

Experimental. Reaction of $\{[\text{Ru}(\text{C}_8\text{H}_{12})(\text{O}_2\text{CCF}_3)_2(\mu\text{-O}_2\text{CCF}_3)_2(\mu\text{-OH}_2)]\}_2$ [(II), $\text{C}_8\text{H}_{12} = 1,5$ -cyclooctadiene; Albers *et al.* (1984)] with 4 mol equivalents of PMe_2Ph in H_2furan at 298 K yielded title complex (I). Long yellow irregular-hexagon prisms obtained by recrystallization from acetone/ethanol solution. Crystal: $0.32 \times 0.20 \times 0.15$ mm cut from longer prism; Philips PW 1100 four-circle diffractometer, graphite-crystal-monochromatized $\text{MoK}\alpha$ radiation; lattice parameters: 25 reflections, least-squares refinement; ω - 2θ scan mode, scan width 1.20° in ω , scan speed $0.048^\circ \text{ s}^{-1}$ in ω , 12.5 s stationary background at each end of scan; 7351 reflections measured in quadrant $\pm h, +k, +l$ with $3 \leq \theta \leq 23^\circ$, 6797 unique reflections, $R_{\text{int}} = 0.034$; 3 standards measured every 60 min showed no significant deviations from mean intensities; no absorption correction; 4625 reflections [$F_o \geq 4\sigma(F_o)$], index range $h \pm 20, k 0-19, l 0-20$, used in analysis; structure solved by Patterson and Fourier methods and refined by least squares with $\sigma^{-2}(F_o)$ weights using *SHELX* (Sheldrick, 1978); two orientations observed for each of the four disordered CF_3 groups, site-occupation factors (s.o.f.'s) refined for each set of F atoms but with s.o.f.'s for the two orientations

Table 1. Fractional coordinates ($\times 10^4, \times 10^5$ for Ru, $\times 10^3$ for H) and equivalent isotropic temperature factors ($\text{\AA}^2 \times 10^3, \text{\AA}^2 \times 10^4$ for Ru) for (I)

$$U_{\text{eq}} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{eq}
Ru(1)	22689 (4)	-2733 (4)	38104 (4)	336 (4)
Ru(2)	24533 (4)	3164 (5)	59185 (4)	374 (4)
O(1)	3031 (4)	48 (3)	5094 (3)	39 (4)
H(1A)	333 (6)	-50 (3)	530 (6)	129 (33)*
H(1B)	324 (6)	62 (3)	504 (6)	129 (33)*
P(1)	1371 (2)	-685 (2)	2587 (1)	43 (1)
C(111)	1139 (5)	-48 (6)	1718 (5)	46 (6)
C(112)	826 (7)	705 (7)	1702 (7)	73 (8)
C(113)	664 (11)	1211 (10)	1065 (10)	95 (12)
C(114)	764 (12)	979 (13)	423 (12)	95 (16)
C(115)	1027 (9)	239 (13)	407 (9)	89 (13)
C(116)	1228 (6)	-302 (9)	1053 (7)	75 (9)
C(12)	1562 (7)	-1671 (6)	2299 (6)	75 (8)
C(13)	359 (6)	-819 (7)	2548 (6)	71 (8)
P(2)	3101 (1)	336 (2)	3365 (1)	39 (1)
C(211)	3278 (5)	-125 (6)	2556 (5)	43 (6)
C(212)	3149 (6)	311 (8)	1855 (5)	60 (7)
C(213)	3314 (8)	-57 (10)	1276 (8)	81 (10)
C(214)	3568 (9)	-818 (10)	1346 (9)	81 (13)
C(215)	3721 (9)	-1249 (8)	2034 (9)	84 (11)
C(216)	3554 (8)	-895 (7)	2630 (7)	71 (9)
C(22)	2882 (6)	1373 (5)	3084 (6)	65 (7)
C(23)	4148 (5)	386 (7)	4115 (5)	61 (7)
P(3)	1695 (2)	691 (2)	6552 (2)	45 (2)
C(311)	1730 (5)	175 (6)	7439 (5)	43 (6)
C(312)	1908 (6)	580 (7)	8154 (6)	55 (7)
C(313)	1896 (7)	167 (10)	8800 (7)	71 (9)
C(314)	1696 (10)	-620 (10)	8736 (10)	83 (13)
C(315)	1535 (8)	-1014 (8)	8039 (9)	88 (11)
C(316)	1532 (7)	-625 (7)	7381 (7)	69 (8)
C(32)	1756 (7)	1738 (6)	6789 (7)	79 (9)
C(33)	626 (6)	561 (7)	5880 (6)	73 (8)
P(4)	3456 (2)	-237 (2)	6997 (1)	44 (1)
C(411)	3713 (5)	191 (6)	7993 (5)	47 (6)
C(412)	3878 (6)	988 (7)	8103 (7)	66 (7)
C(413)	4079 (8)	1329 (8)	8849 (9)	82 (9)
C(414)	4129 (8)	892 (10)	9468 (8)	81 (11)
C(415)	3988 (8)	82 (10)	9381 (7)	82 (10)
C(416)	3765 (6)	-273 (7)	8633 (6)	65 (7)
C(42)	3367 (6)	-1309 (5)	7126 (6)	63 (7)
C(43)	4445 (6)	-186 (7)	6977 (6)	74 (8)

Table 1 (cont.)

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{eq}
O(51)	2888 (4)	-1364 (3)	3968 (4)	43 (4)
O(52)	3725 (5)	-1302 (5)	5269 (4)	66 (5)
C(51)	3428 (7)	-1607 (6)	4613 (7)	49 (7)
C(52)	3735 (11)	-2470 (9)	4535 (8)	82 (10)
F(51A)	3965 (10)	-2521 (9)	3993 (10)	112 (5)*
F(51B)	3360 (10)	-2777 (9)	3805 (10)	99 (5)*
F(52A)	3131 (10)	-3015 (9)	4413 (10)	117 (5)*
F(52B)	3722 (13)	-2953 (10)	5043 (12)	113 (6)*
F(53A)	4279 (10)	-2733 (9)	5247 (9)	114 (5)*
F(53B)	4549 (10)	-2376 (9)	4706 (10)	102 (5)*
O(61)	3038 (4)	1433 (3)	6183 (4)	48 (4)
O(62)	3650 (6)	1446 (5)	5385 (5)	74 (7)
C(61)	3433 (8)	1724 (7)	5858 (7)	63 (8)
C(62)	3741 (12)	2629 (10)	6169 (11)	92 (15)
F(61A)	4295 (9)	2834 (8)	6013 (9)	123 (4)*
F(61B)	3464 (15)	2903 (13)	6633 (16)	88 (7)*
F(62A)	3997 (8)	2692 (7)	6940 (8)	118 (4)*
F(62B)	3514 (20)	3060 (16)	5504 (18)	118 (9)*
F(63A)	3112 (10)	3082 (10)	5736 (11)	168 (7)*
F(63B)	4687 (16)	2546 (16)	6431 (17)	106 (9)*
O(71)	1618 (4)	790 (3)	3672 (3)	41 (4)
O(72)	1546 (4)	943 (4)	4854 (4)	50 (4)
C(71)	1425 (5)	1117 (5)	4169 (5)	37 (5)
C(72)	980 (8)	1926 (8)	3875 (7)	68 (9)
F(71A)	617 (8)	2005 (7)	3090 (7)	82 (3)*
F(71B)	1182 (10)	2330 (9)	3384 (10)	90 (5)*
F(72A)	370 (9)	2001 (8)	4078 (9)	105 (4)*
F(72B)	891 (10)	2373 (9)	4390 (9)	86 (4)*
F(73A)	1455 (10)	2546 (10)	4181 (10)	132 (6)*
F(73B)	148 (12)	1684 (11)	3386 (13)	132 (6)*
O(81)	1554 (4)	-948 (4)	4314 (4)	46 (4)
O(82)	1860 (4)	-797 (3)	5634 (4)	43 (4)
C(81)	1603 (5)	-1144 (5)	4978 (6)	35 (6)
C(82)	1289 (8)	-2006 (8)	5014 (7)	71 (8)
F(81A)	962 (7)	-2056 (6)	5521 (7)	87 (3)*
F(81B)	1329 (16)	-2499 (14)	4466 (16)	101 (7)*
F(82A)	1898 (7)	-2503 (6)	5230 (7)	101 (3)*
F(82B)	552 (18)	-2022 (15)	4952 (19)	120 (9)*
F(83A)	730 (7)	-2241 (6)	4293 (6)	93 (3)*
F(83B)	1714 (16)	-2374 (14)	5700 (15)	105 (8)*

* Isotropic temperature factor.

of each CF_3 group constrained to sum to 1.0 and a common isotropic temperature factor for the six partial F atoms of each group, after refinement of the s.o.f.'s these were fixed and individual isotropic temperature factors refined for the partial F atoms; H atoms of aqua ligand located on difference map and refined with $d(\text{O}-\text{H})$ constrained to 1.04\AA , all other H atoms added in calculated positions [$d(\text{C}-\text{H}) = 0.95 \text{\AA}$ (Churchill, 1973)]; anisotropic temperature factors for all non-F and H atoms, separate common isotropic temperature factors for phenyl and methyl H atoms in ligands coordinated to each Ru atom; least-squares refinement, $\sum w(\Delta F)^2$ minimized, $w = \sigma^{-2}(F)$, with the matrix blocked so that the parameters for the Ru atoms and the aqua ligand refined in every cycle and those for the phosphine ligands on each Ru atom, the terminal carboxylato ligands and the bridging carboxylato ligands refined in separate cycles, converged to give $R = 0.0643$ and $wR = 0.0564$; reductions in wR significant at the 99.5% level (Hamilton, 1965); complex neutral-atom scattering factors used for all atoms (Cromer & Waber, 1974); in the final cycles 603 parameters varied, comprising 243 positional parameters, 330 anisotropic temperature-factor components, 24 isotropic temperature factors, 5 common isotropic temperature factors and one overall scale, $\Delta\rho + 1.7$ to $-0.5 e \text{\AA}^{-3}$ (largest positive $\Delta\rho$ close to Ru atoms), max. Δ/σ in final least-squares cycle 1.66.

Discussion. The final atomic parameters are given in Table 1.* Bond lengths and angles are given in Table 2. Fig. 1 is a perspective view of the complex showing the atom numbering scheme; Fig. 2 shows the unit-cell contents.

* Lists of structure factors, anisotropic thermal parameters and calculated H-atom coordinates and a full list of bond distances and angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42460 (38 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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

Ru(1)—O(1)	2.232 (5)	Ru(1)—P(1)	2.249 (2)
Ru(1)—P(2)	2.253 (3)	Ru(1)—O(51)	2.092 (6)
Ru(1)—O(71)	2.084 (6)	Ru(1)—O(81)	2.204 (7)
Ru(2)—O(1)	2.235 (8)	Ru(2)—P(3)	2.248 (3)
Ru(2)—P(4)	2.238 (2)	Ru(2)—O(61)	2.094 (6)
Ru(2)—O(72)	2.208 (5)	Ru(2)—O(82)	2.096 (6)
O(1)—H(1A)	1.04 (6)	O(1)—H(1B)	1.04 (7)
P(1)—C(111)	1.813 (10)	P(1)—C(12)	1.805 (11)
P(1)—C(13)	1.825 (12)	P(2)—C(211)	1.825 (11)
P(2)—C(22)	1.797 (9)	P(2)—C(23)	1.811 (8)
P(3)—C(311)	1.823 (11)	P(3)—C(32)	1.790 (10)
P(3)—C(33)	1.814 (9)	P(4)—C(411)	1.834 (10)
P(4)—C(42)	1.818 (9)	P(4)—C(43)	1.818 (12)
O(51)—C(51)	1.239 (11)	O(52)—C(51)	1.206 (13)
C(51)—C(52)	1.57 (2)	O(61)—C(61)	1.22 (2)
O(62)—C(61)	1.20 (2)	C(61)—C(62)	1.62 (2)
O(71)—C(71)	1.241 (13)	O(72)—C(71)	1.220 (12)
C(71)—C(72)	1.55 (2)	O(81)—C(81)	1.232 (13)
O(82)—C(81)	1.236 (12)	C(81)—C(82)	1.56 (2)
O(1)—Ru(1)—P(1)	171.2 (2)	O(1)—Ru(1)—O(51)	94.0 (2)
P(1)—Ru(1)—P(2)	94.8 (1)	O(1)—Ru(1)—O(81)	90.6 (2)
P(1)—Ru(1)—O(51)	89.4 (2)	P(2)—Ru(1)—O(51)	92.0 (2)
O(1)—Ru(1)—O(71)	89.2 (2)	P(1)—Ru(1)—O(71)	90.4 (2)
P(2)—Ru(1)—O(71)	90.5 (2)	O(51)—Ru(1)—O(71)	177.5 (3)
O(1)—Ru(1)—O(81)	83.4 (2)	P(1)—Ru(1)—O(81)	87.8 (2)
P(2)—Ru(1)—O(81)	174.4 (2)	O(51)—Ru(1)—O(81)	83.0 (3)
O(71)—Ru(1)—O(81)	94.5 (3)	O(1)—Ru(2)—P(3)	169.6 (1)
O(1)—Ru(2)—P(4)	94.6 (2)	P(3)—Ru(2)—P(4)	95.8 (1)
O(1)—Ru(2)—O(61)	89.7 (3)	P(3)—Ru(2)—O(61)	89.8 (2)
P(4)—Ru(2)—O(61)	91.2 (2)	O(1)—Ru(2)—O(72)	83.1 (2)
P(4)—Ru(2)—O(72)	86.5 (2)	P(4)—Ru(2)—O(72)	174.8 (2)
O(61)—Ru(2)—O(72)	84.1 (2)	O(1)—Ru(2)—O(82)	89.7 (3)
P(3)—Ru(2)—O(82)	90.6 (2)	P(4)—Ru(2)—O(82)	89.6 (2)
O(61)—Ru(2)—O(82)	179.1 (2)	O(72)—Ru(2)—O(82)	95.1 (2)
Ru(1)—O(1)—Ru(2)	120.7 (3)	Ru(1)—O(1)—H(1A)	100 (5)
Ru(2)—O(1)—H(1A)	104 (7)	Ru(1)—O(1)—H(1B)	102 (6)
Ru(2)—O(1)—H(1B)	100 (7)	H(1A)—O(1)—H(1B)	133 (8)
Ru(1)—P(1)—C(111)	120.0 (3)	Ru(1)—P(1)—C(12)	115.1 (3)
C(111)—P(1)—C(12)	105.8 (5)	Ru(1)—P(1)—C(13)	111.2 (3)
C(111)—P(1)—C(13)	101.0 (5)	C(12)—P(1)—C(13)	101.3 (6)
P(1)—C(111)—C(112)	119.4 (3)	P(1)—C(111)—C(116)	122.5 (9)
Ru(1)—P(2)—C(211)	119.4 (3)	Ru(1)—P(2)—C(22)	115.9 (4)
C(211)—P(2)—C(22)	105.4 (5)	Ru(1)—P(2)—C(23)	113.1 (4)
C(211)—P(2)—C(23)	98.4 (5)	C(22)—P(2)—C(23)	101.9 (5)
P(2)—C(211)—C(212)	120.9 (8)	P(2)—C(211)—C(216)	120.2 (9)
Ru(2)—P(3)—C(311)	122.9 (4)	Ru(2)—P(3)—C(32)	114.2 (5)
C(311)—P(3)—C(32)	105.4 (6)	Ru(2)—P(3)—C(33)	109.3 (4)
C(311)—P(3)—C(33)	99.8 (5)	C(32)—P(3)—C(33)	102.6 (5)
P(3)—C(311)—C(312)	121.7 (8)	P(3)—C(311)—C(316)	118.8 (8)
Ru(2)—P(4)—C(411)	119.2 (3)	Ru(2)—P(4)—C(42)	115.9 (3)
C(411)—P(4)—C(42)	104.5 (5)	Ru(2)—P(4)—C(43)	113.4 (4)
C(411)—P(4)—C(43)	100.1 (5)	C(42)—P(4)—C(43)	101.2 (6)
P(4)—C(411)—C(412)	118.4 (9)	P(4)—C(411)—C(416)	122.2 (8)
Ru(1)—O(51)—C(51)	125.1 (7)	O(51)—C(51)—O(52)	130.7 (10)
O(51)—C(51)—C(52)	112.2 (9)	O(52)—C(51)—C(52)	117.0 (9)
Ru(2)—O(61)—C(61)	126.0 (7)	O(61)—C(61)—O(62)	130.9 (11)
O(61)—C(61)—C(62)	112.5 (13)	O(62)—C(61)—C(62)	116.5 (14)
Ru(1)—O(71)—C(71)	127.6 (5)	Ru(2)—O(72)—C(71)	139.9 (7)
O(71)—C(71)—O(72)	132.7 (8)	O(71)—C(71)—C(72)	113.3 (9)
O(72)—C(71)—C(72)	113.9 (10)	Ru(1)—O(81)—C(81)	138.6 (5)
Ru(2)—O(82)—C(81)	126.6 (7)	O(81)—C(81)—O(82)	135.1 (9)
O(81)—C(81)—C(82)	114.6 (9)	O(82)—C(81)—C(82)	112.4 (10)

The structure consists of isolated molecules occupying general positions in the unit cell. The complex is dinuclear with the two ruthenium atoms bridged by two trifluoroacetato ligands and an aqua ligand. The distorted octahedral coordination geometry of each ruthenium atom is completed by two PMe_2Ph ligands and a unidentate trifluoroacetato ligand. On the basis of the effective atomic number (EAN) rule a formal metal-metal bond in this complex is precluded. This is supported by the relatively long $\text{Ru}\cdots\text{Ru}$ internuclear distance. A notable feature of the structure is the strong intramolecular hydrogen bonding between the non-coordinated oxygen atoms of the unidentate trifluoroacetato ligands and the bridging aqua ligand, with $\text{O}(\text{trifluoroacetato})\cdots\text{H}(\text{aqua})$ distances of 1.54 (7) and 1.57 (8) Å and $\text{O}(\text{trifluoroacetato})\cdots\text{O}(\text{aqua})$ distances of 2.53 (1) and 2.54 (1) Å.

The structure of the $\{\text{Ru}_2(\text{O}_2\text{C}_2\text{F}_3)_2(\mu\text{-O}_2\text{C}_2\text{F}_3)_2(\mu\text{-OH}_2)\}$ core of the complex is similar to those of complex (II) (Albers *et al.*, 1984) and a series of dinuclear Ni complexes, for example: $\{[\text{Ni}(\text{Me}_2\text{NC}_2\text{H}_4\text{NMe}_2)(\text{O}_2\text{C}_2\text{F}_3)_2(\mu\text{-O}_2\text{C}_2\text{F}_3)_2(\mu\text{-OH}_2)]\}$ (Ahlgren & Turpeinen, 1982), in which there is also strong intramolecular hydrogen bonding between the non-coordinated oxygen atom of the unidentate

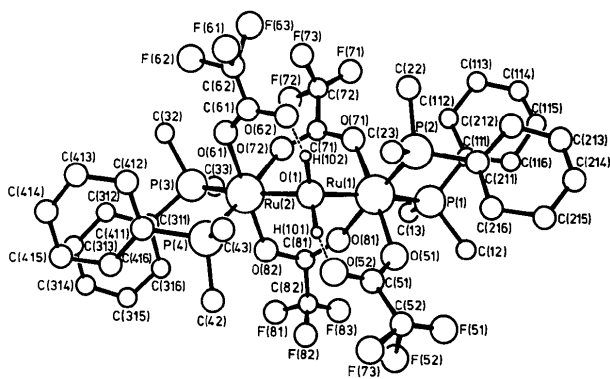


Fig. 1. A perspective view of (I) showing the atom numbering scheme. Only the major orientation of each disordered CF_3 group is shown.

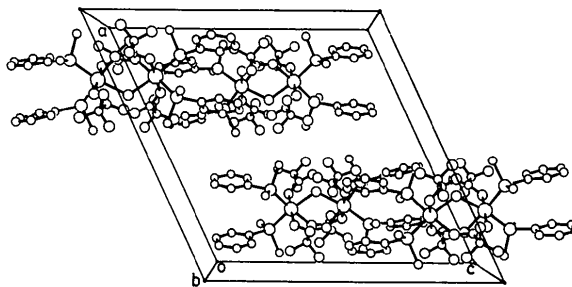


Fig. 2. The unit cell viewed perpendicular to the ac plane.

carboxylato ligands and the bridging aqua ligands. The arrangement of the bridging ligands is also similar to that in the hemerythrin model complexes [$\{\text{Fe}(\text{pz}_3\text{-BH})\}_2(\mu\text{-X})(\mu\text{-O}_2\text{CR})_2\}^n$ [pz = pyrazolyl, X = O, R = H or CH₃, n = 0; Armstrong, Spool, Papefthymiou, Frankel & Lippard (1984)] where the Fe^{III} centres are bridged by an oxo and two carboxylato ligands and a protonated analogue (X = OH, R = CH₃, n = 1+; Armstrong & Lippard, 1984), which has a bridging hydroxo ligand.

The substitution of the two cyclooctadiene ligands in (II) by four PMe₂Ph ligands to give (I) results, as expected, in a marked increase in the Ru–ligand bond lengths *trans* to these phosphine ligands. In (II), the bridging trifluoroacetato ligands are slightly asymmetric with an average Ru–O bond *trans* to a π olefin of 2.128 (5) Å and *trans* to a unidentate trifluoroacetato ligand of 2.088 (5) Å, whereas in (I) the asymmetry is much greater with an average Ru–O bond *trans* to a phosphine ligand of 2.205 (7) Å and *trans* to a unidentate trifluoroacetato ligand of 2.090 (6) Å. The Ru–O(aqua) bond lengths (also *trans* to phosphine ligands) are also longer in complex (I) [average 2.234 (5) Å] compared with (II) [average 2.146 (5) Å]; the aqua bridges are, however, symmetrical within experimental error in both complexes. Commensurate with the increase in some of the bridging Ru–O bond lengths, the non-bonded Ru...Ru distance increases from 3.733 (1) in (II) to 3.881 (1) Å in (I). It is interesting to note here that, despite the lengthening of the Ru–(bridging ligand) bonds in (I),

(II) is significantly more susceptible to breakdown of the $\{\text{Ru}_2(\text{O}_2\text{C}_2\text{F}_3)_2(\mu\text{-O}_2\text{C}_2\text{F}_3)_2(\mu\text{-OH}_2)\}$ core with attack by nucleophiles (e.g. phosphines, amines and isonitriles) than is (I). It can be speculated that this difference in reactivity may be attributable to a more strained ligand arrangement in the core of (II) than in (I). The distortion from ideal octahedral geometry for the core ligands in (I) is slightly less than in (II) with a mean absolute deviation from ideal geometry (90 or 180°) for the O–Ru–O angles of 3.4 in (I) and 5.9° in (II).

Intensity data were collected at the National Institute for Materials Research, CSIR, Pretoria.

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Acta Cryst. (1986). **C42**, 49–51

Structure of (2,2'-Bipyridyl)tetrachloroplatinum(IV)

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(Received 4 June 1985; accepted 17 September 1985)

Abstract. $[\text{PtCl}_4(\text{C}_{10}\text{H}_8\text{N}_2)]$, $M_r = 493.1$, monoclinic, Pn , $a = 8.117$ (1), $b = 6.734$ (4), $c = 12.294$ (3) Å, $\beta = 104.15$ (2)°, $V = 651.6$ Å³, $Z = 2$, $D_x = 2.513$ g cm⁻³, Mo $K\alpha$, $\lambda = 0.71069$ Å, $\mu = 116.1$ cm⁻¹, $F(000) = 456$, $T = 294$ K, final $R = 0.048$ for 1933 reflections. The Pt is six-coordinate in essentially octahedral geometry with Pt–Cl 2.306 (3) to

2.320 (3) Å and Pt–N 2.038 (8) and 2.044 (9) Å. The Pt–Cl bond lengths reveal significant *trans* effects, the shorter bonds being *trans* to N.

Introduction. Oxidative addition reactions of planar four-coordinate Pt^{II} complexes have been the subject of considerable interest (Buse, Keller & Pritzkow, 1977; Gray, Gulliver, Levason & Webster, 1983). Generally, these oxidations have been achieved by reaction with molecular halogens and in some cases coordinated

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