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Structure of *trans*-Trichlorobis[diethyl(phenyl)phosphine]hydroxorhenium(IV)

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Abstract

The crystal-structure determination of one of the three isomers (the violet form) obtained by reacting ReO_4^- , PEt_2Ph and HCl in boiling ethanol is reported. $\text{C}_{20}\text{H}_{31}\text{Cl}_3\text{OP}_2\text{Re}$, $M_r = 642.0$, is monoclinic, $P2_1/a$; $a = 23.899$ (4), $b = 13.017$ (3), $c = 7.781$ (2) Å, $\gamma = 91.21$ (2)°, $Z = 4$, $D_c = 1.76$ Mg m $^{-3}$, $\mu(\text{Mo } K\alpha) = 5.24$ mm $^{-1}$. The structure has been determined from 3338 observed reflections and refined to an R factor of 0.034. Structural data and IR spectra agree in indicating that the violet complex corresponds to the formula $[\text{Re}(\text{OH})\text{Cl}_3(\text{PEt}_2\text{Ph})_2]$.

Introduction

Reaction of perrhenates, diethyl(phenyl)phosphine and hydrochloric acid in boiling ethanol (Chatt & Rowe, 1962) yields a mixture of three complexes, a blue (I), green (II) and violet one (III), whose analyses indicate the formula $[\text{ReOCl}_3(\text{PEt}_2\text{Ph})_2]$. (I) was identified as a *cis* isomer of $[\text{ReOCl}_3(\text{PEt}_2\text{Ph})_2]$ on the grounds of its dipole moment (36.1×10^{-30} Cm), while (II) (dipole

moment = 5.7×10^{-30} Cm) was confirmed to be the *trans* isomer by a two-dimensional X-ray structure determination (Ehrlich & Owston, 1963). (III) has too low a dipole moment (10.0×10^{-30} Cm) to be the other *cis* isomer and, unlike (I) and (II), is paramagnetic in the solid state ($\mu_{\text{eff}} = 17.80 \times 10^{-24}$ J T $^{-1}$); it was assigned the formula *trans*- $[\text{Re}(\text{OH})\text{Cl}_3(\text{PEt}_2\text{Ph})_2]$, because preliminary X-ray results indicated cell parameters almost identical with those of (II) but there was a strong element of doubt as the IR spectrum showed a possible $\text{Re}=\text{O}$ bond and no OH absorption. In a following paper (Chatt, Garforth, Johnson & Rowe, 1964) the same violet complex (III) was interpreted as a solid solution of 10–22% of a new, intensely violet, complex of Re^{IV} , *i.e.* *trans*- $[\text{ReCl}_4(\text{PEt}_2\text{Ph})_2]$ in green *trans*- $[\text{ReOCl}_3(\text{PEt}_2\text{Ph})_2]$ (II), in spite of the fact that the separation of the two components was not actually achieved and the two compounds were not isomorphous.

This paper reports the crystal-structure determination of (III) and will show that structural data and IR spectra agree in indicating that (III) corresponds to the formula first suggested by Chatt & Rowe (1962), *i.e.* *trans*- $[\text{Re}(\text{OH})\text{Cl}_3(\text{PEt}_2\text{Ph})_2]$.

Experimental

Crystals of (III) were prepared according to Chatt & Rowe (1962) and directly crystallized from the mother liquor. The visible, NMR and IR spectra are strictly similar to those reported by Chatt & Rowe (1962) and Chatt *et al.* (1964). However additional IR spectra carried out in solid KBr showed another absorption band centered at 2920 cm^{-1} , which had not been reported in the previous papers.

The crystal used for X-ray investigation was a prism having dimensions 0.03 \times 0.12 \times 0.29 mm in the [100], [010] and [001] directions respectively.

Intensity data were collected on an automatic Siemens AED diffractometer with Zr-filtered Mo $K\alpha$ radiation and an $\omega/2\theta$ scan technique ($\theta < 25^\circ$). In all, 4749 independent reflections were collected, out of which 3338 reflections having $I \geq 3\sigma(I)$ were considered observed. Intensities were corrected for absorption by geometrical methods. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974) and computations were mainly carried out by means of the *SHELX 76* system of crystallographic programs (Sheldrick, 1976).

The structure was solved by means of Patterson and Fourier methods and refined by full-matrix least squares, assuming anisotropic temperature factors for

all the non-H atoms. All H atoms were given calculated positions, except the H atom bonded to oxygen, which was omitted. Final disagreement factors were $R (= \sum |\Delta| / \sum |F_o|) = 0.034$ and $R_w (= \sum w|\Delta|^2 / \sum w|F_o|^2)^{1/2} = 0.035$. Weights were given according to the formula $1.7/w = \sigma^2(F_o) + 0.00017|F_o|^2$. The attempt to locate the H atom linked to oxygen from the difference Fourier map calculated after refinement was unsuccessful. Final values of positional coordinates and U_{eq} are given in Table 1.*

Discussion

The crystal structure consists of a van der Waals packing (Fig. 1) of molecules without significantly short intermolecular contacts. An *ORTEP* (Johnson, 1971) view of the molecule is reported in Fig. 2. Bond distances and angles are reported in Table 2.

The coordination around the Re atom can be described with reference to the mean plane through Re, Cl(1), Cl(3) and O. The displacements of the four atoms from this plane are 0.014, -0.007, -0.007 and -0.001 Å respectively and the Cl(2) atom lies at 0.089 Å from it. This plane is nearly orthogonal (89.3°) to the P(1), Re, P(2) plane, from which Cl(2) and O are displaced by 0.465 and -0.326 Å

Table 1. Positional ($\times 10^4$) and thermal ($\text{\AA}^2 \times 10^3$) parameters with *e.s.d.*'s in parentheses

U_{eq} according to Hamilton (1959).

	x	y	z	U_{eq}
Re	3896.6 (1)	2602.1 (2)	1065.2 (4)	41.5 (2)
Cl(1)	4134 (1)	1223 (1)	-752 (3)	55 (1)
Cl(2)	4307 (1)	3774 (1)	-1034 (3)	52 (1)
Cl(3)	3669 (1)	4080 (1)	2722 (3)	65 (1)
O	3607 (2)	1744 (3)	2654 (6)	49 (3)
P(1)	3059 (1)	2841 (1)	-726 (2)	40 (1)
P(2)	4875 (1)	2634 (1)	2111 (2)	42 (1)
C(1)	2521 (2)	1936 (5)	-19 (9)	40 (4)
C(2)	2150 (3)	2202 (6)	1270 (10)	60 (5)
C(3)	1745 (3)	1521 (8)	1812 (12)	78 (6)
C(4)	1705 (4)	548 (8)	1094 (15)	86 (7)
C(5)	2073 (4)	264 (7)	-144 (14)	83 (7)
C(6)	2488 (3)	963 (5)	-700 (11)	63 (5)
C(7)	2719 (3)	4080 (5)	-609 (10)	56 (5)
C(8)	3036 (3)	4962 (5)	-1458 (12)	72 (6)
C(9)	3169 (3)	2604 (5)	-3001 (9)	49 (4)
C(10)	2648 (3)	2673 (7)	-4129 (10)	69 (5)
C(11)	5393 (3)	2438 (5)	463 (8)	40 (4)
C(12)	5534 (3)	1445 (5)	-32 (9)	45 (4)
C(13)	5919 (3)	1296 (6)	-1300 (10)	58 (5)
C(14)	6175 (3)	2133 (6)	-2126 (11)	63 (6)
C(15)	6044 (3)	3095 (6)	-1642 (10)	56 (5)
C(16)	5654 (3)	3263 (5)	-354 (10)	49 (4)
C(17)	5069 (3)	3828 (6)	3204 (10)	58 (5)
C(18)	5668 (4)	3848 (7)	3921 (12)	81 (6)
C(19)	4985 (3)	1588 (7)	3618 (10)	68 (6)
C(20)	4703 (5)	1689 (9)	5265 (13)	110 (8)

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36152 (22 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

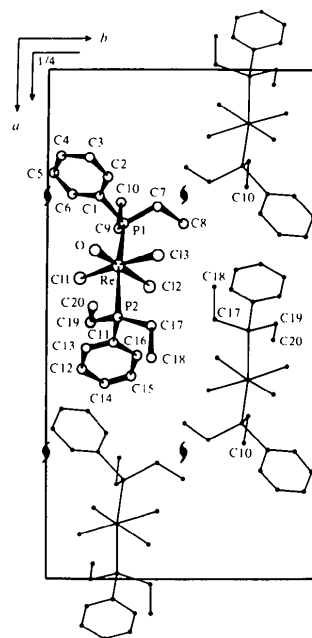


Fig. 1. A projection of the crystal packing along *c*.

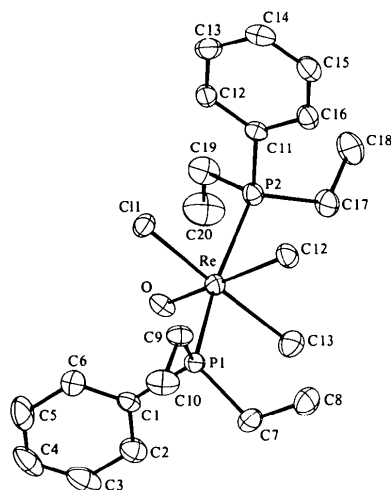


Fig. 2. An ORTEP (Johnson, 1971) view of the molecule.

Table 2. Interatomic distances (Å) and angles (°) with e.s.d.'s in parentheses

Re—Cl(1)	2.364 (2)	C(2)—C(3)	1.37 (1)
Re—Cl(2)	2.428 (2)	C(3)—C(4)	1.39 (1)
Re—Cl(3)	2.389 (2)	C(4)—C(5)	1.36 (1)
Re—O	1.795 (4)	C(5)—C(6)	1.40 (1)
Re—P(1)	2.465 (2)	C(7)—C(8)	1.51 (1)
Re—P(2)	2.475 (2)	C(9)—C(10)	1.53 (1)
P(1)—C(1)	1.812 (6)	C(11)—C(12)	1.40 (1)
P(1)—C(7)	1.823 (6)	C(11)—C(16)	1.39 (1)
P(1)—C(9)	1.817 (7)	C(12)—C(13)	1.37 (1)
P(2)—C(11)	1.805 (7)	C(13)—C(14)	1.40 (1)
P(2)—C(17)	1.823 (7)	C(14)—C(15)	1.35 (1)
P(2)—C(19)	1.820 (7)	C(15)—C(16)	1.39 (1)
C(1)—C(2)	1.39 (1)	C(17)—C(18)	1.54 (1)
C(1)—C(6)	1.37 (1)	C(19)—C(20)	1.46 (1)
Cl(1)—Re—Cl(2)	88.5 (1)	C(11)—P(2)—C(17)	106.8 (3)
Cl(1)—Re—Cl(3)	175.7 (1)	C(11)—P(2)—C(19)	103.9 (3)
Cl(1)—Re—O	92.0 (2)	C(17)—P(2)—C(19)	107.4 (4)
Cl(1)—Re—P(1)	88.1 (1)	P(1)—C(1)—C(2)	120.3 (5)
Cl(1)—Re—P(2)	88.1 (1)	P(1)—C(1)—C(6)	120.6 (5)
Cl(2)—Re—Cl(3)	87.3 (1)	C(2)—C(1)—C(6)	119.0 (6)
Cl(2)—Re—O	178.5 (1)	C(1)—C(2)—C(3)	120.6 (8)
Cl(2)—Re—P(1)	81.9 (1)	C(2)—C(3)—C(4)	120.2 (9)
Cl(2)—Re—P(2)	80.9 (1)	C(3)—C(4)—C(5)	120.0 (8)
Cl(3)—Re—O	92.2 (2)	C(4)—C(5)—C(6)	119.7 (9)
Cl(3)—Re—P(1)	90.2 (1)	C(1)—C(6)—C(5)	120.4 (8)
Cl(3)—Re—P(2)	92.3 (1)	P(1)—C(7)—C(8)	115.0 (5)
O—Re—P(1)	99.5 (1)	P(1)—C(9)—C(10)	115.4 (5)
O—Re—P(2)	97.8 (1)	P(2)—C(11)—C(12)	120.5 (5)
P(1)—Re—P(2)	162.5 (1)	P(2)—C(11)—C(16)	121.0 (5)
Re—P(1)—C(1)	108.3 (2)	C(12)—C(11)—C(16)	118.5 (6)
Re—P(1)—C(7)	117.5 (3)	C(11)—C(12)—C(13)	120.5 (6)
Re—P(1)—C(9)	114.1 (2)	C(12)—C(13)—C(14)	120.5 (7)
C(1)—P(1)—C(7)	103.7 (3)	C(13)—C(14)—C(15)	119.3 (8)
C(1)—P(1)—C(9)	106.8 (3)	C(14)—C(15)—C(16)	121.1 (7)
C(7)—P(1)—C(9)	105.5 (3)	C(11)—C(16)—C(15)	120.1 (6)
Re—P(2)—C(11)	114.5 (2)	P(2)—C(17)—C(18)	113.9 (5)
Re—P(2)—C(17)	113.0 (3)	P(2)—C(19)—C(20)	115.1 (7)
Re—P(2)—C(19)	110.6 (3)		

respectively. A stereographic projection of the coordination polyhedron, where the O atom is located at the south pole, shows that the mean distortions from the octahedral geometry are caused by the repulsion between oxygen and the four atoms lying in the equatorial plane; all of them are pushed upwards, but the two P atoms to a greater angular extent [O—Re—P(1) and O—Re—P(2) of 99.5 (1) and 97.8 (1)° respectively] than the two Cl atoms [O—Re—Cl(1) and O—Re—Cl(3) of 92.0 (2) and 92.2 (2)° respectively]. Such an uneven distortion is paralleled by the different values of the angles P(1)—Re—P(2) = 162.5 (1) and Cl(1)—Re—Cl(3) = 175.7 (1)° and it cannot be accounted for by the balance of the repulsions among second neighbors, as can be seen by comparing the differences, $\Delta = d_{ij} - (r_i + r_j)$, between the contact distances and the sums of the van der Waals radii (Bondi, 1964) inside the coordination polyhedron. In fact the Δ values [O—P(1) = -0.04 , O—P(2) = -0.07 , Cl(2)—P(1) = -0.34 , Cl(2)—P(2) = -0.37 Å; O—Cl(1) = -0.25 , O—Cl(3) = -0.23 , Cl(2)—Cl(1) = -0.16 , Cl(2)—Cl(3) = -0.17 Å] show that the O atom repels the two Cl atoms less and the two P atoms much more than is required by the simple balance of the non-bonded interactions.

The experimental torsion angles defining the conformation of the phosphine ligands are reported in Table 3. The substituents on both ligands are rotated in such a way as to be staggered with respect to Cl(2), while C(1) and C(19) nearly eclipse the oxygen. Moreover the two phenyl rings are nearly perpendicular to the Re—P(1)—C(1) or Re—P(2)—C(11) plane. An attempt was made to establish whether the observed conformation was simply determined by the intramolecular van de Waals forces. Accordingly all the torsion angles of Table 3 have been estimated by minimizing the non-bonded intramolecular potential energy of the free molecule by using semi-empirical atom—atom potentials (Giglio, 1969) and the steepest-descent method. As potentials for P and Re were lacking, the former was assigned atom—atom potentials pertaining to sulfur, whose van der Waals radius is identical, and the latter to carbon, in view of the small van der Waals radius usually associated with metal ions. Anyway, the effect of this last approximation on the final results is to be considered small owing to the central position of the Re atom. Two different minimizations were carried out, with and without rotation around the Re—P bonds. The results, shown in Table 3, seem to indicate that the conformation adopted by the ligands is mainly determined by the intramolecular forces, as the differences between calculated and experimental values are not large and are possibly ascribable to deficiencies of the energetic model.

The remaining aspects of the geometry of the complex need a knowledge of the true formula of the

compound in order to be discussed. As mentioned, two different hypotheses have been put forward: (i) a hydroxo derivative of Re^{IV} having the formula $\text{trans}[\text{Re}(\text{OH})\text{Cl}_3(\text{PEt}_2\text{Ph})_2]$ and (ii) a solid solution of 10–20% of paramagnetic $\text{trans}[\text{ReCl}_4(\text{PEt}_2\text{Ph})_2]$ in diamagnetic $\text{trans}[\text{ReOCl}_3(\text{PEt}_2\text{Ph})_2]$.

In our opinion, the second hypothesis has to be rejected on the grounds of the present structural findings. The anisotropic thermal parameters of oxygen are quite normal (Fig. 2) and the ellipsoid does not show any elongation along the $\text{Re}-\text{O}$ vector. Moreover detailed ΔF maps failed to detect significant features in the zone where the fourth Cl atom should be located.

Conversely, the hypothesis of a hydroxo complex of Re^{IV} seems to be confirmed by several facts. The observed $\text{Re}-\text{O}$ distance is 1.795 (4) Å; this is intermediate between a triple $\text{Re}-\text{O}_{\text{oxo}}$ bond distance, which lies in the range of 1.68–1.72 Å (see Table 4), and that of a simple $\text{Re}-\text{O}$ bond, which is known to be 1.92–2.10 Å (Bombieri, Mazzi, Gilli & Hernández-Cano, 1978; Hursthouse, Jayaweera & Quick, 1979), and is therefore indicative of a double $\text{Re}-\text{O}$ bond. As

the isomer with a triple $\text{Re}-\text{O}$ bond corresponds to $\text{trans}[\text{ReOCl}_3(\text{PEt}_2\text{Ph})_2]$, the double bond is consistent only with a hydroxo complex of Re^{IV} , in agreement with the fact that the IR spectrum shows an absorption at 2920 cm^{-1} , which could be associated with the $\text{O}-\text{H}$ stretching.

A double $\text{Re}-\text{OH}$ bond could explain, at least in part, another characteristic of the IR spectrum, that is the absorption at 978 cm^{-1} , which is usually associated with the multiple-bonded $\text{Re}-\text{O}$ stretching, and, moreover, is the only way by which the observed low magnetic moment of this compound ($17.80 \times 10^{-24}\text{ J T}^{-1}$; Chatt & Rowe, 1962) can be accounted for. Re^{IV} is a d^3 ion and in its octahedral complexes the t_{2g} orbitals would be free to accommodate the three electrons, leading to three unpaired spins. However, two of these orbitals, say d_{xy} and d_{xz} for the z and x axes pointing towards P(1) and O respectively, have the proper symmetry for π bonding with one of the doubly filled p_y or p_z AO's of the OH^- group, leading to a total of one bonding, two non-bonding and one antibonding MO's occupied by five electrons, and therefore to a single unpaired spin, in agreement with the observed

Table 3. Observed and calculated torsion angles ($^\circ$) for the PEt_2Ph ligands with *e.s.d.*'s in parentheses

	Observed value	Calculated value	$ \Delta $	Calculated value	$ \Delta $
O—Re—P(1)—C(1)	−9.2 (3)	−16.3	7.1	—	—
Re—P(1)—C(1)—C(2)	89.3 (6)	95.2	5.9	97.4	8.1
Re—P(1)—C(9)—C(10)	175.9 (5)	168.2	7.7	169.9	6.0
Re—P(1)—C(7)—C(8)	72.0 (6)	70.4	1.6	69.1	2.9
O—Re—P(2)—C(11)	131.9 (3)	113.0	18.9	—	—
Re—P(2)—C(11)—C(12)	−84.9 (6)	−91.9	7.0	−90.0	5.1
Re—P(2)—C(17)—C(18)	177.9 (5)	180.9	3.0	180.8	2.9
Re—P(2)—C(19)—C(20)	−69.5 (7)	−69.2	0.3	−69.4	0.1

Table 4. Comparison of selected bond distances (Å) and angles ($^\circ$) in oxo complexes of Re^{V} and in the present compound with *e.s.d.*'s in parentheses

	$\text{Re}-\text{O}_{\text{oxo}}$	$\text{Re}-\text{Cl}$	Cl <i>trans</i> to	$\text{Re}-\text{P}$	P <i>trans</i> to	Mean $X-\text{Re}-\text{O}_{\text{oxo}}$ angle	Reference
$[\text{ReO}(\text{H}_2\text{O})\text{Cl}_3(\text{tu})]$	1.71 (2)	2.425 (7) 2.385 (7) 2.347 (7)	S Cl Cl			98.7	<i>a</i>
$[\text{ReOCl}_2(\text{PPh}_3)(\text{acac})]$	1.69 (1)	2.376 (7) 2.399 (7)	Cl Cl	2.431 (4)	O	96.9	<i>b</i>
$\mu\text{-O-bis}[\text{ReO}(\text{ddtc})_2]$	1.72 (1) 1.72 (1)					93.2 93.6	<i>c</i>
$[\text{Re}_2\text{O}_2\text{Cl}_4(\text{PPh}_3)_2(\text{salen})]$	1.68 (1) 1.68 (1)	2.367 (3) 2.365 (3) 2.416 (4) 2.409 (4)	N N P P	2.466 (4) 2.472 (4)	Cl Cl	93.1 92.7	<i>d</i>
$[\text{ReOCl}_2(\text{PPh}_3)\{\text{Ph}(\text{O})\text{CNMe}_3\}]$	1.69 (1)	2.344 (4) 2.388 (3)	N P	2.472 (2)	Cl	95.9	<i>e</i>
$[\text{Re}(\text{OH})\text{Cl}_3(\text{PEt}_2\text{Ph})_2]$	1.795 (4)	2.364 (2) 2.389 (2) 2.428 (2)	Cl Cl $\text{O}_{\text{hydroxo}}$	2.465 (2) 2.475 (2)	P P	95.3	<i>f</i>

tu = thiourea; acac = acetylacetonato; ddtc = N,N' -diethyldithiocarbamato; salen = N,N' -ethylenebis(salicylideneiminato).

References: (a) Lis (1977); (b) Lock & Che'ng Wan (1975); (c) Fletcher & Skapski (1972); (d) Bombieri *et al.* (1978); (e) Hursthouse *et al.* (1979); (f) present work.

magnetic moment. As we did not succeed in locating the H atom, we do not know whether the π bond is actually formed by the d_{xy} or d_{xz} AO's, although the observed deformations in the coordination polyhedron seem to indicate that the last AO is more probably used. In fact the greater repulsion exerted by oxygen towards the P atoms, which could not be explained by the balance of the intramolecular contacts, can be understood in terms of increased angular space occupied by the π MO in the P(1)–Re–P(2) plane.

A comparison of the Re–P and Re–Cl distances observed in the present compound with those found in oxo complexes of Re^V is reported in Table 4. The mean Re–P distance of 2.47 Å agrees well with that observed in the other molecules studied, with the exception of [ReOCl₂(PPh₃)(acac)], where this distance was found significantly shorter (2.43 Å). The Re–Cl distances deserve more attention. Data of Table 4 show that Re–Cl distances are systematically longer when the Cl atom is *trans* to a ligand known to exert a relevant *trans* influence (like thiourea or PR₃) and that a similar lengthening of the Re–Cl bond *trans* to the multiply bonded O atom is observed in the compound under examination. However, whether or not such an effect can be interpreted in terms of *trans* weakening remains controversial, as Bright & Ibers (1969) believe that the weakening of bonds *trans* to multiply bonded nitrido or imino ligands is essentially caused by steric repulsion of other ligands, while Shustorovich, Porai-Koshits & Buslaev (1975) uphold with theoretical arguments and structural data the point of view of a true *trans* influence exerted by a multiple metal–ligand bond.

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Structure of Chlorobis(*N*-methylsalicylideneiminato)oxorhenium(V)

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Abstract

C₁₆H₁₆ClN₂O₃Re, $M_r = 505.9$, is monoclinic, space group *I2/c*; $a = 20.731(4)$, $b = 11.949(3)$, $c = 13.578(3)$ Å, $\beta = 94.13(2)^\circ$, $Z = 8$, $D_c = 2.00$ Mg

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m^{-3} , $\mu(\text{Mo } K\alpha) = 7.08 \text{ mm}^{-1}$. The crystal structure has been determined and refined to an *R* value of 0.028 for 2767 observed reflections. The coordination around the Re atom is approximately octahedral. The two bidentate ligands are mutually orthogonal and the oxygen of

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