

équivalents sont rassemblés dans le Tableau 1.* Les longueurs des liaisons, les angles valenciels, les distances K—O entre l'ion K⁺ et les atomes d'oxygène qui constituent son polyèdre de coordination et les angles O—K—O formés par les atomes de ce polyèdre sont rapportés dans le Tableau 2.

Littérature associée. Structure cristalline et moléculaire de l'acide pyroglutamique (oxo-5 proline) (Pattabhi & Venkatesan, 1974). Ce mémoire décrit le racémique. Structure cristalline et moléculaire de l'allo-hydroxy-4 L-proline dihydrate (Shamala, Guru Row & Venkatesan, 1976). Structure de l'acide L-pyroglutamique (van Zoeren, Oonk & Kroon, 1978). Tétra[bis(oxo-5 proline)platinate(II) de potassium] pentahydrate (Viossat, Rodier, Nguyen Huy Dung & Guillard, 1986). Diaquabis(oxo-5 proline)zinc(II) (Rodier, Céolin, Plat & Zumbihl, 1990). Bis(oxo-5 proline)platinate(II) de sodium dihydrate (Viossat, Khodadad & Rodier, 1990). Structure cristalline et moléculaire du diméthylsulfoxyde (Thomas, Shoemaker & Ericks, 1966). Trichloro(diméthylsulfoxyde)platinate(II) de bis(propanediamine-1,3-*N,N'*-platine(II) (2/1) (Viossat, Toffoli, Khodadad & Rodier, 1988). Structure cristalline et moléculaire du bis[μ -(oxo-5 proline-*N*¹,*O*⁵)]-bis[chloro(diméthyl-

sulfoxyde)platine(II)] trihydrate (Viossat, Khodadad, Rodier & Guillard, 1990).

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Structure of Chloro[(1,2,3,4,5- η)-cyclooctadienyl]hydridobis(triphenylphosphine)-rhenium Acetone Solvate

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Abstract. [Re(Cl)(H)(C₈H₁₁)(C₁₈H₁₅P)₂].C₃H₆O, *M*_r = 912.5, monoclinic, *P*2₁/*n*, *a* = 20.287 (3), *b* = 9.866 (3), *c* = 22.194 (5) Å, β = 113.70 (2)°, *V* = 4067 (3) Å³, *Z* = 4, *D*_x = 1.490 g cm⁻³, λ (Mo *K* α) = 0.71073 Å, μ = 32.05 cm⁻¹, *F*(000) = 1840, *T* = 295 K, final *R* = 0.039 for 2361 unique observed reflections. The Re, which has no imposed symmetry,

is coordinated to one Cl, one H, two P and five C of the cyclooctadienyl. The five metal-bonded C atoms are planar within ± 0.05 (2) Å and parallel to the P(1)—P(2)—Cl plane [dihedral angle 2(1)°]. The Re lies 1.60 (1) and 1.39 (1) Å from the C₅ plane and the P₂Cl plane respectively. The C₈ ring takes up a diplanar conformation with C(55), C(56) and C(57)

at 1.19 (2), 1.99 (2) and 1.22 (2) Å above the C₅ plane; the C—C distance between coordinated C averages 1.41 (2) Å, significantly shorter than that for the uncoordinated C atoms [1.51 (2) Å]. No contact is observed between the [ReH(PPh₃)₂Cl(η⁵-C₈H₁₁)] molecule and the acetone solvent, all contacts being greater than 3.55 (3) Å [O(100)—C(29)].

Experimental. Red-brown crystals obtained by recrystallization from acetone-dichloromethane; approximate dimensions 0.20 × 0.20 × 0.40 mm; Nonius CAD-4 diffractometer, graphite-monochromated Mo Kα radiation, ω/2θ scan technique; all parameters from least-squares fit of 25 reflections with 8 < θ < 12°; three standard reflections monitored every hour, decay of 11.1% in 62 h, linearly corrected; 5502 reflections measured in the ranges 1 < θ < 22°, 0 ≤ h ≤ 20, 0 ≤ k ≤ 10, -22 ≤ l ≤ 22, after averaging 4966 unique, 2361 with I ≥ 3σ(I); Lorentz-polarization correction and absorption correction via ψ scans (North, Phillips & Mathews, 1968), max., min. transmissions 0.999, 0.8937; structure solution via Patterson function for Re, ΔF syntheses for the remaining atoms; full-matrix least-squares refinement on F with anisotropic thermal factors for Re and Cl atoms; H atoms of phenyl and cyclooctadienyl groups introduced at calculated positions (C—H = 0.95 Å, B = 1.3B_C), not refined but constrained to ride on their C atoms; H (hydride) not located in the final ΔF map; R = 0.039, wR = 0.048 (w = 1), S = 6.08, (Δ/σ)_{max} = 0.04,

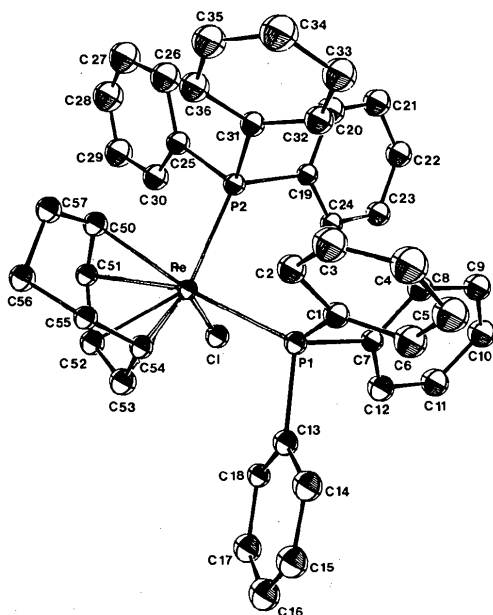


Fig. 1. ORTEP (Johnson, 1976) drawing of the molecule with the atomic numbering scheme.

Table 1. Atomic coordinates and equivalent isotropic thermal parameters

	x	y	z	B(Å ²)
Re	0.23868 (3)	0.03287 (8)	0.02027 (3)	2.17 (1)*
Cl	0.3691 (2)	0.0946 (5)	0.0624 (2)	3.4 (1)*
C(50)	0.1819 (8)	0.014 (2)	0.0882 (7)	3.3 (4)
C(51)	0.2363 (8)	0.115 (2)	0.1125 (8)	2.9 (4)
C(52)	0.2409 (9)	0.227 (2)	0.0752 (8)	3.3 (4)
C(53)	0.2040 (9)	0.252 (2)	0.0064 (8)	3.6 (4)
C(54)	0.1472 (8)	0.167 (2)	-0.0335 (7)	2.6 (3)
C(55)	0.0725 (8)	0.162 (2)	-0.0340 (8)	2.8 (4)
C(56)	0.0721 (9)	0.162 (2)	0.0348 (8)	3.5 (4)
C(57)	0.1026 (8)	0.033 (2)	0.0706 (7)	3.5 (3)
F(1)	0.2436 (2)	0.0170 (5)	-0.0879 (2)	2.6 (1)*
F(2)	0.2747 (2)	-0.1998 (5)	0.0568 (2)	2.8 (1)*
C(1)	0.1667 (8)	-0.069 (2)	-0.1545 (7)	2.7 (4)
C(2)	0.1043 (9)	-0.101 (2)	-0.1455 (9)	3.8 (4)
C(3)	0.047 (1)	-0.162 (2)	-0.199 (1)	5.0 (5)
C(4)	0.051 (1)	-0.191 (2)	-0.257 (1)	5.6 (5)
C(5)	0.114 (1)	-0.158 (2)	-0.264 (1)	5.6 (5)
C(6)	0.172 (1)	-0.094 (2)	-0.213 (1)	5.0 (5)
C(7)	0.3209 (8)	-0.062 (2)	-0.0973 (7)	2.5 (3)
C(8)	0.3192 (9)	-0.198 (2)	-0.1128 (8)	3.1 (4)
C(9)	0.3768 (9)	-0.258 (2)	-0.1213 (9)	4.0 (4)
C(10)	0.439 (1)	-0.187 (2)	-0.1112 (9)	4.1 (4)
C(11)	0.4400 (9)	-0.053 (2)	-0.0958 (9)	4.5 (4)
C(12)	0.3827 (9)	0.012 (2)	-0.0884 (8)	4.0 (4)
C(13)	0.2430 (8)	0.188 (2)	-0.1240 (8)	2.8 (4)
C(14)	0.192 (1)	0.225 (2)	-0.1862 (9)	4.1 (4)
C(15)	0.191 (1)	0.356 (2)	-0.2082 (9)	4.6 (5)
C(16)	0.2424 (9)	0.451 (2)	-0.1720 (8)	4.2 (4)
C(17)	0.293 (1)	0.411 (2)	-0.1093 (9)	4.1 (4)
C(18)	0.2928 (8)	0.281 (2)	-0.0863 (8)	3.1 (4)
C(19)	0.3503 (8)	-0.299 (2)	0.0527 (8)	2.6 (3)
C(20)	0.3521 (9)	-0.439 (2)	0.0640 (9)	4.3 (4)
C(21)	0.4113 (9)	-0.513 (2)	0.0685 (8)	4.5 (4)
C(22)	0.4672 (9)	-0.451 (2)	0.0612 (8)	4.3 (4)
C(23)	0.4675 (9)	-0.314 (2)	0.0496 (8)	3.7 (4)
C(24)	0.4063 (9)	-0.239 (2)	0.0458 (8)	3.2 (4)
C(25)	0.3009 (9)	-0.224 (2)	0.1466 (8)	3.3 (4)
C(26)	0.274 (1)	-0.330 (3)	0.172 (1)	5.8 (6)
C(27)	0.297 (1)	-0.337 (3)	0.242 (1)	8.1 (7)
C(28)	0.342 (1)	-0.246 (3)	0.278 (1)	7.5 (7)
C(29)	0.371 (1)	-0.146 (2)	0.257 (1)	5.6 (5)
C(30)	0.349 (1)	-0.137 (2)	0.1866 (9)	4.3 (5)
C(31)	0.1974 (9)	-0.314 (2)	0.0151 (8)	3.2 (4)
C(32)	0.1907 (9)	-0.378 (2)	-0.0412 (9)	3.8 (4)
C(33)	0.129 (1)	-0.449 (3)	-0.0799 (9)	5.3 (5)
C(34)	0.074 (1)	-0.455 (3)	-0.058 (1)	6.1 (5)
C(35)	0.079 (1)	-0.391 (2)	-0.003 (1)	5.5 (5)
C(36)	0.141 (1)	-0.319 (2)	0.035 (1)	4.9 (5)
O(100)	0.074 (1)	0.685 (3)	0.180 (1)	16 (1)
C(100)	0.058 (2)	0.751 (5)	0.225 (2)	17 (2)
C(101)	0.004 (2)	0.690 (4)	0.237 (2)	13 (1)
C(102)	0.114 (2)	0.836 (4)	0.242 (2)	14 (1)

$$* B_{eq} = (4/3) \sum_i \sum_j B_{ij} a_i \cdot a_j.$$

largest peak in final ΔF map 0.92 e Å⁻³; neutral-atom scattering factors including anomalous dispersion from *International Tables for X-ray Crystallography* (1974); calculations performed on a MicroVAX II computer with the Enraf-Nonius *Structure Determination Package* (Frenz, 1986).

The structure of the title compound and the numbering scheme are shown in Fig. 1. Positional parameters and equivalent values of the anisotropic temperature factors are given in Table 1,* and bond lengths and angles in Table 2.

* Lists of structure factors, anisotropic thermal parameters, bond lengths and angles, and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52878 (19 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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

Re environment			
Re—P(1)	2.448 (2)	Re—C(53)	2.254 (8)
Re—P(2)	2.446 (2)	Re—C(54)	2.202 (7)
Re—Cl	2.502 (2)	Re—C(55)	3.344 (7)
Re—C(50)	2.244 (7)	Re—C(56)	3.744 (7)
Re—C(51)	2.219 (7)	Re—C(57)	3.369 (6)
Re—C(52)	2.264 (8)		
P(1)—Re—P(2)	98.09 (7)	Cl—Re—P(2)	88.53 (6)
Cl—Re—P(1)	85.90 (6)		
Cyclooctadienyl ring			
C(50)—C(51)	1.42 (1)	C(54)—C(55)	1.51 (1)
C(51)—C(52)	1.41 (1)	C(55)—C(56)	1.53 (1)
C(52)—C(53)	1.43 (1)	C(56)—C(57)	1.49 (1)
C(53)—C(54)	1.41 (1)	C(57)—C(50)	1.51 (9)
C(51)—C(50)—C(57)	126.4 (7)	C(53)—C(54)—C(55)	126.1 (6)
C(50)—C(51)—C(52)	123.6 (6)	C(54)—C(55)—C(56)	113.5 (6)
C(51)—C(52)—C(53)	129.2 (7)	C(55)—C(56)—C(57)	111.5 (6)
C(52)—C(53)—C(54)	121.2 (7)	C(56)—C(57)—C(50)	114.7 (7)
Triphenylphosphine group			
P(1)—C(1)	1.864 (7)	P(2)—C(19)	1.851 (7)
P(1)—C(7)	1.836 (6)	P(2)—C(25)	1.860 (8)
P(1)—C(13)	1.862 (8)	P(2)—C(31)	1.849 (8)
C(1)—P(1)—C(7)	101.5 (3)	C(19)—P(2)—C(25)	95.7 (3)
C(7)—P(1)—C(13)	101.1 (3)	C(19)—P(2)—C(31)	102.0 (3)
C(1)—P(1)—C(13)	102.1 (3)	C(25)—P(2)—C(31)	106.2 (3)

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Structure of (Histamine.2H⁺)(ZnCl₄)

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Abstract. 5-(2-Ammonioethyl)imidazolium tetrachlorozincate, [C₅H₁₁N₃][ZnCl₄], $M_r = 320.3$, orthorhombic, $P2_1nb$, $a = 7.343$ (6), $b = 7.619$ (2), $c = 21.455$ (12) Å, $V = 1200$ (1) Å³, $Z = 4$, $D_x = 1.773$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 29.58$ cm⁻¹, $F(000) = 640$, $T = 293$ K, $R = 6.4\%$ for 738 observed reflections. The granular matrix which stores histamine in the mast cell contains zinc cations, but the nature of the cellular interaction between zinc and histamine is unknown. In the complex formed by zinc chloride and histamine.2H⁺, zinc is tetrahedrally bound to four chloride atoms which are hydrogen bonded to the 5-(2-aminoethyl)imidazole molecule (protonated at N4 of the imidazole ring and N8 of the side chain) *via* the protons of the imidazole nitrogen atoms, N2 and N4.

Experimental. Clear plate with dimensions 0.2 × 0.2 × 0.1 mm; prepared from 0.3682 g (2 mmol) of his-

Related literature. Crystals of the title compound were formed by the reaction of HCl with the monohydridocyclooctatriene complex ReH(PPh₃)₂ ($\eta^6\text{-C}_8\text{H}_{10}$), which was itself obtained by treatment of ReH₇(PPh₃)₂ with cyclooctatetraene (Boydell, 1984). This structure is to be compared with the structure of ReH₂(PMe₂Ph)₂($\eta^5\text{-C}_8\text{H}_{11}$) (Trimarchi, Green, Huffman & Gaulton, 1985); the Re coordination is essentially the same with one H atom replaced by one Cl atom.

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tamine dissolved in 60 mL absolute EtOH containing 0.1110 g (1 mmol) anhydrous CaCl₂ and 0.1363 g (1 mmol) ZnCl₂; orthorhombic, space group $P2_1nb$ (No. 33). Intensities collected with a Syntex $P3m$ diffractometer; graphite monochromator; $\theta/2\theta$ scan, $2\theta_{\text{max}} = 45^\circ$; $(\sin\theta/\lambda)_{\text{max}} = 0.538$ Å⁻¹; variable scan rate; three standard reflections remeasured after every 100 reflections, variation 5%; 15 reflections with $10.69 < 2\theta < 22.97^\circ$ used for refinement of lattice parameters; index range $0 \leq h \leq 10$, $0 \leq k \leq 10$, $0 \leq l \leq 22$; 1092 reflections measured, 738 unique reflections observed with $I > 3.0\sigma(I)$, 354 unobserved; L_p corrected (absorption correction not applied); solution for non-hydrogen positions, *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980); hydrogen atoms located from difference Fourier synthesis, hydrogen positional and isotropic thermal parameters fixed ($U = 0.03$); final cycles of refinement including anisotropic thermal parameters for non-hydrogen atoms (function minimized $\sum w(|F_o| -$

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