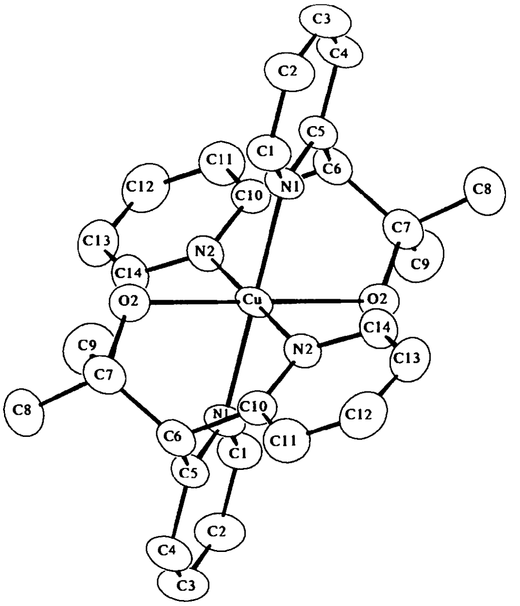

Fig. 1. A perspective view of the molecule with atom numbering. H atoms excluded for clarity.

65·8° for the di(2-pyridyl)methanol ligand in $[Cu(C_{11}H_{10}N_2)(C_{11}H_{10}NO)Cl]ClO_4$ (Garland, Le-Marouille, Spodine & Manzur, 1986).

The Cu—O(2) bonds are 2·294 (2) Å, longer than the Cu—O distances usually reported for the alcohol groups [*International Tables for X-ray Crystallography* (1974, Vols. II and IV)], but shorter than the one found for the copper(II) complex with the di(2-pyridyl)methanol ligand, whose Cu—OH distance is 2·660 (5) Å. The angles of the 2-propanol group are those corresponding to tetrahedral symmetry. The C—O bond is 1·422 Å which is comparable with the usual distance for this group.

The C—N bonds are more similar to those found for the dimeric dibromo[di(2-pyridyl)methane]-copper(II) complex (Garland, Grandjean, Spodine & Manzur, 1987), than to the ones found for the oxygenated ligand. The other distances and angles of the ligand are approximately as expected.

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Structure of a Binuclear Cu^{II} Complex of Tetraazacyclohexadecanetetraacetate

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Abstract. $[Cu(H_2O)_6]^{2+}.\frac{1}{4}[(\{Cu_2(C_{20}H_{32}N_4O_8)Cl\}^-)_4].[Cu_2(C_{20}H_{32}N_4O_8)(H_2O)Cl]^-.15H_2O$, $C_{40}H_{108}Cl_2N_8O_{38}$, $M_r = 1697{\cdot}9$, tetragonal, $I\bar{4}$, $a = 24{\cdot}702\ (7)$, $c = 25{\cdot}768\ (5)$ Å, $V = 15723$ Å^3, $Z = 8$, $D_x = 1{\cdot}435$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 1{\cdot}486$ mm^{-1}, $F(000) = 7095$, $T = 293$ K, $R = 0{\cdot}061$ for 3749 observed data. The structure contains five crystallographically distinct Cu^{II} species. Two are involved in a monomeric binuclear complex with the macrocyclic ligand, two are bound to a similar complex which is linked as a tetramer and the fifth is the aqua complex which acts as a countercation.

0108-2701/91/061185-04$03.00

Table 1. *Data collection and refinement details*

Crystal dimensions (mm)	0·2 × 0·2 × 0·2
Scan mode	$\omega/2\theta$
Scan width (°)	0·70 + 0·345tanθ
Scan speed (° min^{-1})	2·1–16·6
Range 2θ (°)	4–40
Unique reflections measured	7363
$R_{\rm int}$ (for 303 pairs)	0·042
Reflections [$I \geq 2{\cdot}5\sigma(I)$]	3749
Refined parameters	392
R	0·061
wR*	0·078
S	1·03

*$w = [2{\cdot}451t_0(X) + 3{\cdot}113t_1(X) + 1{\cdot}198t_2(X)]^{-1}$, where $X = |F_o|/F_{\max}$ and t_n are polynomial functions of the Chebyshev series (Carruthers & Watkin, 1979) proved satisfactory.

Introduction. The transition metal complexes of tetraaza macrocyclic tetraacetates show a great variety of different structures depending on the size and number of the coordinated metal ions and on the size of the macrocyclic ring (Riesen, Zehnder & Kaden, 1985, 1986, 1988). In this work the structure of a binuclear $\mathrm{Cu^{II}}$ complex of 1,5,9,13-tetraazacyclohexadecane-*N*,*N*′,*N*″,*N*‴-tetraacetate (*L*) is reported, which is the first example of the 16-membered representative of this type of ligand.

Experimental. Dark blue, octahedron-shaped crystals were obtained from recrystallization from water/ethanol (Riesen *et al.*, 1986). The crystals rapidly lost solvent and crystallinity on exposure to the air. Therefore the crystals were mounted in a 0·2 mm glass capillary together with mother liquor. Cell-parameter determination was performed using an Enraf–Nonius CAD-4 diffractometer yielding a *C*-centred cell with $a = 35{\cdot}654$ (5), $b = 24{\cdot}691$ (7), $c = 24{\cdot}714$ (7) Å, $\beta = 133{\cdot}72$ (2)°, $\alpha = \gamma = 90°$, which was used for data acquisition. Four intensity standards were measured every hour of acquisition time and showed a systematic loss of intensity of about 2%. The initial cell and the collected data were transformed to a tetragonal *I*-centred cell. Unit-cell parameters and data collection details are shown in Table 1. Data reduction including Lorentz and polarization corrections and structure solution were carried out using the *NRCVAX* program package (Gabe, Larson, Lee & LePage, 1984). The missing non-H atoms were localized by electron-density Fourier syntheses. An empirical correction to the data was applied after the refinement of the isotropic model was complete using the procedure *DIFABS* (Walker & Stuart, 1983) within the system *CRYSTALS* (Watkin, Carruthers & Betteridge, 1985) on which subsequent calculations were made. Because the crystal was diffracting weakly (a reasonable fraction of reflections was observed only to $\theta = 20°$) we tried to keep the model as simple as possible. Only the Cu and Cl atoms were refined anisotropically. Isotropic temperature factors of chemically

Table 2. *Fractional atomic coordinates* (× 10^4; 10^5 *for* Cu) *and isotropic or equivalent isotropic temperature factors* (Å^2 × 10^4)

$$U_{\rm eq} = (1/3)\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm iso}/U_{\rm eq}$	Occ.
Cu(31)	73990 (13)	52698 (13)	13852 (12)	554	
Cu(11)	70393 (11)	36813 (11)	27683 (10)	456	
Cu(12)	82009 (11)	51909 (11)	33027 (10)	457	
Cu(21)	79222 (11)	65631 (11)	−2489 (10)	466	
Cu(22)	65414 (10)	51935 (10)	−5018 (9)	382	
Cl(11)	6402 (3)	2995 (3)	2292 (2)	617	
Cl(21)	8641 (3)	7236 (3)	113 (2)	581	
N(11)	6574 (7)	3769 (7)	3437 (7)	451 (17)	
C(12)	6452 (10)	3200 (9)	3650 (9)	551 (24)	
C(13)	6948 (10)	2864 (10)	3768 (9)	588 (35)	
C(14)	7250 (9)	2683 (9)	3341 (9)	551 (24)	
N(15)	7555 (7)	3117 (7)	3047 (7)	451 (17)	
C(16)	7981 (9)	3313 (9)	3408 (9)	523 (23)	
C(17)	8249 (9)	3852 (9)	3233 (9)	461 (30)	
C(18)	8558 (9)	4083 (9)	3653 (9)	523 (23)	
N(19)	8757 (7)	4668 (7)	3580 (7)	460 (17)	
C(110)	8975 (10)	4875 (10)	4088 (9)	551 (24)	
C(111)	8596 (10)	4979 (10)	4500 (9)	588 (35)	
C(112)	8179 (9)	5427 (9)	4407 (8)	551 (24)	
N(113)	7794 (7)	5311 (7)	3963 (6)	460 (17)	
C(114)	7427 (10)	4892 (9)	4088 (8)	523 (23)	
C(115)	7062 (9)	4644 (9)	3691 (8)	461 (30)	
C(116)	6834 (9)	4125 (10)	3844 (9)	523 (23)	
C(117)	6086 (9)	4030 (9)	3216 (9)	561 (24)	
C(118)	7799 (10)	2895 (10)	2568 (9)	561 (24)	
C(119)	9184 (10)	4664 (9)	3161 (9)	561 (24)	
C(120)	7470 (10)	5830 (10)	3876 (9)	561 (24)	
C(121)	6230 (10)	4393 (10)	2777 (9)	583 (25)	
C(122)	7813 (10)	3302 (10)	2139 (10)	583 (25)	
C(123)	8950 (10)	4748 (10)	2633 (10)	583 (25)	
C(124)	7306 (11)	5866 (11)	3307 (10)	647 (25)	
O(11)	6655 (6)	4340 (6)	2532 (6)	537 (14)	
O(12)	7532 (6)	3740 (6)	2200 (6)	537 (14)	
O(13)	8460 (6)	4991 (6)	2628 (5)	537 (14)	
O(14)	7596 (6)	5611 (6)	2991 (6)	537 (14)	
O(15)	5865 (7)	4761 (7)	2669 (6)	682 (35)	
O(16)	8105 (7)	3229 (7)	1750 (7)	760 (37)	
O(17)	9160 (7)	4610 (6)	2222 (7)	683 (34)	
O(18)	6924 (8)	6157 (7)	3179 (7)	816 (54)	
O(41)	8763 (6)	5956 (6)	3402 (6)	648 (49)	
N(21)	7412 (7)	7139 (7)	−560 (7)	451 (17)	
C(22)	7720 (9)	7532 (9)	−912 (8)	551 (24)	
C(23)	8106 (9)	7266 (9)	−1360 (8)	588 (35)	
C(24)	8474 (9)	6866 (9)	−1205 (9)	551 (24)	
N(25)	8302 (7)	6377 (7)	−892 (7)	451 (17)	
C(26)	8007 (9)	6003 (9)	−1253 (9)	523 (23)	
C(27)	7733 (9)	5537 (9)	−983 (8)	461 (30)	
C(28)	7318 (9)	5300 (9)	−1362 (9)	523 (23)	
N(29)	6896 (7)	4915 (7)	−1152 (6)	460 (17)	
C(210)	6535 (10)	4765 (9)	−1558 (9)	551 (24)	
C(211)	6168 (10)	5247 (9)	−1725 (9)	588 (35)	
C(212)	5750 (9)	5424 (10)	−1333 (9)	551 (24)	
N(213)	6004 (7)	5703 (7)	−856 (6)	460 (17)	
C(214)	6259 (9)	6235 (10)	−1026 (9)	523 (23)	
C(215)	6630 (9)	6476 (9)	−612 (8)	461 (30)	
C(216)	6976 (9)	6933 (9)	−872 (9)	523 (23)	
C(217)	7225 (10)	7404 (9)	−76 (9)	561 (24)	
C(218)	8801 (10)	6093 (10)	−685 (9)	561 (24)	
C(219)	7208 (10)	4419 (9)	−962 (9)	561 (24)	
C(220)	5598 (9)	5783 (9)	−449 (9)	561 (24)	
C(221)	7169 (10)	7004 (10)	383 (9)	583 (25)	
C(222)	8717 (10)	5775 (10)	−209 (10)	583 (25)	
C(223)	7367 (10)	4484 (10)	−421 (9)	583 (25)	
C(224)	5794 (8)	5743 (8)	99 (8)	360 (58)	
O(21)	7438 (6)	6583 (6)	355 (5)	537 (14)	
O(22)	8291 (6)	5920 (6)	40 (6)	537 (14)	
O(23)	7095 (6)	4794 (6)	−128 (5)	537 (14)	
O(24)	6269 (6)	5543 (6)	144 (5)	537 (14)	
O(25)	6889 (7)	7181 (7)	754 (6)	682 (35)	
O(26)	9014 (7)	5418 (7)	−80 (7)	760 (37)	

Table 2 (*cont.*)

	x	y	z	U_{iso}/U_{eq}	Occ.
O(27)	7769 (7)	4232 (6)	−253 (6)	683 (34)	
O(28)	5547 (6)	5935 (6)	468 (6)	485 (40)	
O(31)	6585 (7)	5218 (7)	1168 (6)	525 (51)	0·86 (2)
O(32)	7438 (7)	4449 (7)	1364 (7)	667 (59)	0·86 (2)
O(33)	7114 (7)	5248 (7)	2145 (7)	634 (59)	0·86 (2)
O(34)	7651 (7)	5317 (8)	650 (7)	687 (62)	0·86 (2)
O(35)	8177 (8)	5352 (8)	1634 (7)	775 (66)	0·86 (2)
O(36)	7271 (8)	6159 (8)	1418 (8)	814 (67)	0·86 (2)
O(331)	7489 (12)	5823 (14)	1906 (13)	74 (141)	0·14 (2)
O(332)	7831 (13)	4776 (13)	1833 (13)	74 (141)	0·14 (2)
O(333)	8126 (18)	5373 (12)	1023 (13)	774 (315)	0·14 (2)
O(334)	6692 (17)	5096 (12)	1685 (13)	774 (315)	0·14 (2)
O(335)	7315 (12)	4667 (14)	854 (13)	74 (141)	0·14 (2)
O(336)	6963 (13)	5685 (13)	882 (13)	74 (141)	0·14 (2)
O(42)	8199 (10)	3957 (10)	684 (9)	1299 (82)	
O(43)	5953 (8)	5544 (8)	1929 (7)	983 (64)	
O(44)	6592 (9)	6441 (9)	2222 (8)	1141 (69)	
O(45)	8920 (9)	4932 (9)	913 (8)	1164 (73)	
O(46)	6212 (13)	7448 (12)	2385 (12)	1894 (117)	
O(47)	6663 (18)	1522 (18)	4160 (17)	2000	0·76 (5)
O(48)	5843 (19)	2006 (19)	2744 (19)	2000	0·68 (5)
O(49)	7331 (21)	4727 (21)	5641 (21)	2000	0·63 (5)
O(50)	6151 (18)	6301 (18)	3809 (18)	2000	0·76 (5)
O(51)	6498 (18)	3485 (18)	5094 (18)	2000	0·73 (5)
O(52)	5000	0	542 (36)	2000	0·52 (4)
O(53)	5000	5000	3250 (20)	2000	0·92 (4)
O(54)	1479 (21)	325 (20)	379 (19)	2000	0·62 (5)
O(55)	9841 (29)	881 (29)	762 (25)	2000	0·47 (5)
O(56)	3686 (18)	1559 (19)	1847 (18)	2000	0·72 (5)
O(57)	2206 (22)	92 (22)	2080 (20)	2000	0·61 (5)
O(58)	2968 (33)	698 (35)	957 (31)	2000	0·40 (5)
O(59)	4649 (26)	1321 (26)	1610 (26)	2000	0·50 (5)
O(60)	8845 (27)	422 (26)	1908 (25)	2000	0·49 (5)

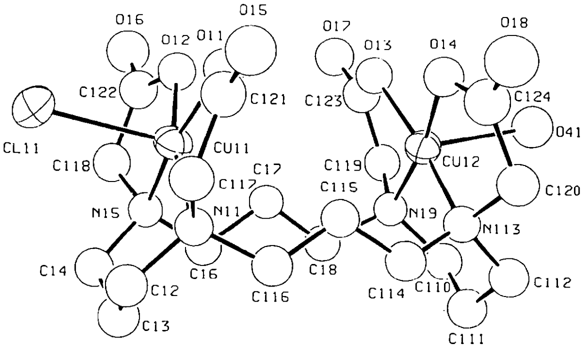

Fig. 1. Structure of the monomeric complex anion *A*. 50% enclosure thermal ellipsoids.

similar atoms were assigned to one of twelve groups (*e.g.* the eight N atoms). The $Cu(H_2O)_6^{2+}$ cation was refined with a disordered model using geometrical restraints to preserve orthogonal geometry of the two orientations which were found to be occupied at 86 (2)% [O(31) until O(36)] and 14% [O(331) until O(336)], respectively. The minor fraction (14%), although not modelled in an entirely satisfactory way, was introduced to maintain the simplicity of the model. Some water molecule sites of the complicated solvent network were not fully occupied and therefore their occupancy was varied while the thermal parameters were kept equivalent. H atoms were included in calculated positions only for the methylene groups of the ligand. In the final blocked-matrix least-squares refinement minimizing $\sum w(|F_o| - |F_c|)^2$ the maximum shift/e.s.d. dropped below 0·04. The difference map had a largest peak of 1·1 (3) e Å^{-3}, 1·5 Å from Cu(31). Computations were performed on a MicroVAX II computer. Fig. 1 was generated using the program *ORTEP* (Johnson, 1965). The final positional and thermal motion parameters are given in Table 2.* Selected bond distances and angles are given in Table 3.

Table 3. *Selected distances* (Å) *and angles* (°)

Cu(11)—Cl(11)	2·619 (7)	Cu(21)—Cl(21)	2·604 (7)
Cu(11)—N(11)	2·08 (2)	Cu(21)—N(21)	2·06 (2)
Cu(11)—N(15)	2·02 (2)	Cu(21)—N(25)	1·96 (2)
Cu(11)—O(11)	1·98 (2)	Cu(21)—O(21)	1·96 (1)
Cu(11)—O(12)	1·91 (1)	Cu(21)—O(22)	1·98 (1)
Cu(12)—N(19)	2·02 (2)	Cu(22)—N(29)	2·01 (2)
Cu(12)—N(113)	2·00 (2)	Cu(22)—N(213)	2·04 (2)
Cu(12)—O(13)	1·92 (1)	Cu(22)—O(23)	1·94 (1)
Cu(12)—O(14)	1·99 (2)	Cu(22)—O(24)	1·99 (1)
Cu(12)—O(41)	2·36 (2)	Cu(22)—O(28)	2·37 (1)
Cu(31)—O(31)	2·09 (2)	Cu(31)—O(331)	1·93 (4)
Cu(31)—O(32)	2·03 (2)	Cu(31)—O(332)	1·99 (4)
Cu(31)—O(33)	2·08 (2)	Cu(31)—O(333)	2·04 (5)
Cu(31)—O(34)	2·00 (2)	Cu(31)—O(334)	1·96 (5)
Cu(31)—O(35)	2·04 (2)	Cu(31)—O(335)	2·03 (4)
Cu(31)—O(36)	2·22 (2)	Cu(31)—O(336)	1·97 (4)

N(11)—Cu(11)—Cl(11)	97·1 (5)	O(24)—Cu(22)—O(23)	92·5 (6)
N(15)—Cu(11)—Cl(11)	95·7 (5)	O(28)—Cu(22)—N(29)	92·5 (6)
N(15)—Cu(11)—N(11)	97·2 (7)	O(28)—Cu(22)—N(213)	94·7 (6)
O(11)—Cu(11)—Cl(11)	95·7 (5)	O(28)—Cu(22)—O(23)	92·0 (6)
O(11)—Cu(11)—N(11)	84·5 (7)	O(28)—Cu(22)—O(24)	95·1 (5)
O(11)—Cu(11)—N(15)	168·2 (7)	O(32)—Cu(31)—O(31)	88·7 (7)
O(12)—Cu(11)—Cl(11)	94·2 (5)	O(33)—Cu(31)—O(31)	85·7 (7)
O(12)—Cu(11)—N(11)	168·0 (7)	O(33)—Cu(31)—O(32)	90·9 (7)
O(12)—Cu(11)—N(15)	85·6 (7)	O(34)—Cu(31)—O(31)	92·8 (7)
O(12)—Cu(11)—O(11)	90·4 (6)	O(34)—Cu(31)—O(32)	91·0 (8)
N(113)—Cu(12)—N(19)	97·9 (7)	O(34)—Cu(31)—O(33)	177·6 (8)
O(13)—Cu(12)—N(19)	85·9 (7)	O(35)—Cu(31)—O(31)	176·3 (7)
O(13)—Cu(12)—N(113)	168·0 (7)	O(35)—Cu(31)—O(32)	93·6 (8)
O(14)—Cu(12)—N(19)	171·6 (7)	O(35)—Cu(31)—O(33)	91·4 (7)
O(14)—Cu(12)—N(113)	83·5 (6)	O(35)—Cu(31)—O(34)	89·9 (8)
O(14)—Cu(12)—O(13)	91·1 (6)	O(36)—Cu(31)—O(31)	86·2 (7)
O(41)—Cu(12)—N(19)	94·3 (6)	O(36)—Cu(31)—O(32)	174·5 (8)
O(41)—Cu(12)—N(113)	94·8 (6)	O(36)—Cu(31)—O(33)	86·7 (7)
O(41)—Cu(12)—O(13)	96·2 (6)	O(36)—Cu(31)—O(34)	91·3 (8)
O(41)—Cu(12)—O(14)	93·9 (6)	O(36)—Cu(31)—O(35)	91·4 (8)
N(21)—Cu(21)—Cl(21)	96·6 (5)	O(332)—Cu(31)—O(331)	88·2 (12)
N(25)—Cu(21)—Cl(21)	97·3 (5)	O(333)—Cu(31)—O(331)	97·4 (12)
N(25)—Cu(21)—N(21)	97·2 (7)	O(333)—Cu(31)—O(332)	82·6 (12)
O(21)—Cu(21)—Cl(21)	96·6 (5)	O(334)—Cu(31)—O(331)	89·0 (12)
O(21)—Cu(21)—N(21)	85·3 (6)	O(334)—Cu(31)—O(332)	96·6 (12)
O(21)—Cu(21)—N(25)	165·5 (7)	O(334)—Cu(31)—O(333)	173·5 (12)
O(22)—Cu(21)—Cl(21)	93·7 (4)	O(335)—Cu(31)—O(331)	178·0 (11)
O(22)—Cu(21)—N(21)	169·2 (6)	O(335)—Cu(31)—O(332)	89·8 (11)
O(22)—Cu(21)—N(25)	84·8 (7)	O(335)—Cu(31)—O(333)	82·7 (11)
O(22)—Cu(21)—O(21)	90·2 (6)	O(335)—Cu(31)—O(334)	90·8 (12)
N(213)—Cu(22)—N(29)	96·9 (7)	O(336)—Cu(31)—O(331)	98·7 (12)
O(23)—Cu(22)—N(29)	86·2 (7)	O(336)—Cu(31)—O(332)	173·1 (12)
O(23)—Cu(22)—N(213)	172·5 (7)	O(336)—Cu(31)—O(333)	96·6 (12)
O(24)—Cu(22)—N(29)	172·3 (7)	O(336)—Cu(31)—O(334)	83·5 (12)
O(24)—Cu(22)—N(213)	83·5 (7)	O(336)—Cu(31)—O(335)	83·3 (11)

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53777 (52 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

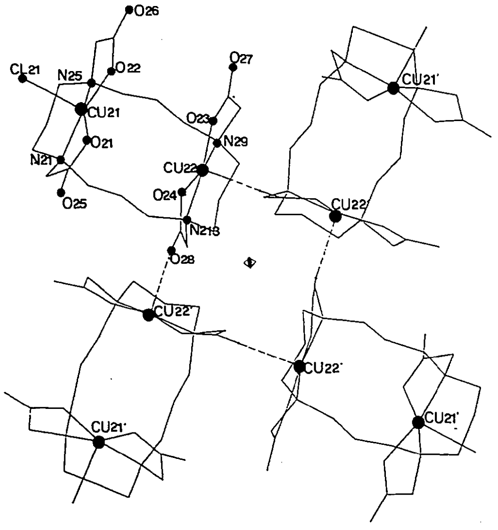

Fig. 2. View of the tetrameric moiety *B* showing the fourfold rotatory inversion axis.

Discussion. The structure consists of two similar anionic units Cu$_2L$(H$_2$O)Cl$^-$ (*A*) and [Cu$_2L$Cl]$_4^-$ (*B*) and a Cu(H$_2$O)$_6^{2+}$ counterion together with extensive solvent. Ion *A* contains two five-coordinated Cu^{2+} ions in a square pyramidal arrangement and is shown in Fig. 1. Cu(11) is bound to two N atoms of the macrocycle, two carboxylate groups and an apical chloride, Cl(11), and Cu(12) shows the same arrangement with a water molecule O(41) instead of the chloride at the apex. Unlike the anions of type *A*, which are isolated in the lattice, the anions *B* are linked by carboxylate bridges into tetramers. The subunits are related by the crystallographic fourfold rotatory inversion axis parallel to **c** (Fig. 2). The carboxylate group O(24)—C(224)—O(28) acts as a bridge between Cu(22) and the symmetry related Cu(22′). O(28‴) is the apex of the pyramid like O(41) in the first moiety.

The intramolecular metal–metal distances are Cu(11)—Cu(12) 4·903 (4) and Cu(21)—Cu(22) 4·848 (4) Å. The solvent part of the structure consists of the seven coordinated water molecules mentioned above and 19 other sites of which 14 are only partially occupied. The arrangement for all of them is reasonable for hydrogen bonds.

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Structures of the $P2_1/c$ Phases of [(CH$_3$)$_4$P]$_2$CoI$_4$ and [(CH$_3$)$_4$P]$_2$CoBr$_4$

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Abstract. Bis(tetramethylphosphonium) tetraiodocobaltate(II), [C$_4$H$_{12}$P]$_2$[CoI$_4$], $M_r = 748{\cdot}8$, monoclinic, $P2_1/c$, $a = 9{\cdot}859\,(2)$, $b = 16{\cdot}762\,(2)$, $c = 13{\cdot}716\,(1)$ Å, $\beta = 90{\cdot}05\,(1)^\circ$, $V = 2266{\cdot}6\,(6)$ Å^3, $Z = 4$, $D_x = 2{\cdot}19$ g cm^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 62{\cdot}7$ cm^{-1}, $F(000) = 1364$, $T = 293$ K, final $R = 0{\cdot}073$ for 3481 unique reflections with $F > 3\sigma(F)$. Bis(tetramethylphosphonium) tetrabromocobaltate(II), [C$_4$H$_{12}$P]$_2$[CoBr$_4$], $M_r = 560{\cdot}8$, monoclinic, $P2_1/c$, $a = 9{\cdot}489\,(2)$, $b = 15{\cdot}985\,(3)$, $c = 13{\cdot}106\,(2)$ Å, $\beta = 89{\cdot}52\,(2)^\circ$, $V = 1987{\cdot}9\,(7)$ Å^3, $Z = 4$, $D_x = 1{\cdot}87$ g cm^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 89{\cdot}7$ cm^{-1}, $F(000) = 1076$, $T = 293$ K, final $R = 0{\cdot}088$ for 2019 unique reflections with $F > 3\sigma(F)$. The two structures are of the slightly distorted β-K$_2$SO$_4$ type. [(CH$_3$)$_4$P]$_2$CoI$_4$ is virtually isostructural with the

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