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Phosphine substituted derivatives of $[Fe_5C(CO)_{15}]$; crystal structure of $[Fe_5C(CO)_{12}(PMe_2Ph)_3]$

André Gourdon * and Yves Jeannin,

Laboratoire de Chimie des Métaux de Transition, CNRS UA 419, Université P. et M. Curie, 4 Place Jussieu, 75252 Paris Cedex 05 (France)

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Abstract

The square based pyramidal cluster $[Fe_5C(CO)_{12}(PMe_2Ph)_3]$ has been synthesised and its structure determined by an X-ray diffraction study. The carbido carbon atom lies 0.2 Å below the basal plane, which indicates a partial localization of the cluster electron density on this atom. Chemical and electrochemical reduction of $[Fe_5C(CO)_{13}(Me_2PCH_2CH_2PMe_2)]$ and $[Fe_5C(CO)_{12}(PMe_2Ph)_3]$ gives unstable anionic electron-rich species.

Introduction

Although the tetranuclear clusters $[Fe_4C(CO)_{13}]$ [1] and $[Fe_4C(CO)_{12}]^{2-}$ [2,3] are reactive at the carbido carbon atom, the exposed carbide in square-pyramidal pentanuclear iron clusters $[Fe_5C(CO)_{15}]$ and $[Fe_5C(CO)_{14}]^{2-}$ is chemically inert [4]. This is mainly attributable to the fact that the carbon orbitals are strongly stabilized by interaction with cluster orbitals [5,6]. Previous observations that an increase of the cluster charge could induce a distortion of the Fe_5C core towards octahedral and partial localization of electron density on this atom [7] led us to prepare same electron-rich phosphine derivatives of $[Fe_5C(CO)_{15}]$.

Experimental

Infrared spectra were recorded as solutions in 0.1 mm KBr cells on a Perkin Elmer 597. ^{31}P NMR spectra were recorded on a Bruker WM 250 with $CDCl_3$ as solvent and aqueous H_3PO_4 as external reference. Chromatography was conducted

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* Author for correspondence. Present address: Groupe Electronique Moléculaire, LOE-CEMES, CNRS, 29 rue Jeanne Marvig, B.P. 4347, 31055 Toulouse (France).

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under pressure on silica gel. Reactions were carried out under argon by standard Schlenk techniques in dried, freshly distilled solvents. 1,2 Bis(dimethylphosphino)ethane (dmpe) and dimethylphenylphosphine were used as purchased (Aldrich). $[Fe_5C(CO)_{15}]$ was prepared by published procedures [13] and recrystallized from warm toluene prior to use.

Syntheses

The method used for the preparation of $[Fe_5C(CO)_{13}(dmpe)]$ and $[Fe_5C(CO)_{12}(PMe_2Ph)_3]$ is very similar to that described by Cookes and Mays [8].

$[Fe_5C(CO)_{13}(dmpe)]$

A solution of dmpe (0.5 ml, 3 mmol) in CH_2Cl_2 (20 ml) was added dropwise to a solution of $[Fe_5C(CO)_{15}]$ (1 g, 1.41 mmol) in CH_2Cl_2 (100 ml). After stirring 3 h at room temperature the solvent was evaporated. The dark residue was dried overnight under vacuum and chromatographed (eluent: CH_2Cl_2). Recrystallisation from CH_2Cl_2/hexane gave red neeldes of $[Fe_5C(CO)_{13}(dmpe)]$ (535 mg, yield: 47%). Anal. Found: C, 29.47; H, 1.81; P, 7.80. $C_{20}H_{16}Fe_5O_{13}P_2$ calcd.: C, 29.82; H, 2.00; P, 7.69%. IR ν(CO) (CH_2Cl_2): 2060 w, 2010 vs, 1995 m, 1940 vw sh, 1935 m br cm^{-1}.

$[Fe_5C(CO)_{12}(PMe_2Ph)_3]$

An excess of PMe_2Ph (1.8 ml, 1.75 g, 12.65 mmol) was added to a solution of $[Fe_5C(CO)_{15}]$ (1 g, 1.41 mmol) in CH_2Cl_2 (80 ml). After 24 h stirring at room temperature, the solution was concentrated under vacuum and chromatographed on silica gel with dichloromethane/petroleum ether as eluent. The red brown fraction left a dark crude product upon evaporation under vacuum. The product was recrystallised by slow evaporation of a solution in a 1/1 mixture of dichloromethane/hexane. Anal. Found: C, 42.57; H, 3.2; P, 8.80. $C_{37}H_{33}Fe_5O_{12}P_3$ calcd.: C, 42.66; H, 3.19, P, 8.92. IR ν(CO) (CH_2Cl_2): 2040 s, 1985 vs, 1965 m, 1940 w sh, 1820 m br cm^{-1}.

X-ray study

A crystal of $[Fe_5C(CO)_{12}(PMe_2Ph)_3]$ (0.2 × 0.2 × 0.6 mm) was mounted in an argon-filled Lindemann tube. After survey photography by precession techniques, the crystal was transferred to a CAD-4F diffractometer. Table 1 summarizes the crystal data and intensity measurement parameters. The structure was solved using CRYSTALS [14] on a VAX 11/725. The data were corrected for Lorentz, polarisation and absorption effects [15]. The iron atom positions were determined by direct methods and the structure was refined with a large-block approximation to the normal matrix using unit weights. Indications of positions of the phenyl group H atoms were obtained from difference maps, but for structure factor calculations these atoms were placed in calculated positions, with the positions recalculated after each refinement cycle. Methyl group H atoms coordinates were refined using slack constraints [16]. Final R and R' values * were 4.0 and 4.3%. Fractional atomic

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* $R = \frac{\sum||F_o|-|F_c||}{\sum|F_o|}$, $R' = \left[\frac{\sum(|F_o|-|F_c|)^2}{\sum|F_o|^2}\right]^{1/2}$

Table 1

Crystal data and data collection parameters

Formula	$C_{37}H_{33}Fe_5O_{12}P_3$
F_w	1041.82
Crystal system	Monoclinic
Space group	C2/c
a (Å)	40.135(10)
b (Å)	9.692(4)
c (Å)	32.396(8)
β (°)	139.31(2)
V (Å^3)	8215
Z	8
$F(000)$	4208
D_c (g cm^{-3})	1.68
μ(Mo-K_α) (cm^{-1})	19.0
2θ limits (°)	$4 < 2\theta < 50$
Reflections with $F_o > 3\sigma(F_o)$ used in refinements	3389
Temperature	25 °C

coordinates are given in Table 2 and selected bond lengths and bond angles in Table 3 *.

Results and discussion

$[Fe_5C(CO)_{13}(dmpe)]$ (**1**) (dmpe = bis(dimethyl)phosphinoethane $Me_2PCH_2CH_2PMe_2$) and $[Fe_5C(CO)_{12}(PMe_2Ph)_3]$ (**2**) was prepared by reaction at room temperature of $[Fe_5C(CO)_{15}]$ with the corresponding phosphine [8].

The structure of **2** was established by a single crystal X-ray study, and is shown in Fig. 1. **2** retains the square pyramidal Fe_5C core found for the parent compound $[Fe_5C(CO)_{15}]$ [7]. As observed for the ruthenium analogue $[Ru_5C(CO)_{13}(PPh_3)_2]$ [9], two of the phosphine ligands (P(3) and P(5)) occupy *trans*-axial sites about the basal plane. The third phosphine ligand (P(4)) is bound to a basal iron atom but in an equatorial position, probably for steric reasons. The ^{31}P NMR spectrum consists of two singlets (18.8 and 22.3 ppm) with relative intensities 1/2, showing that this geometry is maintained in solution at room temperature. The Fe–Fe bond lengths show quite large variations (from 2.544(2) to 2.655(2) Å). Four of the carbonyl groups close to the basal plane (C(25)O(25), C(32)O(32), C(43)O(43), C(54)O(54)) are semi-bridging in this plane, as shown by the Fe–C–O angles (from 144.5(7) to 156.6(7)°) and by the coefficients α ** which range from 0.114 to 0.274. This bridging character is more important than in $[Fe_5C(CO)_{15}]$, in which the Fe–C–O angles range from 170.0(6) to 172.6(6)° and the coefficients α from 0.55 to 0.61. As observed in some other cases [2,10], the increase in cluster electron density, induced here by substitution of 3 CO by 3 phosphine ligands, moves some CO towards more

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* Full tables of bond lengths and angles, thermal vibration parameters, least square best planes, and of observed and calculated structure factors are available from the authors.

** α is defined as $(d_2 - d_1)/d_1$, where d_1 and d_2 are the shorter and the longer M–CO distances. Ligands with $0.1 < \alpha < 0.6$ are regarded as semi-bridging. See ref. 1.

Table 2

Fractional atomic coordinates with e.s.d.'s in parentheses U_{iso} $(U_{11} \times U_{22} \times U_{33})^{1/3}$

Atom	x	y	z	U_{iso}
Fe(1)	0.33917(4)	0.2800(1)	−0.32056(5)	0.0288
Fe(2)	0.33949(4)	0.2429(1)	−0.24251(5)	0.0356
Fe(3)	0.26935(4)	0.1237(1)	−0.35497(5)	0.0304
Fe(4)	0.34334(4)	0.0105(1)	−0.33021(5)	0.0289
Fe(5)	0.41097(4)	0.1276(1)	−0.21830(5)	0.0317
C(1)	0.3399(3)	0.1097(8)	−0.2838(4)	0.0293
C(11)	0.3916(3)	0.3958(9)	−0.2781(4)	0.0451
O(11)	0.4222(2)	0.4786(7)	−0.2538(3)	0.0603
C(12)	0.2915(3)	0.4085(9)	−0.3587(4)	0.0391
O(12)	0.2608(2)	0.4931(6)	−0.3855(5)	0.0487
C(13)	0.3212(3)	0.2679(8)	−0.3895(4)	0.0381
O(13)	0.3057(3)	0.2802(7)	−0.4370(3)	0.0566
C(21)	0.3335(3)	0.188(1)	−0.1959(4)	0.0449
O(21)	0.3296(3)	0.1594(8)	−0.1657(3)	0.0620
C(22)	0.3296(3)	0.4246(9)	−0.2430(4)	0.0413
O(22)	0.3246(3)	0.5378(7)	−0.2416(4)	0.0661
C(25)	0.4100(3)	0.271(1)	−0.1711(4)	0.0477
O(25)	0.4418(2)	0.3329(8)	−0.1234(3)	0.0627
C(31)	0.2148(3)	0.1850(8)	−0.4332(4)	0.0326
O(31)	0.1798(2)	0.2252(7)	−0.4834(3)	0.0517
C(32)	0.2512(3)	0.2315(9)	−0.3286(4)	0.0404
O(32)	0.2237(2)	0.2874(7)	−0.3321(3)	0.0532
P(3)	0.23029(8)	−0.0496(2)	−0.3588(1)	0.0378
C(311)	0.1635(3)	−0.0173(9)	−0.4094(4)	0.039(2)
C(312)	0.1277(4)	−0.022(1)	−0.4740(4)	0.057(3)
C(313)	0.0761(4)	0.000(1)	−0.5132(5)	0.084(4)
C(314)	0.0627(5)	0.028(1)	−0.4872(6)	0.092(4)
C(315)	0.0955(5)	0.038(1)	−0.4263(6)	0.087(4)
C(316)	0.1482(4)	0.015(1)	−0.3844(5)	0.068(3)
C(321)	0.2247(4)	−0.217(1)	−0.3889(5)	0.079(3)
C(331)	0.2599(3)	−0.094(1)	−0.2826(4)	0.074(3)
C(41)	0.3458(3)	−0.1673(8)	−0.3167(4)	0.0364
O(41)	0.3467(2)	−0.2823(6)	−0.3088(3)	0.0489
C(43)	0.2753(3)	−0.0085(9)	−0.4074(4)	0.0400
O(43)	0.2421(2)	−0.0522(7)	−0.4575(3)	0.0508
P(4)	0.36357(8)	−0.0238(2)	−0.3803(1)	0.0322
C(411)	0.3896(3)	−0.1929(8)	−0.3693(4)	0.034(2)
C(412)	0.4400(3)	−0.2119(9)	−0.3333(4)	0.045(2)
C(413)	0.4571(4)	−0.342(1)	−0.3297(4)	0.057(3)
C(414)	0.4236(4)	−0.449(1)	−0.3627(5)	0.061(3)
C(415)	0.3735(4)	−0.432(1)	−0.3985(5)	0.058(3)
C(416)	0.3564(3)	−0.3054(9)	−0.4016(4)	0.043(2)
C(421)	0.3114(3)	−0.0151(8)	−0.4662(3)	0.045(2)
C(431)	0.4096(3)	0.0972(9)	−0.3595(4)	0.054(2)
C(51)	0.4685(3)	0.2138(9)	−0.1789(4)	0.0416
O(51)	0.5052(2)	0.2661(7)	−0.1551(3)	0.0629
C(54)	0.4258(3)	0.0046(9)	−0.2466(4)	0.0389
O(54)	0.4563(2)	−0.0582(7)	−0.2356(3)	0.0483
P(5)	0.44868(8)	−0.0224(3)	−0.1396(1)	0.0411
C(511)	0.5174(3)	−0.0161(9)	−0.0765(4)	0.042(2)
C(512)	0.5437(3)	0.094(1)	−0.0339(4)	0.053(2)
C(513)	0.5963(4)	0.103(1)	0.0145(5)	0.063(3)

Table 2 (continued)

Atom	x	y	z	U_{iso}
C(514)	0.6237(4)	0.004(1)	0.0202(5)	0.063(3)
C(515)	0.5989(4)	−0.101(1)	−0.0227(5)	0.071(3)
C(516)	0.5460(4)	−0.112(1)	−0.0707(5)	0.058(3)
C(521)	0.4361(3)	−0.011(1)	−0.0963(4)	0.070(3)
C(531)	0.4348(3)	−0.205(1)	−0.1597(4)	0.065(3)

bridging positions, where the CO ligands are able to withdraw more of the electron density.

The carbido-carbon atom lies 0.20(1) Å below the basal plane, compared with 0.09(1) Å in $[Fe_5C(CO)_{15}]$, 0.11(1) Å in $[Fe_5N(CO)_{14}]^-$ and in $[Fe_5C(CO)_{13}(NO)]^-$ [11], and 0.18(1) Å in $[Fe_5C(CO)_{14}]^{2-}$; the distance between the carbido-carbon atom and the Fe_4 basal plane increases with the cluster electron density. The same trend is observed in the ruthenium analogues $[Ru_5C(CO)_{15}]$, $[Ru_5C(CO)_{14}(PPh_3)]$, $[Ru_5C(CO)_{13}(PPh_3)_2]$ [9], in which the distance to the Ru_4 plane increases from 0.11(2) to 0.23(1) Å with increasing carbonyl replacements. These observations can be rationalized with the help of the orbital interaction diagram for $[Fe_5X(CO)_{15}]$ [5] (Fig. 2); the substitution of some carbonyl groups by less π-acceptor ligands or by charges leads to a destabilisation of the Fe_5L_{15} fragment, which increases the energy separation between this fragment and the carbido-carbon atom and in particular between the $1a_1$ Fe_5L_{15} orbital and the carbon pz orbital (Fig. 2). Consequently, the interaction between these two fragment orbitals is weaker in $[Fe_5C(CO)_{12}(PMe_2Ph)_3]$ than in $[Fe_5C(CO)_{15}]$. As a result, the distance between the carbido-carbon atom and the iron basal plane increases, and there is a partial localization of electron density at the carbon atom. These results are in good agreement with spectroscopic data published by Sosinsky and coworkers [12], who showed that, in electron rich carbido clusters the electron charge residing in the iron framework is shifted on to the carbido-carbon atom.

In order throw light on this phenomenon, and to obtain pyramidal carbido clusters reactive with electrophiles at the carbide carbon, we studied the chemical and electrochemical reduction of the clusters **1** and **2**.

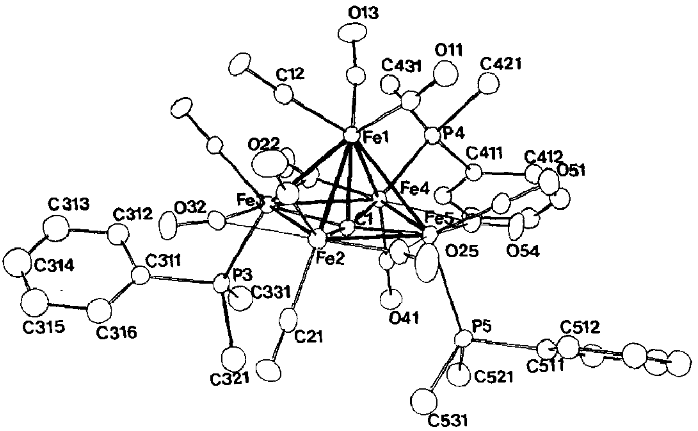


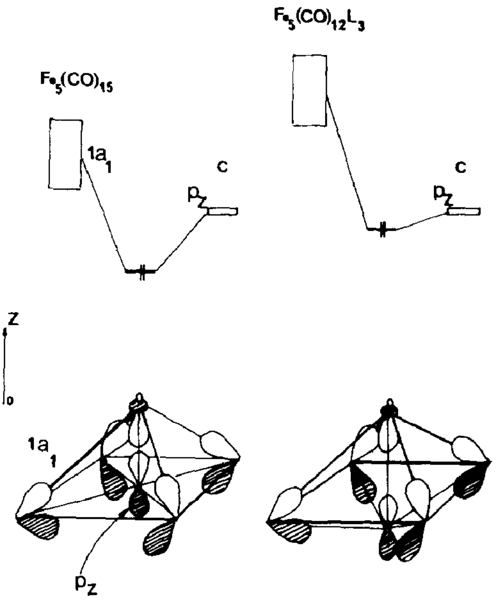
Fig. 1. The molecular structure of $[Fe_5C(CO)_{12}(PMe_2Ph)_3]$.

Table 3

Selected bond lengths and angles with e.s.d.'s in parentheses

Fe(1)–Fe(2)	2.544(2)	Fe(2)–Fe(3)	2.642(2)
Fe(1)–Fe(3)	2.579(1)	Fe(2)–Fe(5)	2.585(2)
Fe(1)–Fe(4)	2.651(2)	Fe(3)–Fe(4)	2.655(2)
Fe(1)–Fe(5)	2.642(2)	Fe(4)–Fe(5)	2.622(2)
Fe(1)–C(1)	2.022(7)	Fe(4)–P(4)	2.323(2)
Fe(1)–C(11)	1.786(9)	Fe(4)–C(54)	2.166(8)
Fe(1)–C(12)	1.775(8)	Fe(5)–C(1)	1.867(8)
Fe(1)–C(13)	1.756(8)	Fe(5)–C(25)	2.087(9)
Fe(2)–C(1)	1.869(8)	Fe(5)–C(51)	1.785(8)
Fe(2)–C(21)	1.780(9)	Fe(5)–C(54)	1.855(8)
Fe(2)–C(22)	1.803(9)	Fe(5)–P(5)	2.249(2)
Fe(2)–C(25)	1.873(8)	P(3)–C(311)	1.818(8)
Fe(2)–C(32)	2.315(8)	P(3)–C(321)	1.82(1)
Fe(3)–C(1)	1.858(8)	P(3)–C(331)	1.80(1)
Fe(3)–C(31)	1.776(8)	P(4)–C(411)	1.827(8)
Fe(3)–C(32)	1.817(9)	P(4)–C(421)	1.816(8)
Fe(3)–P(3)	2.235(2)	P(4)–C(431)	1.833(9)
Fe(3)–C(43)	2.279(9)	P(5)–C(511)	1.799(8)
Fe(4)–C(1)	1.875(8)	P(5)–C(521)	1.817(9)
Fe(4)–C(41)	1.762(8)	P(5)–C(531)	1.82(1)
Fe(4)–C(43)	1.841(8)		
C(11)–Fe(1)–C(1)	125.2(4)	C(54)–Fe(5)–C(51)	85.9(4)
C(12)–Fe(1)–C(1)	120.6(3)	P(5)–Fe(5)–C(1)	111.3(2)
C(12)–Fe(1)–C(11)	96.5(4)	P(5)–Fe(5)–C(51)	95.7(3)
C(13)–Fe(1)–C(1)	120.2(4)	P(5)–Fe(5)–C(54)	87.3(3)
C(13)–Fe(1)–C(11)	97.5(4)	O(25)–C(25)–Fe(2)	144.5(7)
C(13)–Fe(1)–C(12)	88.5(4)	O(32)–C(32)–Fe(3)	156.6(7)
C(21)–Fe(2)–C(1)	118.9(4)	C(311)–P(3)–Fe(3)	114.4(3)
C(22)–Fe(2)–C(1)	142.2(4)	C(321)–P(3)–Fe(3)	119.9(3)
C(22)–Fe(2)–C(21)	97.3(4)	C(321)–P(3)–C(311)	99.8(4)
C(25)–Fe(2)–C(1)	99.1(4)	C(331)–P(3)–Fe(3)	113.5(3)
C(25)–Fe(2)–C(21)	92.6(4)	C(331)–P(3)–C(311)	105.4(4)
C(25)–Fe(2)–C(22)	89.2(4)	C(331)–P(3)–C(321)	101.8(4)
C(31)–Fe(3)–C(1)	149.8(3)	O(43)–C(43)–Fe(4)	153.6(7)
C(32)–Fe(3)–C(1)	103.7(4)	C(411)–P(4)–Fe(4)	116.3(3)
C(32)–Fe(3)–C(31)	88.8(4)	C(421)–P(4)–Fe(4)	117.2(3)
P(3)–Fe(3)–C(1)	111.0(2)	C(421)–P(4)–C(411)	99.5(4)
P(3)–Fe(3)–C(31)	96.8(3)	C(431)–P(4)–Fe(4)	116.1(3)
P(3)–Fe(3)–C(32)	86.8(3)	C(431)–P(4)–C(411)	103.5(4)
C(41)–Fe(4)–C(1)	109.0(4)	C(431)–P(4)–C(421)	101.7(4)
C(43)–Fe(4)–C(1)	101.8(4)	O(54)–C(54)–Fe(5)	147.9(7)
C(43)–Fe(4)–C(41)	89.2(4)	C(511)–P(5)–Fe(5)	113.6(3)
P(4)–Fe(4)–C(1)	155.5(2)	C(521)–P(5)–Fe(5)	119.5(3)
P(4)–Fe(4)–C(41)	92.9(3)	C(521)–P(5)–C(511)	101.8(4)
P(4)–Fe(4)–C(43)	88.9(3)	C(531)–P(5)–Fe(5)	117.5(3)
C(51)–Fe(5)–C(1)	152.6(4)	C(531)–P(5)–C(511)	103.3(4)
C(54)–Fe(5)–C(1)	99.4(3)	C(531)–P(5)–C(521)	98.5(4)

The change in the single sweep voltammogram of [$Fe_5C(CO)_{13}$(dmpe)] with temperature is very similar to that of [$Fe_5C(CO)_{15}$]. At low temperature (below −12 °C), the first reduction process is quasi-reversible with $E_p^{red1} = -0.76$ V and



(a) (b)

Fig. 2. Simplified interaction orbital diagram for: (a) $[Fe_5C(CO)_{15}]$ and (b) $[Fe_5C(CO)_{12}L_3]$.

$E_p^{ox1} = -0.66$ V vs. SCE. At higher temperature, a supplementary reoxidation wave at $E_p^{ox2} = -0.47$ V appears while the wave at -0.66 V collapses. At room temperature this last wave has fully disappeared, and the first reduction of $[Fe_5C(CO)_{13}(dmpe)]$ is fully irreversible with a peak-to-peak separation of -190 mV. These results can be interpreted as in the case of $[Fe_5C(CO)_{15}]$: at low temperature the quasi-reversible electron exchange can be assigned to a $2e^-$ transfer

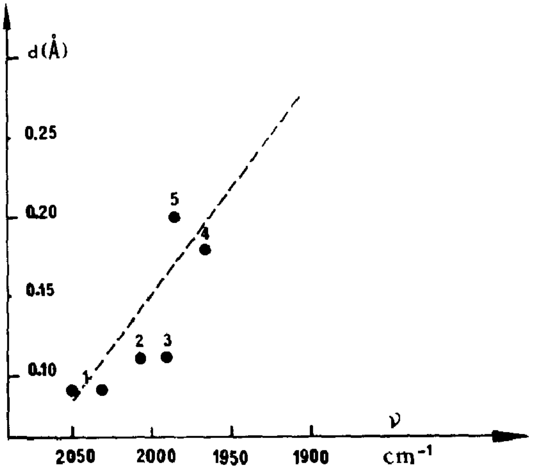


Fig. 3. Plot of the distance d (Å) between the heteroatom and the basal plane against the CO stretching frequency in: (1) $[Fe_5C(CO)_{15}]$, (2) $[Fe_5C(CO)_{13}(NO)]^-$, (3) $[Fe_5N(CO)_{14}]^-$, (4) $[Fe_5C(CO)_{14}]^{2-}$, (5) $[Fe_5C(CO)_{12}(PMe_2Ph)_3]$. Reduction of phosphine derivatives of $[Fe_5C(CO)_{15}]$ led to unstable intermediates with ν(CO) ca. 1920 cm^{-1}.

$[Fe_5C(CO)_{13}(dmpe)] \rightleftharpoons [Fe_5C(CO)_{13}(dmpe)]^{2-}$. At higher temperature, the 76 electron anion $[Fe_5C(CO)_{13}(dmpe)]^{2-}$ loses one CO group to give $[Fe_5C(CO)_{12}(dmpe)]^{2-}$. Reduction of $[Fe_5C(CO)_{13}(dmpe)]$ with Na/Hg amalgam in THF showed that the product obtained, which shows a very strong infrared band at 1920 cm^{-1}, is not stable and decomposes within one hour to give $[Fe_4C(CO)_{12}]^{2-}$ (ν(CO) 2010 w, 1970 s, 1945 s, 1915 w cm^{-1}). In contrast, the cyclic voltammogram of $[Fe_5C(CO)_{12}(PMe_2Ph)_3]$ does not show this temperature dependence, and represents a fully irreversible process even at $-20\,^{\circ}C$, with $E_p^{red1} = -0.97$ V and $E_p^{ox1} = -0.47$ V vs. SCE. Attempts to prepare $[Fe_5C(CO)_{11}(PMe_2Ph)_3]^{2-}$ by chemical reduction with Na/Hg amalgam or Na/benzophenone in THF were unsuccesful. The electron-rich complex obtained, which shows very low frequency ν(CO) absorption bands in THF (1975 m, 1920 vs br, 1865 w sh, 1715 w br cm^{-1}), is very unstable, and decomposes during purification to give a mixture of products that have been not fully characterized.

Acknowledgements

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