

groups form O...Cl hydrogen bonds [O(1)...Cl(2)( $x, \frac{1}{2}-y, \frac{1}{2}+z$ ) 3·148 (5), O(2)...Cl(1)( $-x, \frac{1}{2}+y, \frac{1}{2}-z$ ) 3·160 (4), O(2)...Cl(2)( $x, \frac{3}{2}-y, \frac{1}{2}+z$ ) 3·163 (4) Å]. The Cl(1)<sup>-</sup> ion participates in two coordinate and two hydrogen bonds, whereas the Cl(2)<sup>-</sup> ion acts as an acceptor in as many as five hydrogen bonds.

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## Structures of Ethylenediaminetetraacetato(3-) Metal Complexes. I. Complexes with Co, Mg and Cd Metals

BY X. SOLANS AND M. FONT-BARDÍA

*Dpto Cristalografía y Mineralogía, Universidad de Barcelona, Grand Via 585, 08007-Barcelona, Spain*

M. AGUILÓ

*Cristalografía, Fac. Químicas, Universidad de Barcelona, Pza Imperial Tarraco 1, Tarragona, Spain*

AND M. AROSTEGUI AND J. OLIVA

*Dpto Farmacia, Universidad de Barcelona, Diagonal 645, 08028-Barcelona, Spain*

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**Abstract.** (I): Hexaaquacobalt(II) aqua[ethylenediaminetetraacetato(3-)]cobaltate(II) dihydrate, [Co(H<sub>2</sub>O)<sub>6</sub>][Co(C<sub>10</sub>H<sub>13</sub>N<sub>2</sub>O<sub>8</sub>)(H<sub>2</sub>O)]<sub>2</sub>·2H<sub>2</sub>O,  $M_r = 935.4$ , monoclinic,  $C2/c$ ,  $a = 13.842$  (3),  $b = 9.402$  (2),  $c = 27.936$  (4) Å,  $\beta = 90.80$  (2)°,  $V = 3635$  (2) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.709$  Mg m<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71069$  Å,  $\mu = 1.512$  mm<sup>-1</sup>,  $F(000) = 1932$ ,  $T = 288$  K. (II): Tetra-

aquabis{aqua[ethylenediaminetetraacetato(3-)]cadmium(II)-O,O'}cadmium(II) tetrahydrate, [Cd{Cd-(C<sub>10</sub>H<sub>13</sub>N<sub>2</sub>O<sub>8</sub>)(H<sub>2</sub>O)}<sub>2</sub>(H<sub>2</sub>O)<sub>4</sub>}.4H<sub>2</sub>O,  $M_r = 1095.8$ , monoclinic,  $P2_1/c$ ,  $a = 12.054$  (2),  $b = 16.457$  (2),  $c = 9.118$  (1) Å,  $\beta = 91.11$  (2)°,  $V = 1808.4$  (7) Å<sup>3</sup>,  $Z = 2$ ,  $D_x = 2.012$  Mg m<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71069$  Å,  $\mu = 1.843$  mm<sup>-1</sup>,  $F(000) = 1092$ ,  $T = 288$  K.  $R = 0.054$

(I) and 0.022 (II) for 2431 and 2901 observed reflections, respectively. From powder-diffraction patterns it is observed that the Mg complex is isostructural to the Co complex. Both structures consist of discrete  $[M(H_2O)_6]^{2+}$  and  $[M(Hedta)(H_2O)]^-$  ions, while the Cd complex consists of trimeric molecules, where the  $[Cd(H_2O)_4]^{2+}$  moiety acts as a bridge between two  $[Cd(Hedta)(H_2O)]^-$  units. The  $Co^{2+}$  ions and the  $Cd^{2+}$  ion of the  $[Cd(H_2O)_4]^{2+}$  unit display a distorted octahedral coordination, while the Cd linked to the Hedta ligand displays a distorted pentagonal-bipyramidal coordination, being linked to four O and two N of the Hedta ligand and a water molecule. The Co linked to Hedta is coordinated to three O and two N of the Hedta and a water molecule.

**Introduction.** In preceding papers the crystal structure of ethylenediaminetetraacetato(4-) metal complexes has been studied (Solans, Font-Altaba & Garcia-Oricain, 1985). A new systematic study has begun on complexes synthesized in more acid conditions. In order to determine the geometrical variations, crystal-structure determination of the title compounds has been carried out.

**Experimental.** Red prismatic crystals of (I) ( $0.1 \times 0.1 \times 0.15$  mm); colourless tabular crystals of (II) ( $0.1 \times 0.1 \times 0.008$  mm). Philips PW 1100 diffractometer. Unit-cell parameters from 25 reflections ( $4 \leq \theta \leq 12^\circ$ ) and refined by least squares, graphite monochromator, Mo  $K\alpha$  radiation,  $\omega$ -scan technique, scan width  $1^\circ$  in (I) and  $0.8^\circ$  in (II), scan speed  $0.03^\circ s^{-1}$ . Three reflections every 2 h, significant intensity decay was not observed. In (I) 2554 reflections with  $\theta \leq 25^\circ$ ,  $hkl$  range:  $-16$  to  $16$ ;  $0$  to  $11$ ;  $0$  to  $32$ , respectively, 2431 with  $I \geq 2.5\sigma(I)$ ,  $R_{int} = 0.026$ . In (II) 3124 reflections with  $\theta \leq 25^\circ$ ,  $hkl$  range:  $-14$  to  $14$ ;  $0$  to  $19$ ;  $0$  to  $10$ , respectively, 2901 with  $I \geq 2.5\sigma(I)$ ,  $R_{int} = 0.013$ . Lp corrections, empirical absorption correction in (II), max. and min. transmission factor 0.86 and 0.81, respectively.

Co atoms in (I) from Patterson synthesis. Cd atoms and coordination polyhedra of metals in (II) from direct-methods program *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). The remaining non-H atoms in both structures from a weighted Fourier synthesis. Both structures refined by full-matrix least-squares methods (*SHELX76*, Sheldrick, 1976),  $\sum w(|F_o| - |F_c|)^2$  minimized,  $w = [\sigma^2(F_o) + k|F_o|^2]^{-1}$ ,  $k = 0.0066$  in (I) and 0.0 in (II),  $f$ ,  $f'$  and  $f''$  from *International Tables for X-ray Crystallography* (1974). Non-H atoms refined anisotropically. 20 H of (I) from  $\Delta\rho$  map, refined with an overall isotropic temperature factor. H atoms not located in (II). Final  $R$  0.054 in (I) and 0.022 in (II),  $wR$  = 0.058 and 0.027, respectively, for all observed reflections. Max. shift/e.s.d. =  $-0.34$  for  $z$  of

H(12) in (I) and  $-0.22$  for  $y$  of C(42) in (II). Max. and min. peaks in final  $\Delta\rho$  map  $0.4 e \text{ \AA}^{-3}$   $0.75 \text{ \AA}$  from C(22) and  $-0.4 e \text{ \AA}^{-3}$ , respectively, for (I) and  $0.5 e \text{ \AA}^{-3}$   $0.71 \text{ \AA}$  from O(W1) and  $-0.4 e \text{ \AA}^{-3}$ , respectively, for (II). IBM-3083 computer.

**Discussion.** The atomic coordinates and equivalent isotropic thermal coefficients are listed in Table 1, selected bond lengths and angles of the two compounds in Table 2.\* Fig. 1 shows a view of (II). From powder-diffraction patterns the Mg complex has been determined to be isostructural to the Co complex; its cell parameters are  $a = 13.825$  (4),  $b = 9.394$  (3),  $c = 27.918$  (9)  $\text{\AA}$ ,  $\beta = 90.82$  (4) $^\circ$ .

The structures of the title compounds differ from that of the non-protonated-edta Co complex (Candlish, Michael, Neal, Lingafelter & Rose, 1978), which consists of chains of alternate  $[Co(H_2O)_4]^{2+}$  and  $[Co(edta)]^{2-}$  ions bridged by edta ligands. The structure of the Co complex consists of discrete ions linked by van der Waals forces and hydrogen bonds. The Cd complex consists of trimeric molecules where the  $[Cd(H_2O)_4]^{2+}$  ion acts as a bridge between two  $[Cd(Hedta)(H_2O)]^-$  moieties, while the  $[Cd(edta)Mn(H_2O)_4] \cdot 2H_2O$  crystal structure (Solans, Font-Altaba, Oliva & Herrera, 1985) consists of chains of alternate  $[Cd(edta)]_2^{2-}$  moieties and  $[Mn(H_2O)_4]^{2+}$  pairs.

The Co(1) ion is linked to two N and three O atoms of the Hedta ligand and water molecule, displaying a distorted octahedral coordination. The two N atoms, acetato O and the water molecule are in the equatorial plane. The apical Co—O(acetato) bond lengths [average value  $2.075$  (7)  $\text{\AA}$ ] are shorter than the equatorial Co—O(acetato) length [ $2.114$  (4)  $\text{\AA}$ ], which was also observed in the Mn—Co complex of edta (Solans, Font-Altaba, Oliva & Herrera, 1983). The Co—N bond length alters according to the electronegative character of the *trans* ligand. The small N—Co—N [ $83.1$  (2) $^\circ$ ] and N(1)—Co(1)—O(11) [ $78.3$  (2) $^\circ$ ] bond angles produce larger O(W1)—Co(1)—O(11) [ $99.2$  (2) $^\circ$ ] and O(W1)—Co(1)—N(2) [ $101.1$  (2) $^\circ$ ] bond angles.

The protonation of O(41) produces a C(41)—O(41) [ $1.317$  (8)  $\text{\AA}$ ] bond length larger than that observed for C(41)—O(42) [ $1.194$  (7)  $\text{\AA}$ ], while the remaining C—O bond lengths show intermediate values [average value  $1.26$  (1)  $\text{\AA}$ ].

Co(2) is linked to six O atoms of hydrate water molecules with Co—OW lengths in the range  $1.970$  (9)— $2.123$  (5)  $\text{\AA}$ . The variation of Co—OW bond lengths can be assigned to different roles of the aqueous molecules, the longest Co—OW being that with the

\* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and all bond distances and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43560 (31 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

greatest number of O(acetato) hydrogen-bonded to the water molecule.

In structure (II), the Cd(1) ion is coordinated to two N and four O of the Hedta ligand and a water molecule, displaying a distorted pentagonal-bipyramidal coordination, with O(11) and O(31) in the apical sites. It has not been possible to determine the H-atom positions from a difference synthesis, but the protonated O(acetato) moiety must be assigned to O(22) according to C—O(acetato) bond lengths and hydrogen bonds. As in the Co complex, the C(21)—O(21) length is 1.178 (5) Å, while that of C(21)—O(22) is 1.304 (4) Å and the average value for the remaining C—O lengths is 1.26 (2) Å.

Table 1. Final atomic coordinates ( $\times 10^4$ , for Co and Cd  $\times 10^5$ ) and equivalent isotropic thermal coefficients for structures (I) and (II)

$$B_{eq} = \frac{1}{3} \pi^2 \sum_i \sum_j U_{ij} a_i^* a_j^* a_i a_j$$

Structure (I)	x	y	z	$B_{eq}(\text{Å}^2)$
Co(1)	32386 (4)	37892 (6)	38794 (2)	1.51 (3)
N(1)	4208 (3)	5555 (4)	3805 (1)	1.7 (2)
C(1)	3961 (4)	6573 (6)	4188 (2)	2.5 (2)
C(2)	2876 (4)	6689 (5)	4232 (2)	2.2 (2)
N(2)	2462 (3)	5273 (4)	4355 (2)	1.7 (2)
O(11)	4443 (2)	2812 (4)	3559 (1)	2.2 (1)
C(11)	5224 (4)	3508 (5)	3615 (2)	2.0 (2)
C(12)	5179 (4)	4918 (6)	3866 (2)	2.2 (2)
O(12)	6031 (2)	3054 (4)	3480 (1)	2.6 (2)
O(21)	2701 (3)	4610 (4)	3234 (1)	2.3 (1)
C(21)	3205 (4)	5572 (6)	3054 (2)	2.1 (2)
C(22)	4083 (5)	6139 (6)	3324 (3)	2.6 (2)
O(22)	3024 (3)	6123 (4)	2653 (2)	3.1 (2)
O(31)	3721 (2)	3108 (4)	4542 (1)	2.1 (1)
C(31)	3372 (3)	3653 (5)	4919 (2)	1.5 (2)
C(32)	2677 (4)	4879 (6)	4862 (2)	2.1 (2)
O(32)	3579 (3)	3243 (4)	5329 (1)	2.4 (2)
O(41)	-145 (3)	5892 (5)	4441 (2)	2.9 (2)
C(41)	775 (4)	6199 (5)	4512 (2)	2.1 (2)
C(42)	1416 (3)	5185 (5)	4238 (2)	1.9 (2)
O(42)	1057 (3)	7202 (5)	4735 (2)	3.6 (2)
OW(1)	2279 (3)	2124 (4)	3840 (2)	2.6 (2)
OW(2)	-2540 (3)	5144 (5)	-1776 (2)	3.2 (2)
Co(2)	0	49481 (10)	125506 (26)	1.4 (1)
OW(3)	-1006 (3)	6526 (5)	2327 (2)	3.1 (2)
OW(4)	1030 (3)	3439 (5)	2732 (2)	2.9 (2)
OW(5)	-534 (3)	5029 (4)	3200 (2)	2.5 (2)

Structure (II)	x	y	z	$B_{eq}(\text{Å}^2)$
Cd(2)	0	0	50000	3.73 (2)
Cd(1)	22012 (2)	22202 (1)	16796 (3)	3.11 (2)
OW(1)	2028 (2)	2117 (1)	4127 (4)	3.3 (1)
N(1)	957 (3)	2513 (2)	-369 (4)	2.7 (1)
C(1)	1353 (2)	2109 (2)	-1715 (5)	3.3 (2)
C(2)	2626 (4)	2200 (2)	-1757 (4)	3.8 (2)
N(2)	3193 (2)	1882 (2)	-470 (4)	3.5 (1)
O(11)	892 (2)	1181 (1)	1411 (3)	3.7 (1)
C(11)	-56 (3)	1381 (2)	828 (4)	3.0 (1)
O(12)	-924 (2)	993 (2)	1011 (4)	5.3 (2)
C(12)	-159 (3)	2157 (2)	-4 (5)	3.7 (2)
C(22)	886 (3)	3425 (2)	-464 (5)	3.4 (2)
C(21)	962 (3)	3780 (2)	1140 (5)	3.2 (2)
O(21)	1110 (2)	3363 (1)	2176 (3)	3.7 (1)
O(22)	753 (2)	4557 (1)	1171 (4)	4.8 (1)
C(32)	4340 (3)	2261 (2)	-330 (5)	2.7 (1)
C(31)	4266 (3)	3122 (2)	321 (4)	3.6 (2)
O(31)	3507 (2)	3279 (1)	1239 (4)	4.6 (1)
O(32)	5013 (2)	3593 (2)	-53 (4)	5.3 (1)
O(41)	3454 (2)	1161 (1)	2201 (3)	3.6 (1)
O(42)	3930 (2)	-32 (1)	1271 (4)	4.7 (1)
C(41)	3582 (3)	706 (2)	1118 (6)	3.9 (2)
C(42)	3348 (4)	989 (2)	-413 (6)	5.4 (2)
OW(2)	3627 (2)	4989 (2)	1681 (4)	5.6 (2)
OW(3)	6348 (3)	4966 (2)	1838 (5)	6.0 (2)
OW(4)	3212 (2)	725 (2)	5729 (4)	4.8 (1)
OW(5)	1931 (3)	3588 (2)	5469 (4)	5.7 (2)

Table 2. Selected bond lengths (Å) and angles (°)

Structure (I)		Structure (II)	
N(1)—Co(1)	2.146 (5)	OW(1)—Cd(1)	2.252 (3)
N(2)—Co(1)	2.215 (5)	O(11)—Cd(1)	2.337 (2)
O(11)—Co(1)	2.114 (4)	N(1)—Cd(1)	2.421 (3)
O(21)—Co(1)	2.088 (4)	O(21)—Cd(1)	2.345 (2)
O(31)—Co(1)	2.062 (4)	N(2)—Cd(1)	2.381 (3)
OW(1)—Co(1)	2.055 (4)	O(31)—Cd(1)	2.387 (2)
		O(41)—Cd(1)	2.349 (3)
OW(3)—Co(2)	2.123 (5)	O(32)—Cd(2)	2.316 (3)
OW(4)—Co(2)	2.070 (5)	OW(2)—Cd(2)	2.278 (2)
OW(5)—Co(2)	1.970 (9)	OW(3)—Cd(2)	2.312 (3)
N(2)—Co(1)—N(1)	83.1 (2)	N(2)—Cd(1)—N(1)	74.1 (1)
O(11)—Co(1)—N(1)	78.3 (2)	N(1)—Cd(1)—O(11)	69.9 (1)
O(11)—Co(1)—N(2)	156.9 (2)	N(2)—Cd(1)—O(11)	95.3 (1)
O(21)—Co(1)—N(1)	81.2 (2)	O(21)—Cd(1)—N(1)	69.4 (1)
O(21)—Co(1)—N(2)	96.6 (2)	N(2)—Cd(1)—O(21)	129.7 (1)
O(21)—Co(1)—O(11)	93.9 (2)	O(21)—Cd(1)—O(11)	103.1 (1)
O(31)—Co(1)—N(1)	97.6 (2)	O(31)—Cd(1)—N(1)	97.2 (1)
O(31)—Co(1)—N(2)	79.1 (2)	O(31)—Cd(1)—N(2)	71.9 (1)
O(31)—Co(1)—O(11)	89.9 (2)	O(31)—Cd(1)—O(11)	164.3 (1)
O(31)—Co(1)—O(21)	175.6 (2)	O(31)—Cd(1)—O(21)	79.7 (1)
OW(1)—Co(1)—N(1)	171.3 (2)	N(1)—Cd(1)—OW(1)	135.3 (1)
OW(1)—Co(1)—N(2)	101.1 (2)	N(2)—Cd(1)—OW(1)	148.8 (1)
OW(1)—Co(1)—O(11)	99.2 (2)	O(11)—Cd(1)—OW(1)	88.5 (1)
OW(1)—Co(1)—O(21)	90.8 (2)	O(21)—Cd(1)—OW(1)	78.8 (1)
OW(1)—Co(1)—O(31)	90.7 (2)	O(31)—Cd(1)—OW(1)	107.2 (1)
		O(41)—Cd(1)—OW(1)	79.4 (1)
		O(41)—Cd(1)—O(11)	84.8 (1)
		O(41)—Cd(1)—N(1)	133.6 (1)
		O(41)—Cd(1)—O(21)	156.5 (1)
		O(41)—Cd(1)—N(2)	70.2 (1)
		O(41)—Cd(1)—O(31)	98.8 (1)
		O(32)—Cd(2)—OW(2)	90.7 (1)
		O(32)—Cd(2)—OW(3)	89.2 (1)
		OW(2)—Cd(2)—OW(3)	91.2 (1)
OW(4)—Co(2)—OW(3)	176.5 (4)	OW(1)...OW(5 <sup>a</sup> )	2.716 (4)
OW(5)—Co(2)—OW(3)	89.5 (2)	OW(1)...O(31 <sup>b</sup> )	2.809 (4)
OW(5)—Co(2)—OW(4)	93.9 (4)	OW(2)...O(11 <sup>b</sup> )	2.679 (4)
		OW(3)...O(12 <sup>b</sup> )	2.646 (4)
OW(1)...O(32 <sup>b</sup> )	2.644 (8)	OW(3)...O(12 <sup>c</sup> )	2.730 (5)
OW(1)...O(21 <sup>b</sup> )	2.754 (8)	OW(5)...O(12 <sup>d</sup> )	2.712 (4)
OW(2)...O(12 <sup>b</sup> )	2.708 (8)	O(22)...O(42 <sup>b</sup> )	2.452 (4)
OW(3)...O(22 <sup>b</sup> )	2.796 (8)		
OW(3)...O(12 <sup>c</sup> )	2.673 (8)		
OW(3)...O(22 <sup>d</sup> )	2.820 (8)		
OW(4)...O(22 <sup>b</sup> )	2.766 (8)		
OW(5)...O(11 <sup>b</sup> )	2.802 (8)		
OW(5)...O(21 <sup>b</sup> )	2.783 (9)		
O(41)...O(31 <sup>b</sup> )	2.627 (8)		

Symmetry code: (i)  $\frac{1}{2} - x, \frac{1}{2} - y, 1 - z$ ; (ii)  $\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z$ ; (iii)  $x - 1, 1 - y, z - \frac{1}{2}$ ; (iv)  $-x, 1 - y, -z$ ; (v)  $\frac{1}{2} - x, \frac{1}{2} + y, \frac{3}{2} - z$ ; (vi)  $-x, y, \frac{3}{2} - z$ ; (vii)  $\frac{1}{2} - x, y - \frac{1}{2}, \frac{3}{2} - z$ ; (viii)  $x - \frac{1}{2}, y + \frac{1}{2}, z$ ; (ix)  $x, 1 - y, z + \frac{1}{2}$ ; (x)  $x, y, z$ ; (xi)  $x - \frac{1}{2}, \frac{1}{2} - y, \frac{1}{2} + z$ ; (xii)  $\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$ .

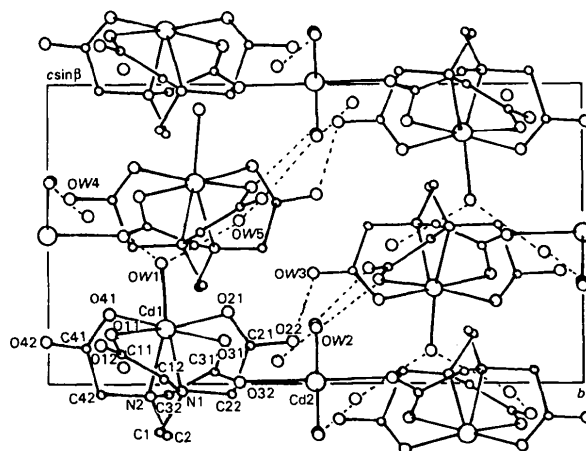


Fig. 1. View of the trimeric units of the Cd complex (structure II).

Cd(2) is located on an inversion centre and it is coordinated to four OW molecules and two O(acetato). As in the Co complex the longest Cd—OW length corresponds to that involving the OW with the greatest number of hydrogen bonds.

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## Structure of $\{N,N\text{-Bis}[(3,5\text{-dimethylpyrazol-1-yl)methyl]aniline\}$ dinitratocobalt(II)

BY KAJ LOCHER, HENK L. BLONK, WILLEM L. DRIESSEN\* AND JAN REEDIJK

*Department of Chemistry, Gorlaeus Laboratories, State University Leiden, PO Box 9502, 2300 RA Leiden, The Netherlands*

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**Abstract.**  $[\text{Co}(\text{NO}_3)_2(\text{C}_{18}\text{H}_{23}\text{N}_5)]$ ,  $M_r = 492.36$ , monoclinic,  $P2_1/n$ ,  $a = 8.400$  (2),  $b = 14.539$  (2),  $c = 17.921$  (3) Å,  $\beta = 95.14$  (1)°,  $V = 2180$  Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.50$  g cm<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71073$  Å,  $\mu = 8.264$  cm<sup>-1</sup>,  $F(000) = 1020$ ,  $T = 295$  K, final  $R = 0.029$  for 1227 significant reflections. The Co<sup>II</sup> ion is coordinated to three ligand N atoms and to three nitrate O atoms; one nitrate ion coordinates monodentately, and one coordinates bidentately. The geometry of the Co<sup>II</sup> ion can be described as being intermediate between a distorted trigonal bipyramid and a square pyramid, in which the O atoms of the bidentately coordinating nitrate group are considered to occupy one donor site. Co—N distances are 2.044 (5) and 2.051 (5) Å for the pyrazole N atoms and 2.401 (5) Å for the aniline N atom. Co—O distances are 2.045 (5) Å for the monodentately coordinating nitrate and 2.036 (5) and 2.470 (5) Å for the nitrate which coordinates anisobidentately.

**Introduction.** Bis[(3,5-dimethylpyrazol-1-yl)methyl]aniline (abbreviated pabd) forms a variety of complexes with transition-metal ions. The synthesis and characterization of these compounds and the X-ray structures of  $[\text{CoCl}_2(\text{pabd})]$  and  $[\text{CuBr}_2(\text{pabd})]$  have been described (Blonk, Driessen & Reedijk, 1985).

From ligand-field data a distorted five-coordinate structure was proposed for the title compound,  $[\text{Co}(\text{NO}_3)_2(\text{pabd})]$ . From infrared data it was proposed that one nitrate group might be coordinated monodentately and one might be coordinated bidentately, and that the aniline N atom would hardly take part in the coordination, if at all (Blonk *et al.*, 1985).

**Experimental.** Red crystals grown from acetonitrile at room temperature. Crystal approximately  $0.5 \times 0.2 \times 0.2$  mm. Enraf–Nonius CAD-4 four-circle diffractometer, graphite-monochromatized Mo  $K\alpha$ . Cell constants from setting angles of 24 reflections,  $10.1 \leq \theta \leq 11.6$ °. Corrections for Lorentz and polarization effects. No absorption correction applied (transmission coefficients 0.92 to 1.04).  $\theta_{\text{max}} = 22.0$ °;  $h$  0 to 8,  $k$  0 to 15,  $l$  -18 to 18. Standard reflections 11 $\bar{9}$ , 35 $\bar{1}$  and 05 $\bar{8}$ , intensity variation 10.64%, corrections applied accordingly. 3027 measured reflections, 2797 independent,  $R_{\text{int}} = 0.052$ , 1570 unobserved with  $I < 2\sigma(I)$ . Structure solved by Patterson method,  $F$  used in LS refinement. Two thirds of the H atoms found in difference Fourier maps; the others placed at 0.95 Å from the parent atoms. Least-squares refinement of positional and anisotropic thermal parameters of non-H atoms; positional parameters of H atoms coupled to parent atoms; fixed isotropic thermal factor of  $5.2 \text{ \AA}^2$  for the H atoms.  $S = 0.828$ ,  $w = 1/\sigma^2(F)$ ,  $R = 0.029$ ,

\* To whom correspondence should be addressed.