H atoms were inserted in calculated positions and assigned isotropic temperature factors equal to those of their attached atoms plus 10%. After the final cycle of refinement $R = 0.065$ and $wR = 0.055$ for all 4388 reflections while $R = 0.048$ and $wR = 0.055$ for the 3345 observed reflections [$w = A + BF_o + CF_o^2 + DF_o^3$, where $A = 4.9106$, $B = -0.05777$, $C = -0.000178$, $D = 0.000017$; function minimized, $w(|F_o| - k|F_c|)^2$]; goodness of fit $S = 0.805$; maximum $\Delta/\sigma = 0.002$. The final difference Fourier map was essentially featureless with maximum and minimum residuals of 0.547 and -0.280 e Å^{-3}, respectively. Atomic scattering factors for non-H atoms were from Cromer & Mann (1968) and from Stewart, Davidson & Simpson (1965) for H atoms.* Final atomic coordinates and thermal factors are given in Table 1, bond lengths in Table 2.† The structure of the title cation (without counterions) is shown in Fig. 1, and the entire unit-cell contents are shown in Fig. 2.

Related literature. The title compound was synthesized as part of a study on ML_n compounds with trigonal bipyramidal and octahedral symmetry (Marat & Janzen, 1977, 1980; Secco, Alam, Blackburn & Janzen, 1986, and references therein). The 3:1 *mer:fac* isomer ratio observed in ^{19}F NMR solution studies (Janzen, Nguyen, Qu & Marat, 1988) could also exist in the solid state; however, intermolecular contacts [< 3.2 Å between C(33) and neighboring atoms] would not allow an F atom to occupy the area around C(33), permitting only four of the eight possible combinations. Bond distances and bond angles (deposited) were generally normal, except for an unusually short C(18)—F(18) bond.

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* Computation was carried out on the University of Manitoba Computer Services Department's Amdahl 5870 mainframe computer using locally written programs for processing and modified versions of the following programs for structure solution, refinement and calculations: *FORDAP* (A. Zalkin, unpublished); *ORFLS* (Busing, Martin & Levy, 1962); *ORFFE* (Busing, Martin & Levy, 1964); *ORTEP*II (Johnson, 1976).

† Lists of bond angles, anisotropic thermal parameters, H-atom positional and thermal parameters, bond distances and angles involving H atoms, torsion angles, and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54544 (29 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: BR0002]

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Acta Cryst. (1992). C**48**, 167–169

Structure of *trans*-Diaquabis[(2-oxopyrrolidin-1-yl)acetamide]copper(II) Perchlorate

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(*Received* 6 *July* 1990; *accepted* 18 *July* 1991)

Abstract. $[Cu(C_6H_{10}N_2O_2)_2(H_2O)_2](ClO_4)_2$, $M_r = 582.78$, monoclinic, $P2_1/c$, $a = 7.231$ (10), $b = 10.633$ (3), $c = 15.767$ (3) Å, $\beta = 103.94$ (5)°, $V = 1176.57$ Å^3, $Z = 2$, $D_x = 1.645$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 12.08$ mm^{-1}, $F(000) = 598$, room temperature, $R = 0.067$ for 1561 observed reflections. The only important structural difference between the oxopyrrolidine ligand in this Cu complex and the free molecule [Pritzkow (1983). *Cryst. Res. Technol.* **18**(8), 1069–1074] is in the orientation of the acetamide group relative to the pyrrolidine ring. In the centrosymmetric cation the CuO_6 octahedron is tetragonally distorted, with long Cu—O(H_2O) distances of 2.450 (5) Å. The perchlorate anions are disordered.

Experimental. Crystals were grown by mixing equal volumes of aqueous solutions of 0.5 *M* 2-oxo-1-

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Table 1. *Fractional atomic coordinates with e.s.d.'s in parentheses and equivalent isotropic thermal parameters* (Å^2)

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
Cu	−0.0000	0.0000	0.0000	3.01
O1	−0.7737 (5)	0.1029 (4)	0.0457 (3)	3.48
O2	−0.1503 (5)	0.0863 (4)	0.0703 (2)	3.60
O3	−0.1296 (7)	0.1149 (4)	−0.1360 (4)	4.54
N1	−0.5621 (7)	0.2531 (4)	0.1017 (3)	3.53
N2	−0.3822 (7)	0.1869 (6)	−0.0293 (3)	4.40
C1	−0.7407 (7)	0.2134 (5)	0.0689 (3)	2.89
C2	−0.8778 (9)	0.3203 (6)	0.0666 (4)	4.09
C3	−0.7479 (12)	0.4355 (6)	0.0912 (8)	5.67
C4	−0.5525 (11)	0.3861 (6)	0.1289 (6)	5.51
C5	−0.4032 (8)	0.1691 (7)	0.1222 (4)	3.70
C6	−0.3031 (8)	0.1447 (5)	0.0502 (3)	3.35
Cl	−0.2106 (3)	0.6590 (2)	0.1921 (1)	6.50
O1*PE*	−0.2811 (12)	0.6566 (7)	0.1046 (4)	8.72
O2*PE*	−0.3141 (19)	0.6138 (11)	0.2431 (6)	15.41
O2*PE*	−0.0651 (19)	0.6061 (49)	0.2076 (11)	40.95
O4*PE*	−0.1466 (51)	0.7687 (21)	0.2181 (12)	39.55

Table 2. *Bond distances* (Å), *bond angles* (°), *torsion angles* (°) *and possible non-bonded interactions with e.s.d.'s in parentheses*

O2—C6	1.240 (7)	C4—N1	1.474 (8)
N2—C6	1.326 (8)	Cu—O1	1.956 (4)
C6—C5	1.508 (8)	Cu—O2	1.958 (4)
C5—N1	1.430 (8)	Cu—O3	2.450 (5)
N1—C1	1.338 (7)		
C1—O1	1.237 (6)		
C1—C2	1.503 (8)		
C2—C3	1.535 (10)		
C3—C4	1.490 (12)		

O2—C6—N2	124.5 (5)	C2—C3—C4	106.5 (5)
O2—C6—C5	117.0 (5)	C3—C4—N1	104.4 (5)
N2—C6—C5	118.5 (5)	O1—Cu—O2	93.4 (2)
C6—C5—N1	116.2 (5)	O1—Cu—O3	98.8 (2)
C5—N1—C1	122.6 (5)	O2—Cu—O3	96.6 (2)
C5—N1—C4	123.7 (5)		
C1—N1—C4	112.9 (5)		
N1—C1—O1	120.9 (5)		
N1—C1—C2	109.9 (5)		
O1—C1—C2	129.2 (5)		
C1—C2—C3	103.7 (5)		

O2—C6—C5—N1	170. (3)	C4—N1—C1—C2	2. (9)
N2—C6—C5—N1	−9. (3)	C5—N1—C4—C3	179. (6)
C6—C5—N1—C1	91. (2)	C1—N1—C4—C3	−12. (3)
C6—C5—N1—C4	−101. (5)	N1—C1—C2—C3	8. (2)
C5—N1—C1—O1	−8. (3)	O1—C1—C2—C3	−172. (2)
C5—N1—C1—C2	172. (2)	C1—C2—C3—C4	−15. (1)
C4—N1—C1—O1	−178. (4)	N1—C4—C3—C2	16. (2)

$A\cdots B$	
O3⋯O2^{ii}	2.955 (6)
N2⋯O3^{i}	2.868 (8)
N2⋯O1*PE*^{iii}	2.952 (9)

Symmetry code: (i) x, y, z; (ii) $2-x, -y, 1-z$; (iii) $x, 0.5-y, 0.5+z$.

pyrrolidinylacetamide and 0.267 *M* $Cu(ClO_4)_2$. The mixed solution was gently stirred, filtered and left undisturbed for three weeks at 303 K. One of the crystals thereby obtained, of dimensions 1 × 0.4 × 0.2 mm, was mounted in a capillary. Data recorded on an Enraf–Nonius CAD-4 diffractometer, Mo *Kα* radiation, graphite monochromator. Cell parameters from 25 reflections ($6° < \theta < 15°$), ω scan technique. Three reflections remeasured every 2 h showed no significant variation in intensity. Lp but no absorption corrections applied. 2080 independent reflections ($\theta < 25°$), 1561 with $I > 3\sigma(I)$ (index range, h −8 to 8, k 0 to 12, l 0 to 18). Structure determined by direct methods (*MITHRIL*; Gilmore, 1983). Anisotropic full-matrix least-squares refinement of 181 parameters on F with weighting scheme $[\sigma^2(F) +$

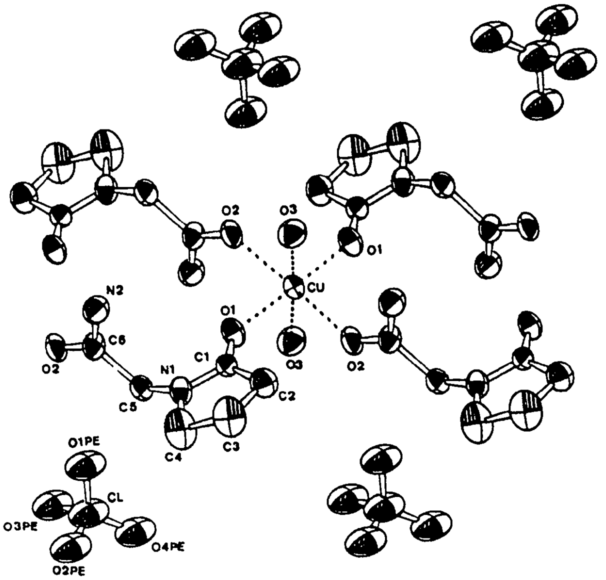

Fig. 1. Molecular structure and atom numbering. Atoms of 2-oxo-1-pyrrolidinylacetamide, copper and water are represented with thermal ellipsoids of 50% probability. Chlorine U_{ij}'s have been assigned to the four perchlorate oxygens, in order to obtain a clear figure.

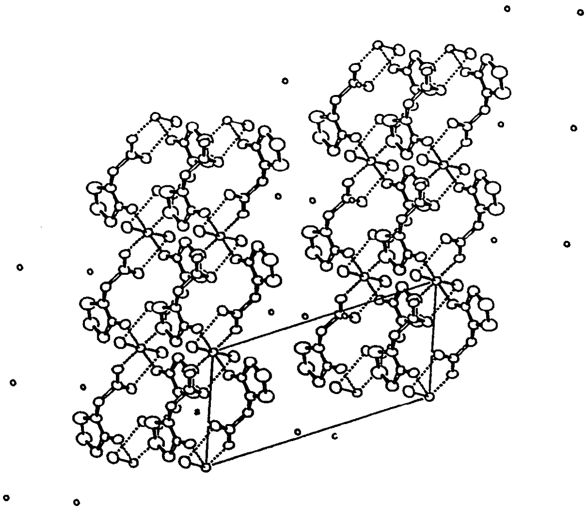

Fig. 2. Crystal packing in the *ac* plane. Broken lines indicate copper coordination.

$0.005004|F|^2]^{-1}$ (*SHELX76*; Sheldrick, 1976). f, f', f'' from *International Tables for X-ray Crystallography* (1974, Vol. IV). HC21, HC22, HC51, HC52 atoms, visible in difference syntheses, have been refined without constraints; the remaining hydrogens were included at calculated positions and refined with geometrical constraints. Final $R = 0.067$, $wR = 0.07$, max. shift/e.s.d. = 0.17, max. and min. heights in final difference Fourier synthesis: 0.96 and -1.25 e Å^{-3}. A VAX 750 computer was used.

The molecular structure with the atomic numbering is shown in Fig. 1 and the final positional and thermal parameters are listed in Table 1.* Bond lengths, bond angles and torsion angles (IUPAC–IUB Commission on Biochemical Nomenclature, 1970) are listed in Table 2. Crystal packing is shown in Fig. 2.

Related literature. The five-membered ring adopts an envelope conformation (Duax & Norton, 1975, 1984) instead of the half-chair conformation found in the pyracetam crystal (Pritzkow, 1983). The asymmetry parameter $C_s = 2.0$ (3) indicates a small distortion of the mirror symmetry.

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* Lists of structure factors, anisotropic thermal parameters, all bond lengths and angles and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54547 (45 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1992). C**48**, 169–171

Structure of Carbonyl(4-methoxy-*N*-methylbenzothiohydroxamato-*κO*,*κS*)(tricyclohexylphosphine)rhodium(I) Benzene Solvate

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(*Received* 15 *April* 1991; *accepted* 9 *July* 1991)

Abstract. $[Rh(C_9H_{10}NO_2S)(CO)\{P(C_6H_{11})_3\}].C_6H_6$, $M_r = 685.7$, triclinic, $P\bar{1}$, $a = 9.764$ (2), $b = 10.185$ (2), $c = 19.885$ (3) Å, $\alpha = 80.03$ (1), $\beta = 87.87$ (1), $\gamma = 61.94$ (1)°, $V = 1716.5$ (5) Å^3, $Z = 2$, $D_m = 1.31$ (1), $D_x = 1.33$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 6.24$ cm^{-1}, $T = 295$ K, $F(000) = 720$, final $R = 0.0503$ for 4858 observed reflections. The complex has approximate square-planar geometry about the Rh atom with the P and S atoms in a *trans* orientation. The O—Rh and S—Rh bonds form an angle of 83.4 (1)°. The least-squares planes consisting of the chelate ring atoms and *N*-substituted C atom on the one hand, and coordinated atoms, including O of CO, on the other hand, form a dihedral angle of 2.1 (5)°. The least-squares plane of the benzene C atoms forms a dihedral angle of 37.2 (5)° with the above-defined coordination plane. Bond distances are Rh—O = 2.029 (4), Rh—S = 2.306 (2), Rh—P = 2.290 (1) and Rh—C = 1.787 (6) Å.

Experimental. The substituted thiohydroxamate ligand was prepared according to methods described by Jensen & Pedersen (1961) and Jensen, Buchardt & Christophersen (1967). The precursor complex, $[Rh(C_9H_{10}NO_2S)(CO)_2]$, was obtained by adding a slight excess of ligand to $[RhCl(CO)_2]_2$ in dimethylformamide. The title complex was prepared by mixing equimolar amounts of PCy_3 and $[Rh(C_9H_{10}NO_2S)(CO)_2]$ in benzene, boiling the solution with activated carbon and evaporating the filtrate to an oily residue. This was dissolved in 70/30 (v/v) benzene/hexane which on slow evaporation at room temperature gave light-yellow tabular crystals. The density was determined by flotation in NaI solution. A crystal with dimensions 0.15 × 0.05 × 0.18 mm was used for data collection on an Enraf–Nonius CAD-4F diffractometer with graphite-monochromated Mo $K\alpha$ radiation. The $\omega/2\theta$-scan technique was used with variable scan width $\Delta\omega =$

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