise addition of iodine in ethanol to the chromium(II) complex results in little or none of the tris(1,10-phenanthroline)chromium(III) complex, whereas *rapid* addition of iodine in excess leads to a good yield of this complex. Other oxidizing agents (bromine, chlorine, potassium dichromate, and 30% hydrogen peroxide) behave similarly, but tend to give lower yields. The product of slow oxidation is a pink salt having a phenanthroline: chromium ratio of less than 3:1. Although it has not been fully characterized, it appears to be a bis(1,10-phenanthroline) complex of chromium(III). A probable explanation for these observations is that, during slow oxidation, $\text{Cr(phen)}_2(\text{OH}_2)_2{}^{2+}$ catalyzes the hydrolysis of $\text{Cr(phen)}_3{}^{3+}$ by means of a rapid electron-transfer reaction. A similar mechanism has been proposed for the hydrolysis of the tris(bipyridyl)chromium(III) ion.[14]

The syntheses of *d*-$\text{Cr(phen)}_3{}^{3+}$ and *d*-$\text{Co(phen)}_3{}^{3+}$ have some similarities to Dwyer's preparations of active tervalent complexes.[3,4,7] The racemic complex $\text{Fe(phen)}_3{}^{2+}$, on standing in solution with the antimonyl-*d*-tartrate ion, eventually precipitates completely as the levorotatory isomer.[3] Similarly, the high yields of the active Cr(III) and Co(III) complexes require that the Cr(II) and Co(II) complexes precipitate completely in only one of the diastereoisomeric forms with antimonyl-*d*-tartrate.

The great lability of the Cr(II) and Co(II) species requires oxidation of a suspension, rather than a solution, for obtaining a product of high optical purity. The synthetic procedures reported are ones which, of several modifications attempted, were most successful in this respect.

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[14] B. R. Baker and B. D. Mehta, *Inorg. Chem.*, **4**, 848 (1965).

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# Metal–Olefin Compounds. VIII. The Crystal Structure of a Norbornadiene–Silver Nitrate Complex, $C_7H_8\cdot 2AgNO_3$

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The structure of $C_7H_8\cdot 2AgNO_3$ was determined by single-crystal X-ray diffraction methods. The silver atoms are connected in chains by $\cdots$O—N—O$\cdots$ links of the nitrate groups. One double bond of the olefin forms the third ligand about the silver atom; the axis of the double bond lies approximately in the plane of the O$\cdots$Ag$\cdots$O link of the chain so that the coordination about Ag is triangular-planar. Both double bonds of the olefin are coordinated to Ag atoms, so that the olefin forms a cross-link between two Ag–$NO_3$ chains.

## Introduction

The crystal structures of several complexes of olefins with silver salts have already been reported—cyclooctatetraene–silver nitrate[1] and cyclooctatetraene dimer–silver nitrate.[2] In the preparation of complexes of norbornadiene (NBD) with metal salts, two different complexes with $AgNO_3$ have been reported, $NBD\cdot AgNO_3$[3,4] and $NBD\cdot 2AgNO_3$.[3-5] The complex $NBD\cdot 2AgNO_3$ is most easily isolated, and its structure

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[1] W. N. Lipscomb and F. S. Matthews, *J. Phys. Chem.*, **63**, 845 (1959).

[2] S. C. Nyburg and J. Hilton, *Acta Cryst.*, **12**, 116 (1959).

[3] J. G. Traynham and J. R. Olechowski, *J. Am. Chem. Soc.*, **81**, 571 (1959).

[4] J. G. Traynham, *J. Org. Chem.*, **26**, 4694 (1961).

[5] E. A. Abel, M. A. Bennett, and G. Wilkinson, *J. Chem. Soc.*, 3178 (1959).

is reported below. Structures of complexes of NBD with CuCl and $PdCl_2$ have already been reported.[6,7]

## Experimental Section

Crystals of the norbornadiene–silver nitrate complex were prepared by adding norbornadiene to an aqueous solution of silver nitrate and recrystallizing the product from ethanol.

Single-crystal diffraction patterns were made by the Weissenberg method (*hk*0–*hk*5) with Cu K$\alpha$ radiation and the Buerger precession method [(*h*0*l*–*h*2*l*) and (0*kl*–6*kl*)] with Mo K$\alpha$ radiation. Intensities were estimated visually. Corrections were made for Lorentz and polarization factors, but not for absorption. Cell dimensions *a* and *b* were determined from back-reflection Weissenberg photographs. Using these dimensions for calibration, *c* was determined from precession diagrams. The crystal is orthorhombic with $a = 17.670 \pm 0.003$ A, $b = 6.065 \pm 0.002$ A, $c = 10.135 \pm 0.014$ A, space group $Cmc2_1$. The calculated density is 2.64 based on four $C_7H_8 \cdot 2AgNO_3$ units per cell; the pycnometric measured density is 2.6 g/cm³.

The structure was solved by interpreting the three-dimensional Patterson vector map. The positions of the silver atom, the nitrate group, and some indication of the location of the norbornadiene molecule were obtained from the vector map. Electron density and least-squares refinement methods led to the atom parameters listed in Table I. The scattering factors used for Ag are the values in the "International Tables for X-Ray Crystallography" corrected for dispersion.[8a] The values for $f_N$, $f_O$, $f_C$, and $f_H$ are from the same reference.[8b] A full-matrix, least-squares refinement with individual isotropic temperature factors and independent scale factors for each data group was carried out until $R = 0.12$. At this point the refinement continued with only one over-all scale parameter and anisotropic temperature parameters for the Ag atom, isotropic temperature parameters for the remaining atoms. The function minimized was $\Sigma w(F_o^2 - F_c^2)^2$. The weighting scheme was $\sqrt{w} = C/F^2$ for $F^2$ larger than $C$ and $\sqrt{w} = F^2/C$ for $F^2$ smaller than $C$, where $C = F_{max}^2/16$. Refinement was continued until changes in the parameters were less than one-third of the standard deviations. The final $R_w$ value was 0.074; the unweighted conventional $R = 0.090$. Anisotropic temperature factors were refined only for the silver atom. Since the Weissenberg and precession camera data suffered from absorption to a different extent, the final refinement was based on precession camera data only. The list of calculated and observed structure factors is in Table II.

TABLE I

ATOM PARAMETERS FOR $C_7H_8 \cdot 2AgNO_3$

| | $x$ ($s_x$)[a] | $y$ ($s_y$) | $z$ ($s_z$) | $B$ ($s_B$) |
|---|---|---|---|---|
| $C_1$ | 0.000 | 0.347 (25) | 0.998 (12) | 2.0 (6) |
| $C_2$ | 0.000 | 0.199 (21) | 0.878 (9) | 1.1 (8) |
| $C_3$ | 0.000 | 0.159 (29) | 0.104 (15) | 3.6 (2.5) |
| $C_4$ | 0.070 (2) | 0.014 (7) | 0.064 (3) | 0.8 (5) |
| $C_5$ | 0.064 (2) | 0.074 (9) | 0.932 (3) | 1.8 (8) |
| $O_1$ | 0.143 (2) | 0.470 (8) | 0.272 (3) | 2.6 (5) |
| $O_2$ | 0.248 (2) | 0.403 (7) | 0.159 (3) | 3.2 (8) |
| $O_3$ | 0.249 (2) | 0.546 (8) | 0.354 (3) | 3.2 (9) |
| N | 0.217 (2) | 0.485 (10) | 0.251 (4) | 1.9 (3) |
| $H_1$ | 0.050 | 0.440 | 0.012 | 3.0 |
| $H_2$ | 0.000 | 0.200 | 0.786 | 3.0 |
| $H_3$ | 0.000 | 0.130 | 0.218 | 3.0 |
| $H_4$ | 0.110 | 0.930 | 0.115 | 3.0 |
| $H_5$ | 0.100 | 0.000 | 0.885 | 3.0 |
| Ag | 0.1734 (5) | 0.251 (2) | 0.000 | . . . |

$b_{11} = 0.00195 \pm 5$, $b_{22} = 0.0237 \pm 8$, $b_{33} = 0.0047 \pm 2$

$b_{12} = -0.006 \pm 2$, $b_{13} = 0.000 \pm 1$, $b_{23} = -0.006 \pm 4$

[a] Estimate of standard deviation in last significant digit unless otherwise specified.

TABLE II

CALCULATED AND OBSERVED STRUCTURE FACTORS

| h | k | l | $F_o$ | $F_c$ |
|---|---|---|---|---|
| 0 | 0 | 4 | 266 | 267 |
| 0 | 0 | 6 | 191 | 185 |
| 0 | 0 | 8 | 181 | 176 |
| 0 | 0 | 10 | 96 | 90 |
| 0 | 0 | 12 | 105 | 96 |
| 2 | 0 | 4 | 133 | 141 |
| 2 | 0 | 6 | 146 | 152 |
| 2 | 0 | 8 | 119 | 111 |
| 2 | 0 | 10 | 31 | 40 |
| 2 | 0 | 12 | 58 | 58 |
| 4 | 0 | 2 | 108 | 111 |
| 4 | 0 | 4 | 157 | 152 |
| 4 | 0 | 6 | 27 | 36* |
| 4 | 0 | 8 | 42 | 29 |
| 4 | 0 | 10 | 42 | 49 |
| 4 | 0 | 12 | 30 | 21* |
| 6 | 0 | 0 | 161 | 199 |
| 6 | 0 | 2 | 216 | 259 |
| 6 | 0 | 4 | 258 | 275 |
| 6 | 0 | 6 | 153 | 162 |
| 6 | 0 | 8 | 168 | 161 |
| 6 | 0 | 10 | 133 | 133 |
| 6 | 0 | 12 | 59 | 64 |
| 8 | 0 | 0 | 118 | 106 |
| 8 | 0 | 2 | 221 | 237 |
| 8 | 0 | 4 | 173 | 186 |
| 8 | 0 | 6 | 75 | 82 |
| 8 | 0 | 8 | 70 | 57 |
| 8 | 0 | 10 | 76 | 71 |
| 8 | 0 | 12 | 47 | 56 |
| 10 | 0 | 0 | 81 | 76 |
| 10 | 0 | 2 | 24 | 8* |
| 10 | 0 | 4 | 43 | 3 |
| 10 | 0 | 6 | 37 | 28 |
| 10 | 0 | 8 | 31 | 11* |
| 10 | 0 | 10 | 37 | 10 |
| 12 | 0 | 0 | 194 | 205 |
| 12 | 0 | 2 | 150 | 167 |
| 12 | 0 | 4 | 85 | 88 |
| 12 | 0 | 8 | 83 | 86 |
| 12 | 0 | 10 | 59 | 67 |
| 14 | 0 | 0 | 85 | 89 |
| 14 | 0 | 2 | 129 | 123 |
| 14 | 0 | 4 | 80 | 79 |
| 14 | 0 | 6 | 140 | 140 |
| 14 | 0 | 8 | 78 | 77 |
| 14 | 0 | 10 | 60 | 64 |
| 16 | 0 | 0 | 66 | 59 |
| 16 | 0 | 2 | 40 | 42 |
| 16 | 0 | 4 | 31 | 15* |
| 16 | 0 | 6 | 32 | 26 |
| 16 | 0 | 8 | 31 | 1* |
| 18 | 0 | 0 | 64 | 71 |
| 18 | 0 | 2 | 87 | 80 |
| 18 | 0 | 4 | 69 | 70 |
| 18 | 0 | 6 | 44 | 42 |
| 20 | 0 | 0 | 78 | 64 |
| 20 | 0 | 2 | 85 | 81 |
| 20 | 0 | 4 | 77 | 80 |
| 20 | 0 | 6 | 63 | 44 |
| 22 | 0 | 0 | 31 | 15* |
| 22 | 0 | 2 | 31 | 18* |
| 1 | 1 | 5 | 103 | 94 |
| 1 | 1 | 6 | 29 | 29* |
| 1 | 1 | 7 | 81 | 64 |
| 1 | 1 | 8 | 34 | 32 |
| 1 | 1 | 9 | 71 | 66 |
| 1 | 1 | 10 | 38 | 13* |
| 1 | 1 | 11 | 48 | 46 |
| 1 | 1 | 12 | 39 | 5* |
| 3 | 1 | 3 | 282 | 278 |
| 3 | 1 | 4 | 30 | 35 |
| 3 | 1 | 5 | 236 | 224 |
| 3 | 1 | 6 | 42 | 44 |
| 3 | 1 | 7 | 164 | 167 |
| 3 | 1 | 8 | 34 | 10* |
| 3 | 1 | 9 | 101 | 115 |
| 3 | 1 | 10 | 38 | 20* |
| 3 | 1 | 11 | 88 | 93 |
| 3 | 1 | 12 | 39 | 2* |
| 5 | 1 | 2 | 28 | 2* |
| 5 | 1 | 3 | 130 | [illegible] |
| 5 | 1 | 4 | 24 | 19 |
| 5 | 1 | 5 | 137 | 135 |
| 5 | 1 | 6 | 30 | 19* |
| 5 | 1 | 7 | 124 | 118 |
| 5 | 1 | 8 | 34 | 4* |
| 5 | 1 | 9 | 91 | 94 |
| 5 | 1 | 10 | 38 | 25* |
| 5 | 1 | 11 | 58 | 62 |
| 5 | 1 | 12 | 39 | 10* |
| 7 | 1 | 0 | 56 | 54 |
| 7 | 1 | 1 | 73 | 69 |
| 7 | 1 | 2 | 29 | 43 |
| 7 | 1 | 3 | 56 | 49 |
| 7 | 1 | 4 | 26 | 22* |
| 7 | 1 | 5 | 40 | 40 |
| 7 | 1 | 6 | 31 | 11* |
| 7 | 1 | 7 | 45 | 47 |
| 7 | 1 | 8 | 35 | 22* |
| 7 | 1 | 9 | 42 | 39 |
| 7 | 1 | 10 | 38 | 7* |
| 7 | 1 | 11 | 39 | 22* |
| 7 | 1 | 12 | 39 | 4* |
| 9 | 1 | 0 | 33 | 37 |
| 9 | 1 | 1 | 187 | 188 |
| 9 | 1 | 2 | 24 | 3* |
| 9 | 1 | 3 | 176 | 189 |
| 9 | 1 | 4 | 28 | 15* |
| 9 | 1 | 5 | 152 | 155 |
| 9 | 1 | 6 | 32 | 23* |
| 9 | 1 | 7 | 102 | 112 |
| 9 | 1 | 8 | 36 | 13* |
| 9 | 1 | 9 | 83 | 84 |
| 9 | 1 | 10 | 38 | 17* |
| 9 | 1 | 11 | 63 | 71 |
| 11 | 1 | 0 | 36 | 47 |
| 11 | 1 | 1 | 158 | 167 |
| 11 | 1 | 2 | 27 | 33* |
| 11 | 1 | 3 | 141 | 138 |
| 11 | 1 | 4 | 30 | 27* |
| 11 | 1 | 5 | 120 | 118 |
| 11 | 1 | 6 | 33 | 15* |
| 11 | 1 | 7 | 94 | 102 |
| 11 | 1 | 8 | 36 | 19* |
| 11 | 1 | 9 | 83 | 88 |
| 11 | 1 | 10 | 39 | 9* |
| 11 | 1 | 11 | 52 | 56 |
| 13 | 1 | 0 | 29 | 24* |
| 13 | 1 | 1 | 32 | 14 |
| 13 | 1 | 2 | 30 | 16* |
| 13 | 1 | 3 | 30 | 8* |
| 13 | 1 | 4 | 32 | 28* |
| 13 | 1 | 5 | 33 | 9 |
| 13 | 1 | 6 | 35 | 20* |
| 13 | 1 | 7 | 36 | 2 |
| 13 | 1 | 8 | 37 | 26* |
| 13 | 1 | 9 | 42 | 4 |
| 13 | 1 | 10 | 39 | 8* |
| 15 | 1 | 0 | 31 | 25 |
| 15 | 1 | 1 | 87 | 87 |
| 15 | 1 | 2 | 37 | 26 |
| 15 | 1 | 3 | 91 | 93 |
| 15 | 1 | 4 | 34 | 7* |
| 15 | 1 | 5 | 87 | 90 |
| 15 | 1 | 6 | 36 | 8* |
| 15 | 1 | 7 | 70 | 69 |
| 15 | 1 | 8 | 38 | 13* |
| 15 | 1 | 9 | 53 | 52 |
| 17 | 1 | 0 | 39 | 24 |
| 17 | 1 | 1 | 102 | 112 |
| 17 | 1 | 2 | 34 | 6* |
| 17 | 1 | 3 | 99 | 90 |
| 17 | 1 | 4 | 36 | 9* |
| 17 | 1 | 5 | 82 | 79 |
| 17 | 1 | 6 | 37 | 16* |
| 17 | 1 | 7 | 74 | 71 |
| 17 | 1 | 8 | 39 | 8* |
| 19 | 1 | 0 | 36 | 15* |
| 19 | 1 | 1 | 36 | 16* |
| 19 | 1 | 2 | 36 | 11* |
| 19 | 1 | 3 | 37 | 23* |
| 19 | 1 | 4 | 38 | 19* |
| 19 | 1 | 5 | [illegible] | [illegible] |
| 19 | 1 | 6 | 37 | 3* |
| 21 | 1 | 0 | 38 | 6* |
| 21 | 1 | 1 | 44 | 38 |
| 21 | 1 | 2 | 38 | 6* |
| 21 | 1 | 3 | 50 | 43 |
| 21 | 1 | 4 | 39 | 10* |
| 0 | 2 | 5 | 45 | 27 |
| 0 | 2 | 6 | 210 | 209 |
| 0 | 2 | 7 | 43 | 49 |
| 0 | 2 | 8 | 140 | 140 |
| 0 | 2 | 9 | 40 | 11* |
| 0 | 2 | 10 | 127 | 134 |
| 0 | 2 | 11 | 44 | 21* |
| 0 | 2 | 12 | 61 | 64 |
| 2 | 2 | 5 | 29 | 26 |
| 2 | 2 | 6 | 93 | 91 |
| 2 | 2 | 7 | 33 | 8* |
| 2 | 2 | 8 | 46 | 42 |
| 2 | 2 | 9 | 40 | 17* |
| 2 | 2 | 10 | 82 | 66 |
| 2 | 2 | 11 | 44 | 12* |
| 2 | 2 | 12 | 53 | 42 |
| 4 | 2 | 5 | 30 | 19 |
| 4 | 2 | 6 | 85 | 81 |
| 4 | 2 | 7 | 34 | 16* |
| 4 | 2 | 8 | 50 | 49 |
| 4 | 2 | 9 | 40 | 16* |
| 4 | 2 | 10 | 42 | 22* |
| 4 | 2 | 11 | 44 | 5* |
| 4 | 2 | 12 | 46 | 34 |
| 6 | 2 | 3 | 68 | 73 |
| 6 | 2 | 4 | 140 | 149 |
| 6 | 2 | 5 | 28 | 13* |
| 6 | 2 | 6 | 158 | 155 |
| 6 | 2 | 7 | 39 | 24* |
| 6 | 2 | 8 | 104 | 116 |
| 6 | 2 | 9 | 40 | 7* |
| 6 | 2 | 10 | 82 | 77 |
| 6 | 2 | 11 | 45 | 10* |
| 6 | 2 | 12 | 55 | 69 |
| 8 | 2 | 0 | 150 | 161 |
| 8 | 2 | 1 | 44 | 47 |
| 8 | 2 | 2 | 103 | 107 |
| 8 | 2 | 3 | 25 | 12* |
| 8 | 2 | 4 | 122 | 128 |
| 8 | 2 | 5 | 30 | 15* |
| 8 | 2 | 6 | 140 | 132 |
| 8 | 2 | 7 | 36 | 19* |
| 8 | 2 | 8 | 94 | 101 |
| 8 | 2 | 9 | 41 | 18* |
| 8 | 2 | 10 | 54 | 64 |
| 8 | 2 | 11 | 45 | 11* |
| 8 | 2 | 12 | 53 | 51 |
| 10 | 2 | 0 | 36 | 39 |
| 10 | 2 | 1 | 30 | 35 |
| 10 | 2 | 2 | 27 | 29 |
| 10 | 2 | 3 | 33 | 16 |
| 10 | 2 | 4 | 37 | 32 |
| 10 | 2 | 5 | 33 | 9* |
| 10 | 2 | 6 | 35 | 11 |
| 10 | 2 | 7 | 38 | 31* |
| 10 | 2 | 8 | 40 | 9* |
| 10 | 2 | 9 | 42 | 8* |
| 10 | 2 | 10 | 44 | 15* |
| 10 | 2 | 11 | 45 | 9* |
| 12 | 2 | 0 | 124 | 131 |
| 12 | 2 | 1 | 29 | 23* |
| 12 | 2 | 2 | 116 | 114 |
| 12 | 2 | 3 | 31 | 1* |
| 12 | 2 | 4 | 138 | 134 |
| 12 | 2 | 5 | 35 | 30* |
| 12 | 2 | 6 | 84 | 83 |
| 12 | 2 | 7 | 39 | 23* |
| 12 | 2 | 8 | 88 | 95 |
| 12 | 2 | 9 | 43 | 8* |
| 12 | 2 | 10 | 57 | 67 |
| 14 | 2 | 0 | 127 | 134 |
| 14 | 2 | 1 | 32 | 15* |
| 14 | 2 | 2 | 96 | 102 |
| 14 | 2 | 3 | 34 | 29* |
| 14 | 2 | 4 | 133 | 134 |
| 14 | 2 | 5 | [illegible] | [illegible] |
| 14 | 2 | 6 | 68 | 56 |
| 14 | 2 | 7 | 41 | 0* |
| 14 | 2 | 8 | 56 | 61 |
| 14 | 2 | 9 | 44 | 13* |
| 14 | 2 | 10 | 50 | 45 |
| 16 | 2 | 0 | 35 | 11 |
| 16 | 2 | 1 | 35 | 20* |
| 16 | 2 | 2 | 36 | 3* |
| 16 | 2 | 3 | 37 | 27* |
| 16 | 2 | 4 | 38 | 28 |
| 16 | 2 | 5 | 39 | 5* |
| 16 | 2 | 6 | 41 | 3* |
| 16 | 2 | 7 | 42 | 3* |
| 16 | 2 | 8 | 43 | 30 |
| 16 | 2 | 9 | 45 | 11* |
| 18 | 2 | 0 | 82 | 75 |
| 18 | 2 | 1 | 38 | 6* |
| 18 | 2 | 2 | 68 | 60 |
| 18 | 2 | 3 | 39 | 27* |
| 18 | 2 | 4 | 50 | 46 |
| 18 | 2 | 5 | 41 | 20* |
| 18 | 2 | 6 | 51 | 57 |
| 18 | 2 | 7 | 44 | 11* |
| 20 | 2 | 0 | 57 | 62 |
| 20 | 2 | 1 | 41 | 9* |
| 20 | 2 | 2 | 53 | 60 |
| 20 | 2 | 3 | 42 | 3* |
| 20 | 2 | 4 | 53 | 52 |
| 20 | 2 | 5 | 43 | 6* |
| 20 | 2 | 6 | 59 | 66 |
| 22 | 2 | 0 | 43 | 40 |
| 22 | 2 | 2 | 44 | 33 |
| 22 | 2 | 1 | 43 | 12* |
| 0 | 0 | 6 | 179 | 186 |
| 0 | 0 | 8 | 189 | 176 |
| 0 | 0 | 10 | 100 | 90 |
| 0 | 0 | 12 | 105 | 96 |
| 0 | 2 | 0 | 120 | 113 |
| 0 | 2 | 2 | 321 | 325 |
| 0 | 2 | 4 | 233 | 201 |
| 0 | 2 | 6 | 200 | 209 |
| 0 | 2 | 8 | 147 | 140 |
| 0 | 2 | 10 | 151 | 134 |
| 0 | 4 | 0 | 206 | 194 |
| 0 | 4 | 2 | 139 | 130 |
| 0 | 4 | 4 | 169 | 163 |
| 0 | 4 | 6 | 84 | 84 |
| 0 | 4 | 8 | 91 | 97 |
| 0 | 4 | 10 | 46 | 61 |
| 0 | 6 | 0 | 65 | 67 |
| 0 | 6 | 2 | 84 | 81 |
| 0 | 6 | 4 | 46 | 61 |
| 0 | 6 | 6 | 80 | 77 |
| 1 | 1 | 2 | 78 | 67 |
| 1 | 1 | 3 | 146 | 140 |
| 1 | 1 | 4 | 60 | 54 |
| 1 | 1 | 5 | 96 | 94 |
| 1 | 1 | 6 | 33 | 20* |
| 1 | 1 | 7 | 86 | 84 |
| 1 | 1 | 8 | 37 | 32* |
| 1 | 1 | 9 | 75 | 66 |
| 1 | 1 | 10 | 40 | 11* |
| 1 | 1 | 11 | 50 | 46 |
| 1 | 3 | 0 | 26 | 2* |
| 1 | 3 | 1 | 79 | 80 |
| 1 | 3 | 2 | 54 | 50 |
| 1 | 3 | 3 | 73 | 73 |
| 1 | 3 | 4 | 31 | 8* |
| 1 | 3 | 5 | 85 | 80 |
| 1 | 3 | 6 | 35 | 13* |
| 1 | 3 | 7 | 72 | 69 |
| 1 | 3 | 8 | 38 | 20* |
| 1 | 3 | 9 | 49 | 60 |
| 1 | 3 | 10 | 40 | 5* |
| 1 | 5 | 0 | 34 | 0* |
| 1 | 5 | 1 | 50 | 55 |
| 1 | 5 | 2 | 35 | 13* |
| 1 | 5 | 3 | 46 | 54 |
| 1 | 5 | 4 | 37 | 26* |
| 1 | 5 | 5 | 38 | 34 |
| 1 | 5 | 6 | 39 | 25* |
| 1 | 5 | 7 | [illegible] | [illegible] |
| 1 | 5 | 8 | 40 | 6* |
| 2 | 0 | 4 | 146 | 141 |
| 2 | 0 | 6 | 153 | 152 |
| 2 | 0 | 8 | 105 | 111 |
| 2 | 0 | 10 | 39 | 40* |
| 2 | 2 | 0 | 76 | 75 |
| 2 | 2 | 1 | 53 | 55 |
| 2 | 2 | 2 | 234 | 223 |
| 2 | 2 | 3 | 73 | 70 |
| 2 | 2 | 4 | 143 | 135 |
| 2 | 2 | 5 | 30 | 26 |
| 2 | 2 | 6 | 90 | 91 |
| 2 | 2 | 7 | 34 | 8* |
| 2 | 2 | 8 | 45 | 42 |
| 2 | 2 | 9 | 37 | 14* |
| 2 | 2 | 10 | 62 | 66 |
| 2 | 4 | 0 | 109 | 104 |
| 2 | 4 | 1 | 34 | 45 |
| 2 | 4 | 2 | 73 | 80 |
| 2 | 4 | 3 | 31 | 15* |
| 2 | 4 | 4 | 85 | 102 |
| 2 | 4 | 5 | 34 | 11* |
| 2 | 4 | 6 | 57 | 57 |
| 2 | 4 | 7 | 37 | 9* |
| 2 | 4 | 8 | 62 | 71 |
| 2 | 6 | 0 | 54 | 53 |
| 2 | 6 | 1 | 36 | 25* |
| 2 | 6 | 2 | 45 | 55 |
| 2 | 6 | 3 | 37 | 28* |
| 2 | 6 | 4 | 37 | 28* |
| 2 | 6 | 5 | 38 | 15* |
| 2 | 6 | 6 | 48 | 47 |
| 3 | 1 | 3 | 325 | 278 |
| 3 | 1 | 4 | 34 | 35 |
| 3 | 1 | 5 | 274 | 224 |
| 3 | 1 | 6 | 48 | 44 |
| 3 | 1 | 7 | 188 | 167 |
| 3 | 1 | 8 | 44 | 10* |
| 3 | 1 | 9 | 123 | 115 |
| 3 | 1 | 10 | 49 | 20* |
| 3 | 1 | 11 | 84 | 93 |
| 3 | 3 | 0 | 38 | 24 |
| 3 | 3 | 1 | 254 | 213 |
| 3 | 3 | 2 | 36 | 33 |
| 3 | 3 | 3 | 208 | 194 |
| 3 | 3 | 4 | 51 | 43 |
| 3 | 3 | 5 | 178 | 169 |
| 3 | 3 | 6 | 42 | 16* |
| 3 | 3 | 7 | 122 | 117 |
| 3 | 3 | 8 | 46 | 25* |
| 3 | 3 | 9 | 97 | 100 |
| 3 | 3 | 10 | 50 | 15* |
| 3 | 3 | 11 | 74 | 74 |
| 3 | 5 | 0 | 40 | 32* |
| 3 | 5 | 1 | 111 | 113 |
| 3 | 5 | 2 | 42 | 11* |
| 3 | 5 | 3 | 105 | 110 |
| 3 | 5 | 4 | 43 | 12* |
| 3 | 5 | 5 | 94 | 90 |
| 3 | 5 | 6 | 46 | 12* |
| 3 | 5 | 7 | 86 | 88 |
| 3 | 5 | 8 | 49 | 12* |
| 3 | 5 | 9 | 63 | 63 |
| 3 | 7 | 0 | 49 | 7* |
| 3 | 7 | 1 | 56 | 57 |
| 4 | 0 | 4 | 167 | 152 |
| 4 | 0 | 6 | 29 | 36* |
| 4 | 0 | 8 | 35 | 29* |
| 4 | 0 | 10 | 39 | 49* |
| 4 | 0 | 12 | 42 | 21* |
| 4 | 2 | 2 | 84 | 75 |
| 4 | 2 | 3 | 31 | 42 |
| 4 | 2 | 4 | 34 | 55 |
| 4 | 2 | 5 | 28 | 19 |
| 4 | 2 | 6 | 78 | 81 |
| 4 | 2 | 7 | 34 | 16* |
| 4 | 2 | 8 | 42 | 49 |
| 4 | 2 | 9 | 38 | 16* |
| 4 | 2 | 10 | 40 | 21* |
| 4 | 2 | 11 | 41 | 5* |
| 4 | 4 | 0 | 63 | 72 |
| 4 | 4 | 1 | [illegible] | [illegible] |
| 4 | 4 | 2 | 40 | 58 |
| 4 | 4 | 3 | 36 | 21 |
| 4 | 4 | 4 | 41 | 48 |
| 4 | 4 | 5 | 33 | 8* |
| 4 | 4 | 6 | 35 | 47 |
| 4 | 4 | 7 | 37 | 18* |
| 4 | 4 | 8 | 39 | 28* |
| 4 | 4 | 9 | 40 | 14* |
| 4 | 4 | 10 | 41 | 28* |
| 4 | 6 | 0 | 41 | 24 |
| 4 | 6 | 1 | 36 | 16* |
| 4 | 6 | 2 | 36 | 29* |
| 4 | 6 | 3 | 37 | 17* |
| 4 | 6 | 4 | 38 | 29* |
| 4 | 6 | 5 | 39 | 12* |
| 4 | 6 | 6 | 39 | 15* |
| 4 | 6 | 7 | 41 | 15* |
| 5 | 1 | 4 | 26 | 19 |
| 5 | 1 | 5 | 143 | 135 |
| 5 | 1 | 6 | 34 | 19* |
| 5 | 1 | 7 | 106 | 118 |
| 5 | 1 | 8 | 41 | 4* |
| 5 | 1 | 9 | 95 | 94 |
| 5 | 1 | 10 | 46 | 15* |
| 5 | 1 | 11 | 58 | 62 |
| 5 | 1 | 12 | 50 | 10* |
| 5 | 3 | 0 | 54 | 46 |
| 5 | 3 | 1 | 133 | 139 |
| 5 | 3 | 2 | 28 | 8* |
| 5 | 3 | 3 | 125 | 119 |
| 5 | 3 | 4 | 32 | 19* |
| 5 | 3 | 5 | 91 | 92 |
| 5 | 3 | 6 | 38 | 26* |
| 5 | 3 | 7 | 84 | 76 |
| 5 | 3 | 8 | 43 | 12* |
| 5 | 3 | 9 | 55 | 61 |
| 5 | 3 | 10 | 48 | 6* |
| 5 | 3 | 11 | 54 | 46 |
| 5 | 5 | 0 | 36 | 6* |
| 5 | 5 | 1 | 53 | 60 |
| 5 | 5 | 2 | 37 | 15* |
| 5 | 5 | 3 | 54 | 61 |
| 5 | 5 | 4 | 40 | 7* |
| 5 | 5 | 5 | 52 | 60 |
| 5 | 5 | 6 | 43 | 16* |
| 5 | 5 | 7 | 52 | 52 |
| 5 | 5 | 8 | 47 | 14* |
| 5 | 7 | 0 | 45 | 17* |
| 5 | 7 | 1 | 45 | 34* |
| 6 | 0 | 6 | 183 | 162 |
| 6 | 0 | 8 | 183 | 161 |
| 6 | 0 | 10 | 130 | 133 |
| 6 | 2 | 3 | 84 | 73 |
| 6 | 2 | 4 | 162 | 149 |
| 6 | 2 | 5 | 34 | 13* |
| 6 | 2 | 6 | 184 | 155 |
| 6 | 2 | 7 | 43 | 24* |
| 6 | 2 | 8 | 115 | 116 |
| 6 | 2 | 9 | 50 | 7* |
| 6 | 2 | 10 | 70 | 77 |
| 6 | 2 | 11 | 55 | 10* |
| 6 | 4 | 0 | 156 | 131 |
| 6 | 4 | 1 | 35 | 11* |
| 6 | 4 | 2 | 122 | 114 |
| 6 | 4 | 3 | 38 | 9* |
| 6 | 4 | 4 | 119 | 126 |
| 6 | 4 | 5 | 43 | 30* |
| 6 | 4 | 6 | 111 | 109 |
| 6 | 4 | 7 | 48 | 28* |
| 6 | 4 | 8 | 90 | 86 |
| 6 | 4 | 9 | 55 | 17* |
| 6 | 4 | 10 | 61 | 65 |
| 6 | 6 | 0 | 45 | 52 |
| 6 | 6 | 1 | 46 | 15* |
| 6 | 6 | 2 | 62 | 70 |
| 6 | 6 | 3 | 47 | 25* |
| 6 | 6 | 4 | 61 | 71 |
| 6 | 6 | 5 | 50 | 19* |
| 6 | 6 | 6 | 52 | 69 |
| 6 | 6 | 7 | 53 | 14* |

* Absent reflection.

## Discussion of the Structure

A projection of part of the structure onto (010) is shown in Figure 1. It is obvious that the silver atoms are connected in chains by the ····O—N—O··· links of the nitrate group. A double bond of the olefin forms the third ligand of the silver atom. The axis of the double bond lies approximately in the plane of the O···Ag···O link of the chain, making the principal coordination about the Ag atom triangular-

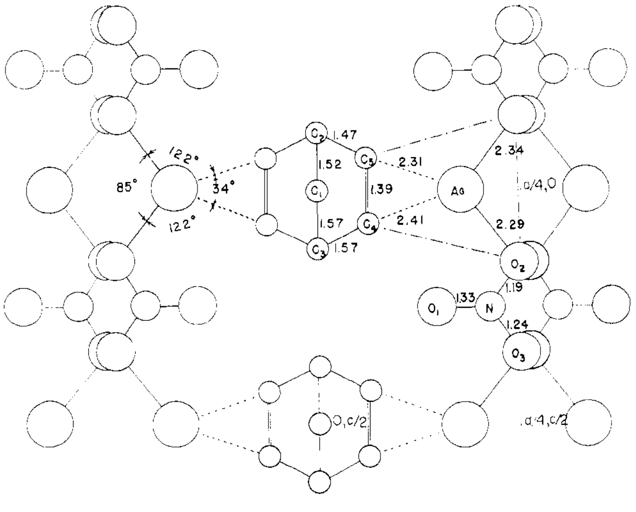

Figure 1.—The structure of $C_7H_8 \cdot 2AgNO_3$ projected onto (010).

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(6) N. C. Baenziger, H. L. Haight, and J. R. Doyle, *Inorg. Chem.*, **3**, 1535 (1964).

(7) N. C. Baenziger, G. F. Richards, and J. R. Doyle, *Acta Cryst.*, **18**, 924 (1965).

(8) "International Tables for X-Ray Crystallography," Vol. III, K. Lonsdale, Ed., The Kynoch Press, Birmingham, England, 1962: (a) p 206; (b) p 202.