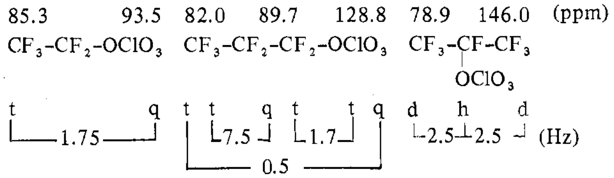
Chart I



with higher resolution data than previously reported[8] for $C_2F_5OClO_3$, are summarized in Chart I (d = doublet, t = triplet, q = quartet, h = heptet). The resonances of fluorines geminal to a perchlorato group were broadened due to chlorine quadrupole relaxation. On the basis of their relative peak areas, the ratio of the two isomers was *n* 68% and iso 32%.

The fact that in the reaction of $FOClO_3$ with $CF_3CF{=}CF_2$ both isomers are formed significantly differs from the previously reported[6] $ClOClO_3$ and $BrOClO_3$ reactions where exclusive Markownikoff type additions occurred. The latter produced 100% of $CF_3CFXCF_2OClO_3$, as expected for a polar addition of the positively polarized terminal halogen to the carbon with the highest electron density.[7]

$$CF_3CF{=}CF_2 + Cl^{\delta+}{-}O^{\delta-}ClO_3 \rightarrow CF_3CFClCF_2OClO_3$$

The formation of both isomers (*n* and iso) in the corresponding $FOClO_3$ reaction suggests that the F–O bond in $FOClO_3$ is not strongly polarized in either direction. This is not surprising in view of the known very small dipole moment (0.023 D) of the closely related $FClO_3$ molecule[9] and the expected similar electronegativities of a $-ClO_3$ and an $-OClO_3$ group. The fact that the percentage of *n* isomer was somewhat higher than that of the iso isomer can be explained by steric effects (bulky CF_3 group) and is insufficient reason to postulate a strongly positive fluorine in $FOClO_3$. The occurrence of a free-radical mechanism is unlikely in view of the high yield of the products (74%), the mild (−45 °C) and well-controlled reaction conditions, and the absence of detectable amounts of C_3F_8 and $C_3F_6(OClO_3)_2$ in the reaction products.

In contrast to the olefin addition reactions, the reaction of $FOClO_3$ with CF_3I was more difficult to control. The primary reaction path appears to have involved oxidation of the iodine followed by degradation to oxygenated and fluorinated species. Nevertheless, a modest yield (8%) of the desired perchlorate, CF_3OClO_3, was realized. By comparison, the $ClOClO_3$–CF_3I reaction is also vigorous but can be controlled to give a nearly quantitative yield of CF_3OClO_3.[8]

In summary, it has been shown that $FOClO_3$ can add to carbon–carbon double bonds to produce alkyl perchlorates in good yield. The formation of two isomers with the unsymmetrical olefin $CF_3CF{=}CF_2$ indicates that the O–F bond in $FOClO_3$ is of low polarity and does not justify the assumption of significant positive character for fluorine. Since a CF_3O- group is considerably less electronegative than a O_3ClO- group, the above results imply that, contrary to general acceptance, covalent hypofluorites, such as CF_3OF, do not contain a positive fluorine. Indeed, it would be most difficult to rationalize how the addition of fluorine to a less electronegative element, such as carbon, would render the latter more electronegative than fluorine itself.

Acknowledgment. We gratefully acknowledge helpful discussion with Dr. L. R. Grant and financial support of this work by the Office of Naval Research, Power Branch.

Registry No. $FOClO_3$, 10049-03-3; $CF_3CF_2CF_2OClO_3$, 70749-47-2; $CF_3CF(OClO_3)CF_3$, 70749-48-3; $C_2F_5OClO_3$, 22675-67-8; CF_3OClO_3, 52003-45-9; C_3F_6, 116-15-4; C_2F_4, 116-14-3; CF_3I, 359-37-5.

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Crystal and Molecular Structure of Bis(μ-acetato)-dichlorobis(dimethylphenylphosphine)dipalladium(II) Chloroformate

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and S. C. Nyburg*

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Powell and Jack[1] examined a series of bridging *cis*-di-μ-carboxylate complexes of palladium(II) and, using IR spectra and variable temperature ^1H NMR techniques, inferred the possible existence of two conformational isomers. We report here details of a structure analysis of one Pd complex of this series, a preliminary account of which has been given in the paper by Powell and Jack.

Experimental Section

Crystals were obtained as red prisms from chloroform.[1] The crystal employed for this investigation had dimensions 0.32 × 0.64 × 0.24 mm and was sealed in a capillary tube with a few other small crystals of the same material at both ends. Preliminary crystal data were determined photographically. Fifteen general reflections in the range $31.0° < 2\theta < 35.3°$ were well centered on a computer-automated Picker diffractometer using filtered Mo Kα radiation to obtain the best least-squares crystal parameters. The density of the crystals was measured by flotation in a mixture of diethyl ether and tribromomethane.

Crystal Data

Bis(μ-acetato)-dichlorobis(dimethylphenylphosphine)dipalladium(II)–chloroform: $C_{20}H_{28}O_4Cl_2P_2Pd_2\cdot{}^1/_2CHCl_3$, $M_r = 797.5$; mp 132–140 °C; monoclinic, space group $P2_1/c$, $a = 8.119$ (3) Å, $b = 15.684$ (4) Å, $c = 22.406$ (6) Å, $\beta = 97.7$ (3)°; $D_m = 1.69$, $D_c = 1.70$ (25 °C) g cm^{-3}; $Z = 4$; λ 0.7107 Å, μ(Mo Kα) = 15.6 cm^{-1}.

A total of 5844 intensities was collected in the θ–2θ scan mode at 2° min^{-1}; $\sigma(I)$ was taken as $(\text{scan} + \sum B_c)^{1/2}$ where B_c is the background count. The 3972 reflections with $I > 2\sigma(I)$ were considered to have significant intensity. The structure was solved by the heavy-atom method, scattering factors used being those of Cromer and Mann[2] with anomalous dispersion corrections. For P, Cl, and Pd atoms, $\Delta f'$ and $\Delta f''$ are 0.1 and 0.2, 0.1 and 0.2, and −1.1 and 1.2, respectively.[3] Idealized positions for hydrogen atoms (C–H = 1.00 Å) were included in F_c with isotropic temperature factors, B, taken as 6.0 Å^2. The R value with anisotropic thermal factors for nonhydrogen atoms was 0.083. Atomic coordinates are given in Table I. At this stage of refinement several peaks with electron density as high as 3.5 e A^{-3} were found around the centers of symmetry (0, $^1/_2$, 0) and (0, 0, $^1/_2$). The crystal density indicated that these peaks correspond to solvent chloroform molecules. By use of program HOLE[4] it was found that each site could accommodate only one chloroform molecule, that is, a total of two solvent molecules per unit cell. Since $CHCl_3$ lacks a center of symmetry, it must be disordered at the two sites. The electron density map shows in fact six relatively heavy peaks about the center, and assuming them to be of chlorine atoms, we derived a best-fitting set of chloroform atom coordinates based on a C–Cl distance of 1.762 Å and a Cl–C–Cl angle of 110.9°. These atoms were included (multiplicity 0.5) with fixed positions in the last cycle of least-squares refinement. The temperature factors were large

Table I. Positional and Thermal Parameters (×10^4)

(a) Dimer Molecules (Nonhydrogen Atoms)[a]

	x	y	z	β_{11} (or B)	β_{22}	β_{33}	β_{12}	β_{23}	β_{13}
Pd(1)	1489 (1)	1996 (1)	2831 (0)	113 (2)	32 (1)	13 (0)	2 (1)	7 (0)	−3 (0)
Pd(2)	1885 (1)	1169 (1)	1684 (0)	95 (2)	27 (0)	16 (0)	0 (1)	13 (0)	−2 (0)
Cl(3)	373 (6)	990 (3)	3379 (2)	263 (9)	48 (2)	22 (1)	−16 (3)	27 (2)	1 (1)
Cl(4)	4458 (4)	566 (3)	1834 (2)	104 (5)	51 (2)	33 (1)	14 (3)	12 (2)	−5 (1)
P(5)	3922 (5)	1872 (3)	3401 (2)	141 (6)	53 (2)	17 (1)	16 (3)	−1 (2)	−7 (1)
P(6)	846 (4)	−113 (2)	1777 (2)	107 (5)	30 (1)	17 (1)	0 (2)	11 (1)	0 (1)
O(7)	−829 (11)	2223 (7)	2308 (4)	114 (14)	56 (5)	17 (2)	16 (7)	8 (4)	−3 (3)
O(8)	−399 (11)	1668 (6)	1434 (4)	142 (15)	39 (4)	19 (2)	15 (7)	7 (5)	−2 (3)
O(9)	2827 (12)	2417 (6)	1568 (4)	187 (18)	31 (4)	25 (3)	−24 (7)	31 (5)	−3 (3)
O(10)	2397 (14)	3024 (6)	2425 (5)	220 (20)	32 (4)	22 (3)	−12 (7)	20 (6)	−4 (3)
C(11)	−1246 (14)	2077 (8)	1779 (6)	84 (18)	30 (5)	23 (3)	−2 (8)	12 (6)	−1 (3)
C(12)	2767 (15)	3033 (8)	1883 (6)	100 (19)	38 (6)	20 (3)	−6 (9)	13 (6)	0 (4)
C(13)	−2954 (20)	2425 (12)	1475 (7)	186 (28)	67 (10)	20 (4)	26 (14)	4 (8)	−3 (5)
C(14)	3329 (23)	3874 (10)	1704 (8)	253 (36)	41 (7)	28 (4)	−23 (13)	12 (10)	−1 (4)
C(15)	4568 (29)	812 (13)	3594 (11)	354 (50)	72 (12)	49 (7)	91 (20)	−72 (15)	−17 (7)
C(16)	5621 (19)	2265 (18)	2999 (8)	104 (24)	172 (19)	27 (4)	−31 (18)	6 (8)	−17 (8)
C(17)	3940 (14)	2492 (10)	4077 (6)	84 (18)	56 (8)	15 (3)	14 (9)	2 (5)	−4 (4)
C(18)	4225 (22)	3364 (12)	4041 (8)	222 (33)	62 (10)	24 (4)	33 (15)	−14 (9)	−11 (5)
C(19)	4264 (24)	3853 (14)	4611 (14)	163 (34)	71 (13)	83 (11)	−3 (16)	12 (15)	−53 (10)
C(20)	3929 (28)	3386 (24)	5133 (9)	237 (42)	213 (28)	15 (5)	95 (30)	0 (11)	−16 (10)
C(21)	3640 (31)	2544 (20)	5145 (11)	331 (52)	115 (18)	33 (6)	26 (26)	−12 (14)	−24 (9)
C(22)	3695 (23)	2142 (13)	4614 (7)	263 (38)	82 (12)	17 (4)	−6 (17)	0 (9)	−4 (5)
C(23)	1739 (21)	−690 (10)	2431 (6)	286 (35)	40 (7)	15 (3)	2 (12)	9 (8)	7 (4)
C(24)	−1295 (16)	−82 (10)	1896 (8)	98 (20)	51 (8)	43 (5)	−18 (10)	36 (8)	4 (5)
C(25)	992 (15)	−791 (8)	1131 (6)	111 (19)	39 (6)	19 (3)	−9 (9)	13 (6)	−6 (3)
C(26)	617 (19)	−1648 (10)	1150 (6)	187 (27)	45 (7)	18 (3)	−9 (11)	11 (7)	−4 (4)
C(27)	769 (26)	−2160 (10)	656 (9)	323 (44)	32 (7)	34 (5)	−5 (14)	12 (12)	−7 (5)
C(28)	1117 (22)	−1828 (11)	132 (7)	257 (35)	48 (8)	21 (4)	−2 (14)	13 (9)	−9 (4)
C(29)	1414 (26)	−988 (14)	105 (7)	333 (44)	81 (12)	13 (3)	25 (18)	35 (10)	1 (5)
C(30)	1411 (22)	−449 (10)	609 (7)	289 (36)	43 (7)	18 (3)	1 (13)	35 (9)	1 (4)
Cl(A)	−1730	−452	4739	922 (115)	591 (32)	185 (13)	−210 (49)	−150 (31)	112 (18)
Cl(B)	300	850	5400	544 (86)	382 (20)	157 (11)	−204 (36)	−141 (24)	103 (12)
Cl(C)	186	−621	4924	133 (96)	250 (25)	151 (12)	74 (42)	100 (27)	98 (14)
C(A)	180	97	4812	42 (18)					
H(A)	250	434	4400	32 (13)					

(b) Hydrogen Atom Coordinates[b]

	x	y	z		x	y	z
H(13A)	−280	270	130	H(23C)	91	−110	256
H(13B)	−360	270	179	H(24A)	−150	10	160
H(13C)	−364	197	127	H(24B)	−156	28	223
H(14A)	280	390	130	H(24C)	−183	−67	193
H(14B)	295	434	195	H(18)	443	362	364
H(14C)	457	390	172	H(19)	452	452	457
H(15A)	400	70	380	H(20)	399	377	553
H(15B)	578	77	380	H(21)	332	228	553
H(15C)	449	40	323	H(22)	356	149	462
H(16A)	545	290	294	H(26)	20	−191	152
H(16B)	650	220	318	H(27)	65	−282	69
H(16C)	555	198	260	H(28)	113	−221	−24
H(23A)	210	−80	240	H(29)	163	−74	−28
H(23B)	207	−28	278	H(30)	172	9	59

[a] Temperature factors are of the form $\exp[-(h^2\beta_{11} + k^2\beta_{22} + l^2\beta_{33} + 2hk\beta_{12} + 2kl\beta_{23} + 2hl\beta_{13})]$. Last five atoms listed are for the $CHCl_3$ molecule. B values are in Å^2. [b] $B = 6.0$ Å^2.

(see Table I). After refinement the residual R fell from 0.082 to 0.068.

An ORTEP plot of the molecule is shown in Figure 1. Bond lengths and angles are shown in Figure 2. Fractional atomic coordinates and temperature factors are listed in Table I; Table II lists the best molecular planes. The structure factors are listed in Table III.[5] A packing diagram showing the positions of disordered $CHCl_3$ molecules is given in Figure 3.

Discussion

It is clear from Figure 1 that the central part of the complex has the boat conformation, its geometry closely matching that found in μ-allyl palladium acetate[6] with a very similar Pd···Pd distance, 2.944 (2) vs. 2.936 (4) Å. There is no direct interaction between the two metal ions. A similar conformation is found in bis(μ-acetato)-bis{2-(methylallyl)-3-norbornyl}-dipalladium(II)[7] and, where three Pd atoms are bridged in the one complex, in tris{μ-acetato-μ-acetoximato-palladium(II)}–hemibenzene,[8] and in trimeric palladium(II) acetate.[9] Acetate groups bridge in the same manner in copper(II) acetate.[10]

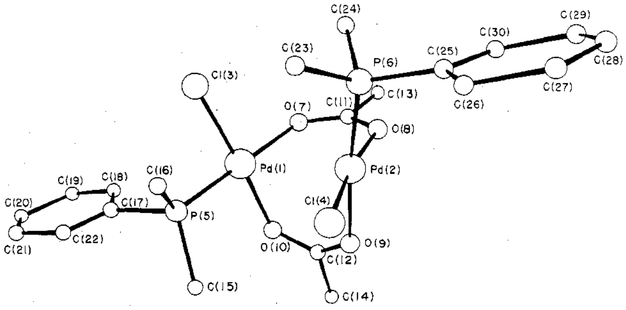

Figure 1. ORTEP plot of the molecule.

Table II. Best Least-Squares Planes

(a) Coefficients of Planes $AX + BY + CZ = D^a$

plane	description	A	B	C	D
1	sq plane about Pd(1)	0.352 (2)	−0.694 (4)	−0.628 (4)	6.002 (1)
2	sq plane about Pd(2)	0.109 (2)	−0.153 (2)	−0.982 (4)	3.835 (1)
3	OAc group 1	0.473 (16)	0.850 (29)	−0.232 (14)	−1.127 (6)
4	OAc group 2	−0.903 (24)	0.191 (11)	−0.384 (16)	2.250 (6)

(b) Deviations from Planes (Å)^b

	planes					planes			
atoms	1	2	3	4	atoms	1	2	3	4
Pd(1)	−0.006 (1)		(−0.243 (1))	(−0.114 (1))	O(8)		−0.225 (9)	−0.001 (10)	
Pd(2)		0.005 (1)	(−0.048 (1))	(−0.240 (1))	O(9)		−0.037 (10)		0.008 (10)
Cl(3)	0.040 (4)				O(10)	0.244 (10)			0.008 (11)
Cl(4)		−0.035 (4)			C(11)			0.005 (12)	
P(5)	0.019 (4)				C(12)				−0.038 (13)
P(6)		−0.004 (3)			C(13)			−0.003 (18)	
O(7)	0.119 (10)		−0.001 (10)		C(14)				0.019 (18)

(c) Angles between Normals to Planes (deg)

	plane		
plane	1	2	3
2	40.4 (2)		81.4 (12)
3	106.1 (12)		
4	102.1 (10)	75.6 (10)	100.1 (16)

^{a} X, Y, and Z are real orthogonal coordinates (Å) with X along x and Z along z^*. ^{b} Those values in parentheses are for atoms not included in the calculation of the plane.

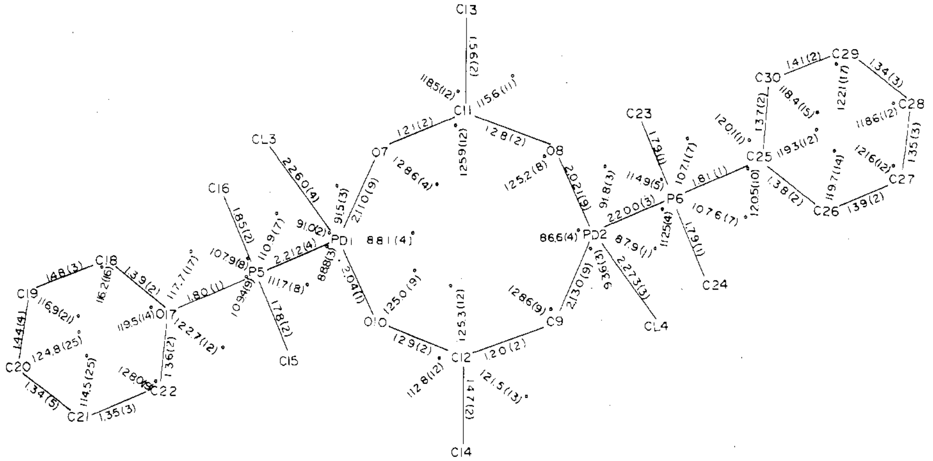

Figure 2. Bond lengths and angles.

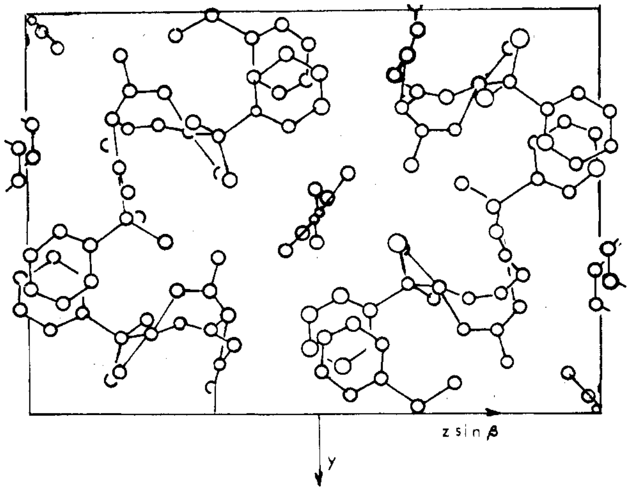

Figure 3. Structure in x projection.

Coordination at the Pd atom is square planar (Figure 3) with the oxygen atoms of the bridging carboxylate groups cis. The two square-planar planes subtend 40.4 (2)°. The carboxylate groups are planar, but the Pd atoms lie significantly out of these planes (Table II). The two carboxylate groups are also not quite symmetrical, C(13) subtending, at C(11), an angle of 115.6 (11)° with O(8) and of 118.5 (12)° with O(7); similarly C(14) subtends, at C(12), an angle of 112.8 (12)° with O(10) and of 121.5 (13)° with O(9). (Such asymmetry is fairly common; compare, for example, angles of 116 and 120° in copper(II) acetate.[10])

There is possible trans influence on the Pd–O lengths, those trans to chlorine being 2.04 (1) and 2.02 (1) Å and those trans to phosphorus being 2.110 (9) and 2.130 (9) Å.

No intermolecular bonding between the solvent molecule and the dimer is observed. Other stereochemical features appear normal.

Acknowledgment. Financial assistance from the National Research Council of Canada is gratefully acknowledged.

Registry No. $\{(PPhMe_2)PdCl(CH_3CO_2)\}_2\cdot{}^1/_2CHCl_3$, 70629-72-0.

Supplementary Material Available: Table III, listing of structure factor amplitudes (17 pages). Ordering information is given on any current masthead page.

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Mechanism of Olefin Metathesis and Cyclopropanation

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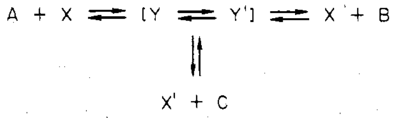
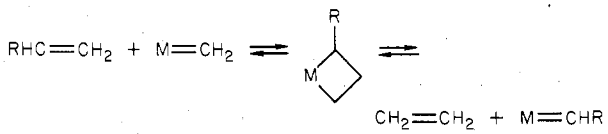
A serious objection to the Chauvin[1] mechanism for olefin methathesis has recently been raised by Mango.[2] He calculated that more cyclopropane than is observed should be present in olefin metathesis systems at equilibration conditions. Since Gassman and Johnson[3] have reported that cyclopropanes can undergo "metathesis" reactions, the cyclopropane to olefin interconversion is a kinetically accessible pathway. If this is the case, the calculation can be based on the following scheme where A and B are related olefins, C is related cyclopropanes, X and X′ are catalytic species, and Y and Y′ are intermediates.



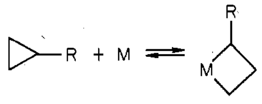
If, as by definition, the catalyst remains unchanged and therefore its free energy change is 0, the equilibrium constant is easily calculated from $\Delta\Delta G° = 3(\Delta G°(\text{ethylene})) - 2(\Delta G°(\text{cyclopropane}))$. The resulting equilibrium constant as calculated is 0.17, and 20% of ethylene should be converted to cyclopropane.[2] Since only traces of cyclopropanes are observed[4,5] in metathesis reactions, the Chauvin mechanism appears incorrect. However, as will be shown below, it is the analysis that is in error.

The important steps in metathesis as required by the Chauvin mechanism are

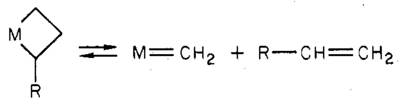


(1)

In order for there to be a related catalytic process to convert cyclopropanes into olefins as suggested in the earlier analysis, steps 2–4 are required. The key reaction is step 4. Without this reaction the metal is not a catalyst and does not cancel from the calculation.



(2)



(3)

$$2\underset{X}{M{=}CH_2} \rightleftarrows \underset{X'}{M} + CH_2{=}CH_2 \qquad (4)$$

Gassman's results analyzed in these terms are instead described by reaction 5, an apparent common

$$n\underset{X}{M{=}CH_2} \rightarrow n\underset{X'}{M} + \text{polymer} \qquad (5)$$

fate of tungsten catalysts without sufficient olefin present.[5] This reaction does not relate olefin and X′ to X as is required by eq 4.[6] Reaction 4 can be considered equivalent to an initiation mechanism for metathesis.[7] In all cases observed to date, initiation of metathesis requires added reagents and is very slow relative to the catalytic reaction itself. The information of cyclopropane is a chain-termination step, and the *concentration of cyclopropane cannot be greater than the concentration of X.* This being the case, the final concentration of cyclopropane cannot be calculated exactly since the necessary equilibrium is

$$X + A \rightleftarrows X' + C$$

$$\Delta\Delta G = (\Delta G°(\text{ethylene}) + \Delta G°(X)) - (\Delta G°(\text{cyclopropane}) + \Delta G°(X'))$$

$$\Delta = (\Delta G°(X) - \Delta G°(X')) = \text{unknown}$$

On the basis of studies of related organometallic catalysts, the value of Δ would be expected to vary greatly.[8] At one extreme would be excellent catalysts for the cyclopropanation of olefins with diazo compounds but very poor metathesis catalysts while the opposite extreme would give outstanding metathesis systems.

If a complex formed cyclopropanes in significant amounts, it would give low yields of metathesis products and be a very short-lived catalyst.[9]

As can be seen, the Chauvin mechanism is not inconsistent with the thermodynamic calculations and remains as the mechanism most compatible with a large body of other experimental investigations.[7]

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A New Series of Imidazole Thioether Chelating Ligands for Bioinorganic Copper

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Authentic synthetic analogues of the "blue" copper proteins (type I)[1] would be valuable in understanding the detailed geometrical and electronic structure of copper in its biological environment. All copper(II) model compounds reported so

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