$Nb_2Cl_6(C_4H_8S)(PEt_3)_2$, $Nb_2Cl_6(C_4H_8S)(C_4H_8O)_2$ and $Ta_2Cl_6(C_4H_8S)(PMe_3)_2$ are 2·718 (1), 2·684 (2), 2·682 (2) Å, 76·5 (2), 78·0 (1), 76·0 (2), 65·6 (4), 65·1 (1), 64·8 (3)°, respectively.

## References

CHAKRAVARTY, A. R., COTTON, F. A., DIEBOLD, M. P., LEWIS, D. B. & ROTH, W. J. (1986). *J. Am. Chem. Soc.* **108**, 971–976.

COTTON, F. A., DIEBOLD, M. P. & ROTH, W. J. (1987). *Inorg. Chem.* **26**, 4130–4133.

COTTON, F. A., DIEBOLD, M. P. & ROTH, W. J. (1987). *J. Am. Chem. Soc.* **109**, 5506–5144.

COTTON, F. A. & ROTH, W. J. (1983). *Inorg. Chem.* **22**, 3654–3656.

FRENZ, B. A. (1985). *Enraf–Nonius SDP-Plus Structure Determination Package*, Version 3.0. Enraf–Nonius, Delft, The Netherlands.

GILLETTI, P. F., YOUNG, V. G. & BROWN, T. M. (1989). *Inorg. Chem.* **28**, 4034–4036.

JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

MASS, E. T. & McCARLEY, R. E. (1973). *Inorg. Chem.* **12**, 1096–1101.

NORTH, A. C. T., PHILLIPS, D. C. & MATTHEWS, F. S. (1968). *Acta Cryst.* **A24**, 351–359.

SHELDRICK, G. M. (1986). *SHELXS86*. Program for the solution of crystal structures. Univ. of Göttingen, Germany.

# Structure of a Monochloro Bridged Polymeric Copper(II)–Di-2-pyridylamine Complex: [Cu(dipyam)Cl(NO₃)].0·5H₂O

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**Abstract.** Di-2-pyridylaminechloronitratocopper(II) hemihydrate, $[CuCl(NO_3)(C_{10}H_9N_3)].0\cdot5H_2O$, $M_r = 341\cdot21$, monoclinic, $P2_1/a$, $a = 7\cdot382$ (1), $b = 21\cdot494$ (4), $c = 8\cdot032$ (1) Å, $\beta = 94\cdot26$ (1)°, $V = 1270\cdot9$ Å$^3$, $Z = 4$, $D_m = 1\cdot78$, $D_x = 1\cdot782$ g cm$^{-3}$, $\lambda(Mo\ K\alpha) = 0\cdot7107$ Å, $\mu(Mo\ K\alpha) = 19\cdot47$ cm$^{-1}$, $F(000) = 688$. The structure was solved by the heavy-atom method and refined to a final $R$ value of 0·034 for 2736 reflections collected at 294 K. The structure consists of polymeric $[Cu(dipyam)Cl(NO_3)]$ units bridged by a chloride ion.

**Introduction.** $Cu^{II}$ halides show a wide variety of stereochemical complexity (Smith, 1976; Willett & Geiser, 1984). Observed geometries include four-coordinate, five-coordinate and six-coordinate species. Both the inherent flexibility of the $Cu^{II}$ coordination sphere and the non-stereospecific nature of the rather large spherical halide ion play a crucial role in this respect. Further interest in the crystal chemistry of $Cu^{II}$ halides derives from the bridging ability of the halide ions. The halide ions involved in bridge formation can either form two $Cu—X$ bonds of normal length (a symmetrical bridge) or one normal $Cu—X$ and one long, semi-coordinate $Cu\cdots X$ bond (unsymmetrical bridge). The

$Cu^{II}$ ions can be connected by combinations of one, two, or three such bridges.

The di-2-pyridylamine (dipyam) ligand used in this study and similar ligands (rigid or semirigid) are well known for their stabilizing effect of the five-coordinate state of $Cu^{II}$ (Hanson & Hathaway, 1980). However, in complexes containing dipyam, the flexible nature of this ligand results in a greater variety of geometries (Fuller & Jacobson, 1981).

In this paper we report the crystal structure of an unsymmetrically bridged polymeric monochloro-copper complex containing the dipyam ligand.

**Experimental.** The compound was isolated as a by-product in the synthesis of a vitamin $B_6$ complex. Dark green crystals were obtained on slow evaporation of a 2:1:2 mixture of dipyam, pyridoxal hydrochloride and $Cu(NO_3)_2.3H_2O$ in a water/methanol mixture. Density was measured by flotation in a mixture of $CHCl_3$ and $CHBr_3$. A crystal of size 0·28 × 0·21 × 0·12 mm was used in the data collection on a CAD-4 diffractometer. The unit-cell parameters are based on 25 centred reflections within the $\theta$ range 12–18°. Intensity data were collected for 3400 reflections with $\omega/2\theta$ scan, $-9 \leq h \leq 9$, $0 \leq k \leq 28$, $0 \leq l \leq 10$, $(\sin\theta/\lambda)_{max} = 0\cdot66$ Å$^{-1}$. Variations in three standard reflections (441, 413, 4$\bar{8}$2) were less than 8%. The intensities were scaled by the use of these

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standards. The intensities were averaged to 2736 unique reflections $[F > 3\sigma(F)]$, $R_{int} = 0.023$, corrected for Lp and absorption effects with transmission factors in the range 0.958–1.182. The structure was solved by the heavy-atom method and refined on $F$ by full-matrix least squares (SHELX76; Sheldrick, 1976). A peak of 1.76 e Å$^{-3}$ in the difference electron density map at a distance of 0.8 Å from a centre of symmetry was assigned as a statistically disordered O atom of a hydrate water molecule and refined with an occupancy of 0.5. The H-atom positions were located in difference maps and refined in the last few cycles. 212 parameters were refined. Final discrepancy indices are $R = 0.034$, $wR = 0.041$ and $S = 1.35$ with $w = 2.0481/[\sigma^2(F) + 0.000177F^2]$, where $\sigma(F)$ was derived from counting statistics. In the final cycle of refinement $(\Delta/\sigma)_{max} = 0.02$ and the max. and min. heights in the final $\Delta\rho$ maps are 0.38 and $-0.53$ e Å$^{-3}$. Scattering factors used for H, C, N, O, Cl were as available in SHELX and for Cu from International Tables for X-ray Crystallography (1974, Vol. IV, p. 99).

**Discussion.** The fractional coordinates of the non-H atoms are given in Table 1.* The coordination geometry around the copper (Fig. 1) is a trigonally distorted square-based pyramid (SBP). The base is formed by two cis N atoms from the dipyam, a bridging chloride ion and a nitrate O atom while the axial site is occupied by another Cl atom. The complex consists of polymeric [Cu(dipyam)Cl(NO$_3$)]$_x$ zigzag chains running along the crystallographic $a$ axis. A view of one segment is shown in Fig. 1. A chain is propagated through the axial chloride ion Cl' (Fig. 1), which forms part of the base of the screw related molecule. This type of chain structure (Fig. 2) is also observed in the [Cu(dipyam)(CO$_3$)] complex (Sletten, 1984), in which the carbonate group acts as the bridge. The axial Cl ion is tipped slightly towards the O atom of the nitrate group in the basal position [Cl'—Cu—O(1) = 85.25 (4)°].

The equatorial coordination distances (Table 2) vary from 1.974 (2) for N(1) to 2.302 (1) Å for Cl, whereas the axial Cu—Cl' distance is much longer at 2.576 (1) Å, as expected for square pyramidal Cu$^{II}$ complexes. The observed difference of 0.033 (3) Å in the Cu—N distances, 2.007 (2) and 1.974 (2) Å, could be explained either by invoking the difference in trans influence between the O and Cl atoms or it could be considered as a reflection of the trigonal

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Table 1. *Fractional atomic coordinates ($\times 10^5$ for Cu and Cl and $\times 10^4$ for the others) and equivalent isotropic temperature factors ($\times 10^4$) with their e.s.d.'s in parentheses*

$$U_{eq} = (1/3)\Sigma_i \Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i . \mathbf{a}_j.$$

|        | $x$        | $y$       | $z$       | $U_{eq}$ (Å$^2$) |
|--------|-----------|-----------|-----------|---------|
| Cu     | 1063 (4)  | 80549 (1) | 77289 (3) | 197 (1) |
| Cl     | −20173 (8)| 73780 (3) | 65141 (6) | 362 (2) |
| N(1)   | 346 (3)   | 7570 (1)  | 9821 (2)  | 307 (5) |
| C(1)   | 522 (3)   | 6942 (1)  | 9724 (3)  | 361 (7) |
| C(2)   | 373 (3)   | 6560 (1)  | 11067 (3) | 439 (8) |
| C(3)   | −3 (4)    | 6828 (2)  | 12586 (3) | 503 (10)|
| C(4)   | −123 (4)  | 7455 (2)  | 12708 (3) | 461 (8) |
| C(5)   | 102 (3)   | 7828 (1)  | 11306 (3) | 346 (7) |
| N(2)   | 68 (3)    | 8462 (1)  | 11480 (3) | 428 (7) |
| C(6)   | 954 (3)   | 8898 (1)  | 10574 (3) | 359 (7) |
| C(7)   | 1515 (4)  | 9459 (1)  | 11366 (3) | 456 (8) |
| C(8)   | 2429 (4)  | 9885 (1)  | 10471 (4) | 534 (10)|
| C(9)   | 2804 (4)  | 9757 (1)  | 8833 (4)  | 519 (9) |
| C(10)  | 2198 (4)  | 9204 (1)  | 8148 (3)  | 423 (8) |
| N(3)   | 1245 (3)  | 8783 (1)  | 8973 (2)  | 350 (6) |
| N(4)   | −1563 (3) | 8933 (1)  | 5550 (3)  | 431 (7) |
| O(1)   | −177 (2)  | 8567 (1)  | 5627 (2)  | 365 (5) |
| O(2)   | −1908 (4) | 9218 (1)  | 4245 (3)  | 761 (9) |
| O(3)   | −2473 (3) | 8980 (1)  | 6758 (3)  | 613 (7) |
| O(W)*  | 648 (12)  | 4694 (4)  | 5289 (11) | 1619 (38)|

* Occupancy 0.5, isotropic temperature factor.

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distortion in bond lengths, the Cu—N(3) bond, equatorial $(r_e)$ in a trigonal bipyramid, being longer than the axial Cu—N(1) bond $(r_a)$. This is in agreement with previous reports (Muetterties & Guggenberger, 1974; Wood, 1972) that the ratio of $r_a/r_e$ in $d^8$ and $d^9$ complexes of trigonal bipyramidal geometry tends to be slightly less than unity. The equatorial Cu—Cl bond is normal, whereas the axial bond, 2.576 (1) Å, is shorter than that expected for a bridging chloride in the axial position (Hathaway, 1987a). However, this distance is very close to the value of 2.570 (2) Å reported for the monomeric pentachlorocuprate(II) with a square-based pyramidal structure (Antolini, Marcotrigiano, Menabue & Pellacani, 1980).

The cis angles around the Cu atom (Table 2) vary from 88.0 to 92.4 (1)°. The N—Cu—N angle, 88.8 (1)°, is close to the reported value of 89.9 (1)° found in the [Cu(dipyam)(H$_2$O)F]$^+$ complex (Jacobson & Jensen, 1981). This value varies from ~86 to ~96° in dipyam complexes. The N—N distance between the pyridyl N atoms, 2.786 (3) Å, is smaller than the corresponding values (2.824 and 2.806 Å) found in the related [Cu(dipyam)$_2$Cl] (Jensen & Jacobson, 1981) and [Cu(dipyam)(H$_2$O)F]$^+$ complexes.

The equatorial atoms show significant distortion [−0.236 (2) to 0.189 (2) Å] from the best plane through these atoms, the Cu atom being displaced by 0.2011 (3) Å from this plane towards the axial Cl' atom. The percentage of trigonal distortion, $\tau$ (Hathaway, 1987b), observed in the present complex, 27.5%, is intermediate between that found for

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* Lists of structure factors, anisotropic thermal parameters, least-squares planes, some torsion angles, distances and angles involving H atoms, and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53943 (25 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

bis(dipyam) complexes ($\simeq 120°$) and for the [Cu(dipyam)(H₂O)F]⁺ complex (15·2%). The relative decrease in steric effect of the ligands in these complexes seems to be one of the factors for this decrease. The six-membered chelate ring, Cu—N(1)—C(5)—N(2)—C(6)—N(3), is in the boat configuration with Cu and bridging N atoms 0·7133 (3)
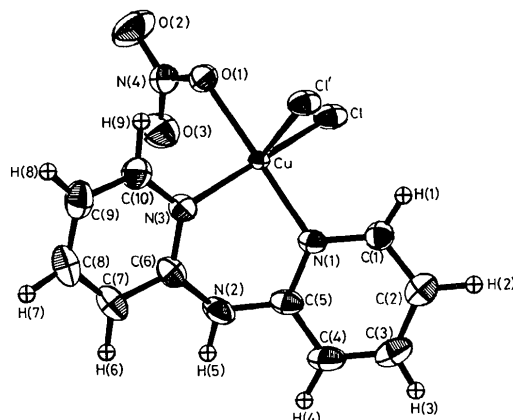
Fig. 1. *ORTEP* (Johnson, 1976) drawing of the asymmetric unit with labelling scheme used. Thermal ellipsoids scaled to 50% probability. Cl' is related to Cl by symmetry.
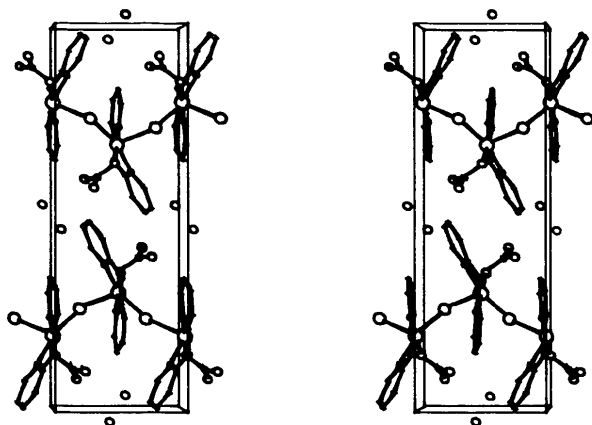
Fig. 2. Stereoview (Motherwell & Clegg, 1978) of the unit cell down the c axis (a horizontal, b vertical). The hydrate water has occupancy of 0·5.

and 0·268 (2) Å, respectively, above the plane of the other four atoms. A similar configuration is found in most dipyam complexes (Jacobson & Jensen, 1981; Jensen & Jacobson, 1981; Johnson & Jacobson, 1973a).

There are no unusual bond distances or bond angles in the dipyam ligand (Table 2). While two pyridine rings are individually planar they make a dihedral angle of 30·4°. This is close to the value of 33° reported for [Cu(dipyam)₂I] (Johnson & Jacobson, 1973a). In dipyam complexes this dihedral angle can range from 9·8 to 41·4°, as compared to 23° found in the solid state for dipyridylamine (Johnson & Jacobson, 1973b).

The monoatomic asymmetric bridging nature shown here by the halide ion is noteworthy. There are no linear chain structures for the Cu^{II} ion involving a single short-bonded monoatomic bridging anion (Cu—$X$—Cu—$X$), but twisted linear chains

Table 2. *Bond lengths (Å) and angles (°) involving non-H atoms with their e.s.d.'s in parentheses*

| | | | |
|---|---|---|---|
| Cu—Cl' | 2·576 (1) | C(5)—N(2) | 1·371 (3) |
| Cu—Cl | 2·302 (1) | N(2)—C(6) | 1·381 (3) |
| Cu—N(1) | 1·974 (2) | C(6)—C(7) | 1·410 (4) |
| Cu—N(3) | 2·007 (2) | C(6)—N(3) | 1·342 (3) |
| Cu—O(1) | 2·012 (2) | C(7)—C(8) | 1·371 (4) |
| N(1)—C(1) | 1·359 (3) | C(8)—C(9) | 1·392 (4) |
| N(1)—C(5) | 1·339 (3) | C(9)—C(10) | 1·370 (4) |
| C(1)—C(2) | 1·367 (4) | C(10)—N(3) | 1·351 (3) |
| C(2)—C(3) | 1·395 (4) | N(4)—O(1) | 1·289 (3) |
| C(3)—C(4) | 1·355 (5) | N(4)—O(2) | 1·224 (3) |
| C(4)—C(5) | 1·402 (4) | N(4)—O(3) | 1·225 (3) |
| | | | |
| Cl'—Cu—Cl | 99·55 (3) | N(1)—C(5)—N(2) | 120·5 (2) |
| Cl'—Cu—N(1) | 96·2 (1) | C(4)—C(5)—N(2) | 118·8 (2) |
| Cl'—Cu—N(3) | 98·2 (1) | C(5)—N(2)—C(6) | 127·5 (2) |
| Cl'—Cu—O(1) | 85·25 (2) | N(2)—C(6)—C(7) | 118·5 (2) |
| Cl—Cu—N(1) | 92·4 (1) | N(2)—C(6)—N(3) | 119·7 (2) |
| Cl—Cu—N(3) | 161·9 (1) | C(7)—C(6)—N(3) | 121·8 (2) |
| Cl—Cu—O(1) | 87·99 (4) | C(6)—C(7)—C(8) | 118·3 (3) |
| N(1)—Cu—N(3) | 88·8 (1) | C(7)—C(8)—C(9) | 120·0 (3) |
| N(1)—Cu—O(1) | 178·4 (1) | C(8)—C(9)—C(10) | 118·0 (3) |
| N(3)—Cu—O(1) | 90·3 (1) | C(9)—C(10)—N(3) | 123·4 (2) |
| Cu—N(1)—C(1) | 118·6 (2) | Cu—N(3)—C(6) | 122·0 (2) |
| Cu—N(1)—C(5) | 122·0 (2) | Cu—N(3)—C(10) | 119·5 (2) |
| C(1)—N(1)—C(5) | 118·8 (2) | C(6)—N(3)—C(10) | 118·2 (2) |
| N(1)—C(1)—C(2) | 122·5 (2) | O(1)—N(4)—O(2) | 117·5 (2) |
| C(1)—C(2)—C(3) | 118·4 (2) | O(1)—N(4)—O(3) | 119·5 (2) |
| C(2)—C(3)—C(4) | 119·5 (3) | O(2)—N(4)—O(3) | 123·1 (2) |
| C(3)—C(4)—C(5) | 119·9 (3) | Cu—O(1)—N(4) | 114·0 (1) |
| N(1)—C(5)—C(4) | 120·7 (2) | | |

Table 3. *Crystallographic data on copper complexes involving a single bridge (Cl) chain*

| Number | Complex | Stereo-chemistry | τ (%) | Cu—Cl_{eq} (Å) | Cu—Cl_{ax} (Å) | Cu—Cl—Cu (°) | Cl—Cu—Cl (°) | Cu—Cu—Cu (°) | Reference |
|---|---|---|---|---|---|---|---|---|---|
| (1) | [CuCl₂(DMSO)₂] | SBP | 44·8 | 2·290 (2) | 2·702 (2) | 145 | 112·67 (5) | 115·7 | (a) |
| (2) | [Cu(imH)₂Cl₂] | SBP | 29·0 | 2·365 (4) | 2·751 (6) | 117·0 | 97·1 (1) | 102·3 | (b) |
| (3) | [Cu(caf)(H₂O)Cl₂] | SBP | 29·7 | 2·319 (2) | 2·788 (2) | 128·1 | 89·5 (1) | 78·3 | (c) |
| (4) | [Cu(maep)Cl₂] | SBP | 17·2 | 2·300 (2) | 2·785 (2) | 113·6 | 93·97 (4) | 99·8 | (d) |
| (5) | [Cu(dipyam)(NO₃)Cl] | SBP | 27·5 | 2·302 (1) | 2·576 (1) | 128·5 | 99·55 (3) | 114·2 | (e) |

imH = imidazole, maep = 2-(2-methylaminoethyl)pyridine, caf = caffeine.

References: (a) Willet & Chang (1970); (b) Lundberg (1972); (c) Bandoli, Biagini, Clemente & Rizzardi (1976); (d) Bream, Estes & Hodgson (1975); (e) This work.

are observed (Hathaway, 1987a). In general, a trigonally distorted $CuCl_2L_2Cl'$ chromophore is involved, with a long Cu—Cl bridging distance ($\simeq 2\cdot75$ Å) and a non-linear Cu—Cl—Cu angle of 114–145°. Table 3 gives some crystallographic data on copper complexes involving single bridge ($Cl^-$) chains. It may be noted that the present complex contains a trigonally distorted $CuN_2OClCl'$ chromophore. A perusal of Table 3 shows that the axial Cu—Cl distance is shortest in the present complex. It has been reported (Willett & Geiser, 1984) that for $Cu^{II}$ halides the bonding nature of the bridging halide atoms affects the $Cu^{II}$ geometry. Both $Cu^{II}$ coordination geometry and the parameters involving the bridging atom are important in the formation of Cu—X—Cu linear chains. It is interesting to note (Table 3) that even though the Cu—Cl—Cu angle in the present complex is equal to that found in complex (3), the former is more linear as is evident from the Cu—Cu—Cu angle. The Cl—Cu—Cl angles for these two complexes are 99·55 (3) and 89·5 (1)°, respectively. This shows that the angle made by Cl atoms at Cu is one of the factors responsible for linearity in the present system. The Cu—Cu—Cu angle in the present complex is very close to that in complex (1), the best linear chain observed so far among these complexes.

The chain structure is further stabilized by hydrogen bonds, van der Waals and stacking interactions. A pyridine ring of dipyam is involved in stacking interactions with the pyridine ring of the glide-related dipyam moiety with a separation of 3·50 Å (Fig. 2). The possible hydrogen bonds and van der Waals contact distances ($<3\cdot5$ Å) have been deposited.*

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\* See deposition footnote.

**References**

ANTOLINI, L., MARCOTRIGIANO, G., MENABUE, L. & PELLACANI, G. C. (1980). *J. Am. Chem. Soc.* **102**, 1303–1309.
BANDOLI, G., BIAGINI, M. C., CLEMENTE, D. A. & RIZZARDI, G. (1976). *Inorg. Chim. Acta*, **20**, 71–78.
BREAM, R. A., ESTES, E. D. & HODGSON, D. J. (1975). *Inorg. Chem.* **14**, 1672–1675.
FULLER, G. C. & JACOBSON, R. A. (1981). *Inorg. Chim. Acta*, **48**, 191–197.
HANSON, W. D. & HATHAWAY, B. J. (1980). *Acta Cryst.* B36, 1069–1074.
HATHAWAY, B. J. (1987a). *Comprehensive Coordination Chemistry*, Vol. 5, edited by G. WILKINSON, pp. 640–641. Oxford: Pergamon Press.
HATHAWAY, B. J. (1987b). *Comprehensive Coordination Chemistry*, Vol. 5, edited by G. WILKINSON, pp. 607–609. Oxford: Pergamon Press.
JACOBSON, R. A. & JENSEN, W. P. (1981). *Inorg. Chim. Acta*, **52**, 205–209.
JENSEN, W. P. & JACOBSON, R. A. (1981). *Inorg. Chim. Acta*, **49**, 199–204.
JOHNSON, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
JOHNSON, J. E. & JACOBSON, R. A. (1973a). *J. Chem. Soc. Dalton Trans.* pp. 580–584.
JOHNSON, J. E. & JACOBSON, R. A. (1973b). *Acta Cryst.* B36, 1669–1674.
LUNDBERG, B. K. S. (1972). *Acta Chem. Scand. Ser. A*, **26**, 3977–3983.
MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.
MUETTERTIES, E. L. & GUGGENBERGER, L. J. (1974). *J. Am. Chem. Soc.* **96**, 1748–1756.
SHELDRICK, G. M. (1976). *SHELX*76. A program for crystal structure determination. Univ. of Cambridge, England.
SLETTEN, J. (1984). *Acta Chem. Scand. Ser. A*, **38**, 491–496.
SMITH, D. W. (1976). *Coord. Chem. Rev.* **21**, 93–158.
WILLETT, R. D. & CHANG, K. (1970). *Inorg. Chim. Acta*, **4**, 447–451.
WILLETT, R. D. & GEISER, U. (1984). *Croat. Chim. Acta*, **57**, 737–747.
WOOD, J. S. (1972). *Progress in Inorganic Chemistry*, Vol. 16, edited by S. J. LIPPARD, pp. 227–486. New York: Interscience.

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# Structure of the Decahydrated Octaacetate of Dineodymium(III) and Cobalt(II)

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**Abstract.** Octakis($\mu$-acetato-$O{:}O'$)-decaaquacobalt-(II)dineodymium(III), $[CoNd_2(\mu\text{-}C_2H_3O_2)_8(H_2O)_{10}]$, $M_r = 999\cdot92$, triclinic, $P\bar{1}$, $a = 8\cdot243$ (2), $b =$ 10·368 (3), $c = 11\cdot497$ (3) Å, $\alpha = 111\cdot64$ (1), $\beta = 107\cdot94$ (1), $\gamma = 93\cdot45$ (1)°, $V = 851$ (1) Å$^3$, $Z = 1$, $D_x = 1\cdot95$ Mg m$^{-3}$, Mo $K\alpha$, $\lambda = 0\cdot71069$ Å, $\mu =$