of ion pairs providing highly reactive unsolvated anions.[7]

We have applied this method with excellent results to the basic degradation of 1,12-$C_2B_{10}H_{12}$, which has previously proved to be very resistant to the standard techniques employed for the basic degradation of 1,2-$C_2B_{10}H_{12}$ and 1,7-$C_2B_{10}H_{12}$. Yields of the desired [*nido*-2,9-$C_2B_9H_{12}]^-$ of >95% were achieved in relatively short reaction times by using KOH solubilized in benzene with 18-crown-6. The exact mechanism by which the removal of the boron vertex takes place is not known at this time, but the stoichiometry we have observed clearly indicates that 4 equiv of 18-crown-6/mol of 1,12-$C_2B_{10}H_{12}$ is required. The aprotic nature of the reaction medium is also important. The addition of only 10% ethanol to the normal benzene solvent reduces the yield to only 20% of that observed in pure benzene.

The product is isolated as a [K(18-crown-6)] salt, which is very mildly air sensitive in solution and the solid state. It is moderately soluble in acetone and acetonitrile and somewhat soluble in methylene chloride, benzene, ethanol, and tetrahydrofuran. The cation may be exchanged by a two-phase acidification procedure in which the [K(18-crown-6)] cation extracts into the water layer and the carborane intermediate, presumably 2,9-$C_2B_9H_{13}$ reported by Plesek and Hermanek,[3] extracts into the hydrocarbon layer. Treatment with trialkylamines gives trialkylammonium salts, $[NR_3H]$[*nido*-2,9-$C_2B_9H_{12}$], in essentially quantitative yields. The cesium salt, Cs[*nido*-2,9-$C_2B_9H_{12}$], has also been prepared in crystalline form by neutralizing the intermediate with aqueous CsOH. The method is general with different purification methods depending upon the salt.

On the basis of analytical, chemical, and spectral properties, especially the symmetry indicated by the ^{11}B NMR spectrum, the anion almost certainly has a structure analogous to that depicted in Figure 1: an 11-membered icosahedral fragment with the carbons in the 2- and 9-positions. This is the only thermally stable [*nido*-$C_2B_9H_{12}]^-$ species known in which only one of the two carbons is in the open face. Stanko et al. have reported the preparation of [*nido*-11-R-2,7-$C_2B_9H_{11}]^-$ by the low-temperature alkylation of [*nido*-7,8-$C_2B_9H_{11}]^{2-}$ with alkyl halides[8] and have confirmed the structure by X-ray crystallography;[9] however, [*nido*-11-R-2,7-$C_2B_9H_{11}]^-$ is reported to rearrange rapidly at room temperature to give [*nido*-8-R-7,9-$C_2B_9H_{11}]^-$.

The impetus behind the preparation of [*nido*-2,9-$C_2B_9H_{12}]^-$ was the desire to investigate icosahedral metallacarboranes of the form $L_nMC_2B_9H_{11}$ in which only one carbon atom would be adjacent to the metal vertex. Two representative complexes have been synthesized by using established methods. The reaction of cobaltous chloride and cyclopentadiene with [*nido*-2,9-$C_2B_9H_{12}]^-$ in the presence of KOH led to the preparation of a yellow compound, which was spectroscopically identical with the material previously isolated in small quantities from the thermal isomerization of [*closo*-3-(η^5-C_5H_5)-3,1,2-$CoC_2B_9H_{11}$] and identified as the compound [*closo*-2-(η^5-C_5H_5)-2,1,12-$CoC_2B_9H_{11}$] on the basis of ^{11}B NMR and physical properties.[10]

Of more interest is a new rhodacarborane [*closo*-2,2-$(PPh_3)_2$-2-H-2,1,12-$RhC_2B_9H_{11}$] (I), which was synthesized from [K(18-crown-6)][*nido*-2,9-$C_2B_9H_{12}$] and $RhCl(PPh_3)_3$ by using the method of Paxson and Hawthorne.[11] Details of its spectral and physical properties along with those of its isomeric analogues, [*closo*-3,3-$(PPh_3)_2$-3-H-3,1,2-$RhC_2B_9H_{11}$] (II) and [*closo*-2,2-$(PPh_3)_2$-2-H-2,1,7-$RhC_2B_9H_{11}$] (III), which have previously been reported,[11] will be discussed elsewhere. I is only mildly air sensitive in the solid state and in solution, decomposing over a period of time to a blue material presumably similar to the rhodacarborane dimer recently reported by Hawthorne et al.[12] Like II and III, I is a catalyst for the hydrogenation and isomerization of olefins. Its catalytic behavior is similar in some ways to the other two isomers, but I gives faster rates for hydrogenation, in general, and is notably more stable than II and III under catalytic conditions.

Acknowledgment. We wish to thank Professor F. A. L. Anet for assistance in obtaining the ^{11}B NMR spectra and Mr. Paul E. Behnken for helpful discussions. This work was supported in part by the Office of Naval Research.

Registry No. I, 82848-97-3; [K(18-crown-6)][*nido*-2,9-$C_2B_9H_{12}$], 82918-08-9; $[NEt_3H]$[*nido*-2,9-$C_2B_9H_{12}$], 82951-14-2; [*closo*-2-(η^5-C_5H_5)-2,1,12-$CoC_2B_9H_{11}$], 38882-31-4; 1,12-$C_2B_{10}H_{12}$, 20644-12-6; 18-crown-6, 17455-13-9; $RhCl(PPh_3)_3$, 14694-95-2.

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Contribution from the Department of Chemistry
and Biochemistry, Utah State University, Logan, Utah 84321

1,2-Bis(bis(pentafluorophenyl)phosphino)ethane: Synthesis, Characterization, and Some Comparisons to 1,2-Bis(diphenylphosphino)ethane[1]

Ronald L. Cook and Joseph G. Morse*

Received April 2, 1982

A perusal of the literature since 1970 indicates how widespread the use of the ligand 1,2-bis(diphenylphosphino)ethane has become. Aside from its ability to act as a chelating and/or bridging ligand with a large number of transition metals, its widespread use can also be attributed to its relatively low cost and ease of handling.

Replacement of a C_6H_5 group by a C_6F_5 group would be expected to modify the electronic properties of the phosphorus donor atom while keeping stereochemical changes to a minimum (van der Waals radii are 1.2 Å for H and 1.35 Å for F).[2] Specifically, introduction of a C_6F_5 group would permit the use of ^{19}F NMR spectroscopy in characterization of the ligand and its resulting complexes. Also ^{19}F NMR methods can be used for investigation of the competition of the C_6F_5 π cloud and the metal d orbitals for the vacant phosphorus 3d orbitals.[3-5]

We wish to report the synthesis and characterization of the new ligand 1,2-bis(bis(pentafluorophenyl)phosphino)ethane (pfpe).

Experimental Section

All solvents were dried by standard methods. The reactants were stirred with a paddle stirrer equipped with an oil seal under an atmosphere of prepurified nitrogen. 1,2-Bis(dichlorophosphino)ethane (Strem Chemicals, Inc.) and bromopentafluorobenzene and penta-

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[1] Presented in part at the 181st National Meeting of the American Chemical Society, Atlanta, GA, March 1981.

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fluorobenzene (Alfa-Ventron) were used as purchased.

Method A. Grignard Reaction. A suspension of magnesium (2.2 g, 91 mmol) in ether (75 mL) was activated by the addition of 1,2-dibromoethane[6] (1.0 g, 5.3 mmol) and then cooled to 0 °C. A solution of bromopentafluorobenzene (22 g, 90 mmol) in ether (50 mL) was added dropwise to the stirred suspension. The suspension was allowed to warm to room temperature, and additional portions (14 g, 75 mmol) of 1,2-dibromoethane were added dropwise.

After 1 h of stirring the brown solution was cooled again to 0 °C and $Cl_2PCH_2CH_2PCl_2$ (5 g, 22 mmol) in 50 mL of ether was added dropwise. When it was allowed to warm to room temperature and stirred for 1 h, the dark brown solution lightened in color and a small amount of white precipitate appeared. The solution was then hydrolyzed with 50 mL of saturated NH_4Cl solution. The solution was filtered and the organic layer separated and dried over anhydrous Na_2SO_4. The filtered solids were extracted with hot acetone and the washings combined with the ether layer. The combined solutions were taken to dryness in a rotary evaporator. The resulting solid was dissolved in hot acetone and recovered as white crystals when the solution was cooled to −20 °C; mp 191–192 °C (uncorrected). Anal. Calcd for $C_{26}H_4F_{20}P_2$: C, 41.19; H, 0.531. Found: C, 41.26; H, 0.540.

Method B. Metalation. C_6F_5H (98%, 6.1 g, 36 mmol) in 50 mL of ether was added dropwise to a −90 °C solution of *n*-BuLi (18 mL, 2.0 M) in 100 mL of ether. $Cl_2PCH_2CH_2PCl_2$ (2 g, 9 mmol) in 50 mL of ether was then added dropwise to the −90 °C solution of C_6F_5Li. The solution was allowed to warm to room temperature and stirred for 1 h. The workup was identical with that used in method A; mp 191–192 °C (uncorrected). Anal. Calcd for $C_{26}H_4F_{20}P_2$: C, 41.19; H, 0.531. Found: C, 41.15; H, 0.535.

Mass Spectra. The mass spectra of pfpe prepared from method A or B were consistent with the formulation $(C_6F_5)_2PCH_2CH_2P(C_6F_5)_2$. The mass spectral data that follow include only the major peaks essential for the identification of pfpe. Assignments and relative intensities of peaks are included in parentheses: *m/e* 758 ($(C_6F_5)_2PCH_2CH_2P(C_6F_5)_2^+$, 28.8), 591 ($(C_6F_5)_2PCH_2CH_2PC_6F_5^+$, 14.1), 532 ($(C_6F_5)_3P^+$, 20.8), 365 ($(C_6F_5)_2P^+$, 100.0), 296 ($(C_{12}F_8^+$, 19.7), 217 ($C_6F_5PF^+$, 20.6), 198 ($C_6F_5^+$, 11.1), 129 ($C_6F_3^+$, 23.3), 69 ($CF_3^+$, 59.3).

Results and Discussion

1,2-Bis(bis(pentafluorophenyl)phosphino)ethane (pfpe) is obtained as an air-stable, white, crystalline solid. It is only sparingly soluble in CH_2Cl_2, Et_2O, C_6H_6, and acetone and is insoluble in alcohols, amines, and alkanes. It is appreciably soluble in hot acetone. The yields from the two methods are comparable, with the highest yield for method B being about 5% higher than that from method A (54 vs. 49.5%). The yields of pfpe have ranged from poor (9%) to fair (54%), with typical yields being in the range 30–35%. When 1,2-dibromoethane is used to activate the magnesium, pentafluorobenzoic acid is recovered in excess of 60% yields upon treatment of C_6F_5MgBr with dry ice and hydrolysis with saturated NH_4Cl. We conclude that the low yields are, at least in part, a consequence of the low solubility of pfpe in ether and THF and consequent poor recovery of product rather than poor product formation.

1,2-Bis(diphenylphosphino)ethane (dppe), available from a number of suppliers, is also an air-stable, white, crystalline solid that melts at 143–145 °C. dppe is soluble in a range of solvents including CH_2Cl_2, Et_2O, C_6H_6, and acetone. Like pfpe, dppe is also insoluble in alcohols and alkanes.

The ^{31}P NMR spectrum of pfpe in acetone is a multiplet centered at −44.5 ppm (from 85% H_3PO_4). This is considerably more shielded than dppe (−12.5 ppm) and similar to the shielding of phosphorus in 1,2-bis(dimethylphosphino)ethane (−49.4 ppm). The two most important variables in determining ^{31}P chemical shifts are the electronegativity of the substituents and the substituent–phosphorus–substituent bond angles.[7,8] Consideration of both of these variables for pfpe would lead to the expectation that pfpe would be more deshielded than dppe. Specifically, (1) the electronegativity of the C_6F_5 group (somewhat more electronegative than Br)[9] would lead to more deshielding and (2) the larger C_6F_5 group would require an opening up of the substituent–phosphorus–substituent bond angles (when compared to those in dppe), leading to a decrease in the s character of the phosphorus lone pair and consequently a deshielding effect on the phosphorus nucleus. Clearly neither of these arguments leads to agreement with the experimental results. Hogben and Graham have suggested[3] that phosphorus "lone pair donation to the C_6F_5 ring (a π(p-p) effect) is outweighed by back–donation to the phosphorus (a π(p-d) effect) in which the vacant phosphorus 3d orbitals accept electrons from the π cloud". This would be expected to result in an overall shielding of the phosphorus nucleus and implies that the phosphorus atom in pfpe is a stronger π acceptor than is that in dppe. It is not clear, however, how the two molecules would be expected to compare as ligands in this regard. Comparison of the ^{31}P chemical shift of pfpe with the ^{31}P chemical shifts of other organopentafluorophenylphosphines is given in Table I.

Table I. ^{19}F and ^{31}P Chemical Shifts[a] of Some Organophosphines and Organo(pentafluorophenyl)phosphines

	^{19}F[b]			
compd	ortho	meta	para	^{31}P[c]
$C_6F_5P(C_6H_5)_2$[d,e]	−127.7	−150.6	−161.0	−26.3
$(C_6F_5)_2PC_6H_5$[d,e]	−129.2	−150.5	−160.8	−49.7
$(C_6F_5)_3P$[d,e]	−130.6	−147.7	−159.8	−75.5
$C_6F_5P(CH_3)_2$[d,g]	−132.0	−154.2	−163.0	−47.8
$(C_6F_5)_2PCH_2CH_2P(C_6F_5)_2$	−132.1	−152.7	−163.1	−44.5
$(C_6H_5)_3P$[f]				−6.0
$(C_6H_5)_2PCH_2CH_2P(C_6H_5)_2$[h]				−12.5

[a] The chemical shift convention is that a minus sign denotes up upfield. [b] In ppm from $CFCl_3$. [c] In ppm from external 85% H_3PO_4. [d] ^{19}F values from ref 4. [e] ^{19}F values from ref 14. [f] ^{31}P values from ref 16. [g] ^{31}P and ^{19}F values from ref 16. [h] ^{31}P values from ref 19.

The ^{19}F NMR spectra of pfpe consists of three sets of multiplets resulting from the ortho, para, and meta fluorines. The chemical shift values are −132.1, −152.7, and −163.1 ppm, respectively (from $CFCl_3$ as an external standard). These values are comparable to the ^{19}F chemical shift values of other organopentafluorophenylphosphines (see Table I).

The mass spectrum of pfpe shows similarities to the mass spectra of $(C_6F_5)_3P$[10,11] and dppe.[12] pfpe gives a molecular ion peak at *m/e* 758. The most abundant ion is at *m/e* 365 and corresponds to $(C_6F_5)_2P^+$. The peak at *m/e* 532 can be attributed to $(C_6F_5)_3P^+$, which is generated by a C_6F_5 migration similar to the migration of a C_6H_5 group in dppe to form $(C_6H_5)_3P^+$.[12] The peaks with *m/e* below 300 can be accounted for by fragmentations of $(C_6F_5)_2P^+$.[10,11] In particular, the formation of the octafluorobiphenylene ion at *m/e* 296, which is characteristic of pentafluorophenylphosphines containing two or more C_6F_5 groups, was observed.[10]

Comparison of the mass spectra of pfpe with those of dppe shows many similarities although the intensities of the resulting fragments are not similar. The similarities are presumably due to the $-PCH_2CH_2P-$ backbone common to both ligands.

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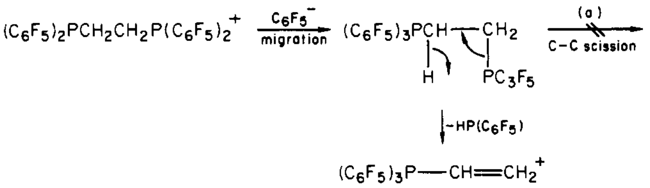
Scheme I



Similar to the formation of $(C_6H_5)_3P^+$ from dppe[12] is the formation of $(C_6F_5)_3P^+$ from pfpe. The peak at *m/e* 559 can be rationalized by C_6F_5 migration followed by loss of C_6F_5PH to give $(C_6F_5)_3P—CH{=}CH_2^+$. The C–C bond fission, which occurs at point a (Scheme I) in dppe to give $(C_6H_5)_3P{=}CH(C_6H_5)^+$, does not occur in pfpe to give the corresponding peak at *m/e* 545 for $(C_6F_5)_3P{=}CH(C_6F_5)^+$. The differences in the mass spectra of dppe and pfpe are presumably due to changes in the stability of the generated ions when F is substituted for H.

Known methods used to prepare organophosphine ligands from $RPCl_2$ precursors were expected to work in the syntheses of $R_2PCH_2CH_2PR_2$ from the $Cl_2PCH_2CH_2PCl_2$ precursors. Our recent synthesis of pfpe from $Cl_2PCH_2CH_2PCl_2$ using Grignard and metalation reactions and the recent syntheses of $R_2PCH_2CH_2PR_2$ (where R is $HOOCCH_2$–, CH_3O–, CH_3–, C_2H_5–, and cyclohexyl)[13,17,18] from $Cl_2PCH_2CH_2PCl_2$ have shown this to be the case.

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Registry No. pfpe, 76858-94-1; C_6F_5Br, 344-04-7; C_6F_5Li, 1076-44-4; $Cl_2PCH_2CH_2PCl_2$, 28240-69-9.

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Communications

Synthetic Models for the Iron-Sulfur Protein Rubredoxin: Synthesis, Structure, and Properties of a Highly Symmetric Iron(III) Tetrathiolate Anion

Sir:

Most Fe(III) and Cu(II) compounds react with thiolates to give disulfides; in the process the metal is reduced. There are several important metalloproteins in which the Fe(III)-to-cysteine and Cu(II)-to-cysteine ligation modes are stabilized with regard to autoredox reactions.[1] Herein, we report the use of a sterically hindered thiolate to prepare the first example of a stable iron(III) tetrathiolate complex containing only monodentate ligands, $(Et_4N)(Fe(SC_{10}H_{13})_4)$ (**1**).

The reaction of 4 equiv of lithium 2,3,5,6-tetramethylbenzenethiolate[2] directly with $FeCl_3$ and Et_4NBr or with $FeCl_2$ and Et_4NBr followed by controlled air oxidation gives in both cases high yields (80–90%) of **1**. Compounds **1** is thermally stable as evidenced by the fact that it is recrystallized without decomposition from hot (100 °C) DMF. The room-temperature electronic spectrum (in CH_3CN) of **1** exhibits bands at $\lambda = 295$ nm ($\epsilon = 14300$), 344 nm (6880), and 450 nm (7230).

Stable iron(III) tetrathiolate complexes are rare in spite of considerable efforts to prepare such compounds.[3] Besides the protein rubredoxin[4] the only other example is $(Fe(S_2\text{-}o\text{-}xyl)_2)^-$ (**2**), where the iron is chelated by two *o*-xylenedithiolate ligands.[5] Attempts to oxidize $(Fe(SC_6H_5)_4)^{2-}$ (**3**) to the Fe(III) derivative have not been successful.[6] A comparison of the Fe^{3+}/Fe^{2+} redox potential of **1** (–0.85 V vs. SCE), **2** (–1.0 V),[5] and **3** (–0.52 V)[7] shows the 3+ oxidation state to be more stable in **1** and **2** than it would be in the Fe(III) analogue of **3**. This result is consistent with the idea that electron-rich thiolates can stabilize highly charged metal ions. However, this effect is two-edged, since electron-rich thiolates are potential reducing reagents and could participate in the reduction of Fe(III).[8] The steric and conformational properties of the thiolate ligands are crucial in accounting for the stability of iron(III) tetrathiolate complexes.

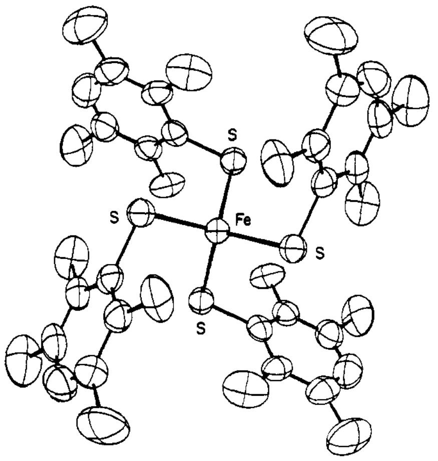

Figure 1. ORTEP diagram of $[Fe(SC_{10}H_{13})_4]^-$ viewed down the crystallographic S_4 axis. The Fe–S distance is 2.284 (2) Å.

An X-ray diffraction study of **1**[9] reveals the highly sym-

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