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A new stable germaphosphene and some of its chemical properties

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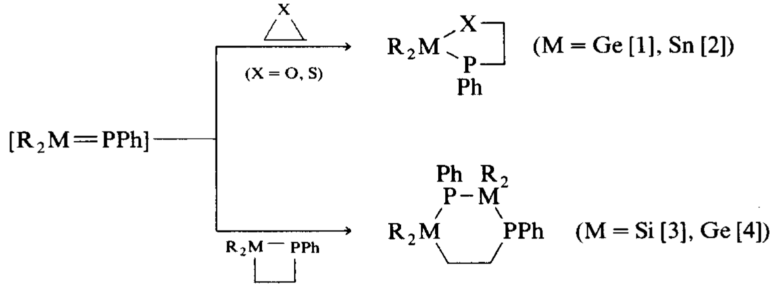
Abstract

A new stable germaphosphene, the 2,2-dimesityl-1-(2,4,6-triisopropylphenyl)germaphosphene (**1**), has been made. It reacts quantitatively with benzaldehyde and α-phenyl-*N*-tert-butylnitrone to afford, by [2+2]- and [2+3]-cycloadditions, respectively, the corresponding four- and five-membered heterocycles **10** and **13**. The conformations of these heterocycles are discussed. The reactions of **1** and the 2,2-dimesityl-1-(2,4,6-tri-tert-butylphenyl)germaphosphene (**2**) with these reagents are compared.

Introduction

Transient metallaphosphenes R_2M=P– (M = Si, Ge, Sn) were first detected by trapping reactions with oxirane and thiirane or by their insertion into the metal–phosphorus bond of the precursor 2-metallaphosphetane:



Although the stable metallaphosphenes R_2M=P– (M = Si [5], Ge [6], Sn [7]) synthesized until now are very reactive towards protic reagents [5–7], hydrides [7,8], lithium compounds [8,9] or disulfides [8,9], they seem reluctant, perhaps for steric reasons, to undergo cycloadditions: the sole reactions of this type are the [2 + 1]-cycloadditions with group 16 elements such as tellurium [5b,d], sulfur [5d,9,10] and selenium [5d,9,10].

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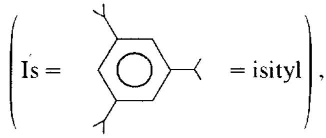
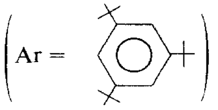
We describe here novel types of [2 + 2]- and [2 + 3]-cycloadditions between the germaphosphenes **1** and **2** and benzaldehyde or α-phenyl-*N*-tert-butylnitrone.

Mes$_2$Ge=PIs
(**1**)



R$_2$Ge=PAr



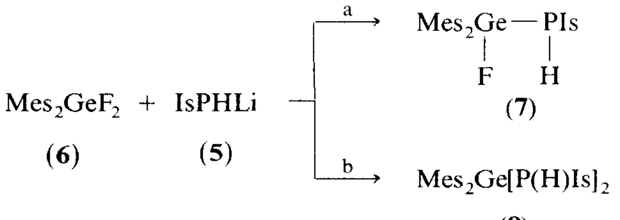
(R$_2$ = Mes$_2$ (**2**); Mes, ^{t}Bu (**3**); ^{t}Bu$_2$ (**4**))

Results and discussion

All the stable germaphosphenes synthesized up to now [6] had on the phosphorus the very bulky 2,4,6-tri-tert-butylphenyl group. In order to give a more reactive germaphosphene, particularly in cycloadditions, we used the 2,4,6-tri-isopropylphenyl group (isityl) on phosphorus; this slightly less bulky group should allow a better approach of reagents and yet provide the stabilization, which varies considerably even with small changes in the size of substituents round the double bond.

The best route to the previously synthesized germaphosphenes **2**–**4** was a two-step sequence involving the initial formation and isolation of fluorogermylphosphines R$_2$Ge(F)P(H)–, followed by a dehydrofluorination with tert-butyllithium. A procedure also involving the initial preparation of fluoro-precursors was used for the synthesis of stannaphosphene [7] and germenes [11].

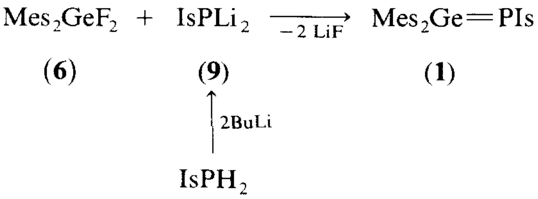
However, **1** could not be obtained by such a sequence, because addition of the lithio(isityl)phosphide (**5**) to dimesityldifluorogermane (**6**) gave the expected **7** only in poor yield; the major product was the diphosphinogermane **8**, in its two diastereoisomeric forms (route b) (**8**/**7** = 90/10).



$$\underset{(\mathbf{6})}{\mathrm{Mes_2GeF_2}} + \underset{(\mathbf{5})}{\mathrm{IsPHLi}} \begin{cases} \xrightarrow{a} \underset{(\mathbf{7})}{\mathrm{Mes_2Ge(F)\text{—}P(H)Is}} \\ \xrightarrow{b} \underset{(\mathbf{8})}{\mathrm{Mes_2Ge[P(H)Is]_2}} \end{cases}$$

7: δ (^{31}P): -146.8 (^2J(PF) 15.5, ^1J(PH) 205.0 Hz)
8: δ (^{31}P): **8a** -128.8 (^1J(PH) 207.0 Hz), **8b** -133.0 (^1J(PH) 205.6 Hz), **8a**/**8b** = 30/70

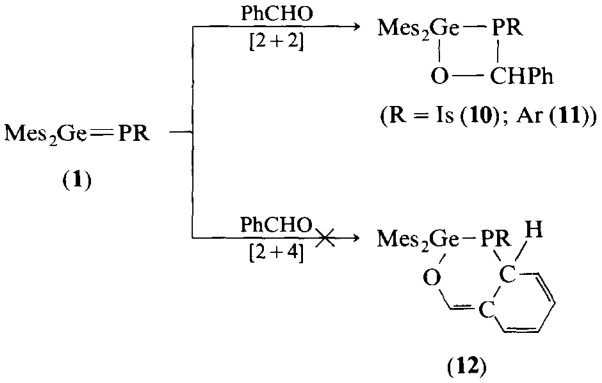
We prepared **1** by a one-step reaction between dimesityldifluorogermane (**6**) and isityldilithiophosphide (**9**), obtained from isitylphosphine and two equivalents of butyllithium at room temperature in Et$_2$O. This type of process, generally less clean than the two-step reaction, gave good results in our case, giving the germaphosphene **1** in high yield (85%):



$$\underset{(\mathbf{6})}{\mathrm{Mes_2GeF_2}} + \underset{(\mathbf{9})}{\mathrm{IsPLi_2}} \xrightarrow[-2\,\mathrm{LiF}]{} \underset{(\mathbf{1})}{\mathrm{Mes_2Ge{=}PIs}}, \qquad \mathrm{IsPH_2} \xrightarrow{\mathrm{2BuLi}} \mathrm{IsPLi_2}\ (\mathbf{9})$$

Compound **1** has not yet been isolated in pure form by crystallization, but its structure is unambiguously revealed by the characteristic δ (^{31}P) signal at +145.3 ppm (+175.4 for **2**, +157.2, +168.8 for **3**, and +158.5 for **4**). It is extremely air- and moisture-sensitive, but shows good thermal stability, since it is recovered unchanged after some days in solution at room temperature.

Germaphosphene **1** easily reacts with benzaldehyde by [2 + 2]-cycloaddition to give the corresponding four-membered heterocycle **10**. Germaphosphene **2** likewise gives **11**:



The first step of this reaction is probably the nucleophilic attack of oxygen on the germanium, and this is followed by the cyclisation. Although the Ge–P bond is only slightly polarized, a regioselective reaction takes place, with oxygen becoming bonded exclusively to germanium.

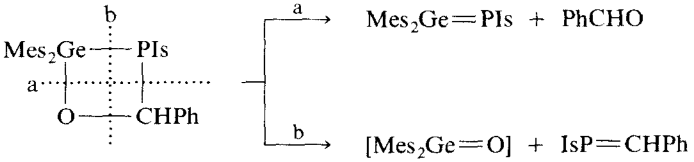
We obtained only the four-membered ring heterocycles **10** and **11**; the six-membered ring species **12** was not observed; it is noteworthy that such [2 + 4]-cycloadditions involving a carbonyl and a phenyl group have been reported in the reactions between benzophenone and some germenes $>$Ge=C$<$ [12] or silenes $>$Si=C$<$ [13].

In the case of **10** and **11**, only one diastereoisomer is obtained. For steric reasons this diastereoisomer has its phenyl and isityl groups in a *trans* disposition. This stereochemistry is also unambiguously confirmed by the ^2J(PCH) coupling constant. The magnitude of this coupling constant depends on the dihedral angle between the direction of the lone pair and of the CH, according to a Karplus type curve [14]. The value observed corresponds to a dihedral angle of about 145–150°, which arises only when hydrogen and phosphorus lone pair are trans.

The ^{1}H NMR spectrum shows two doublets for the methyls of the *o*-isopropyl groups of the isityl, as a consequence of the magnetic inequivalence of the two methyls of each isopropyl group.

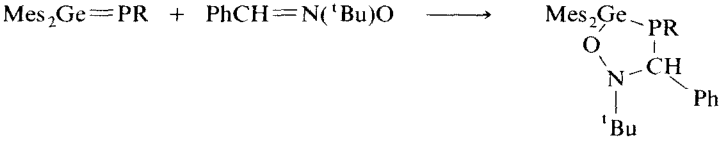
In ^{31}P NMR, the signal from **10** appears at much lower field than expected, viz. +45.6 ppm rather than −10 to −40 ppm expected for a tricoordinated phosphorus bonded to a germanium and two carbons. This could be due to substantial folding of the four-membered ring along the Ge–C axis, giving rise to an interaction between phosphorus and oxygen lone pairs. Such folding to relieve steric strain, particularly between bulky isityl and mesityl groups, is common in other four-membered ring species: for example in the germaoxetane $Mes_2GeOCHPhCR_2$ (CR_2 = fluorenylidene) [15], which, like **10**, contains the $Mes_2GeOCHPh$ moiety, the folding is 23.7° [15]. For compound **11** the signal is at +82.5 ppm: this lower field chemical shift relative to that for **10** could be due to a stronger interaction between phosphorus and oxygen lone pairs caused by a larger folding (2,4,6-tri-tert-butylphenyl is bulkier than 2,4,6-tri-isopropylphenyl).

In mass spectroscopy of **10**, besides the molecular peak, there are ions arising from two types of [2 + 2]-decomposition; route a, regenerating the starting material is the most important (a/b = 95/5), but route b, involving formation of phosphene IsP=CHPh and germanone Mes_2Ge=O confirms that **10** has oxygen bonded to germanium and not phosphorus:



As usual [15], the germanone Mes_2Ge=O gives an ion at *m/e* 329 instead of the expected 328, and it seems either that the germanone abstracts an hydrogen immediately after its formation or that **10** is protonated in the spectrometer ion source and gives Mes_2GeOH^+ and alkene directly.

Germaphosphene **1** also reacts with α-phenyl-*N*-tert-butylnitrone by [2 + 3]-cycloaddition to give the corresponding five-membered ring **13**:



(R = Is, (**13**) Ar (**14**))

In contrast to the outcome for **10**, two diastereoisomers are obtained: δ (^{31}P) **13a** −11.1 and **13b** −18.8 ppm in the ratio **13a**/**13b** 30/70. The ^{31}P NMR spectrum of **13a** shows a $^2J(PH)$ coupling constant of 20.8 Hz, compared with 4.0 Hz for **13b**. Thus **13b** is the diastereoisomer with phenyl and isityl in a *trans* disposition as in **10** and **13a** the diastereoisomer with phenyl and isityl in a *cis* disposition: in this case the dihedral angle between the phosphorus lone pair and the CH is, according to the Karplus type curve [14], around 20°, which is in agreement with the angle determined from molecular models.

The ratio **13a**/**13b** does not vary in solution, rules out a thermodynamic equilibrium. **13b** was obtained pure by fractional crystallization, and characterized by ^1H and ^{13}C NMR spectroscopy. As in the formation of **10** and **11** the first step of this regioselective reaction is probably the nucleophilic attack of germanium by oxygen.

We also observed a reaction between germaphosphene **2** and the nitrone, to give the cycloadduct **14**, but the [2 + 3]-cycloaddition is much more difficult than with **1** and is incomplete at room temperature. Because of the very large steric hindrance by the 2,4,6-tri-tert-butylphenyl group, only one diastereoisomer is observed, giving rise to a peak in the ^{31}P NMR spectrum at +11.0 ppm.

The new germaphosphene **1** although thermally stable, appears to be very reactive in cycloaddition reactions and is markedly more reactive than germaphosphene **2**. Its reactions with various reagents are being studied.

Experimental

All the syntheses were performed by use of high-vacuum line techniques and carefully deoxygenated solvents, which must be freshly distilled from sodium benzophenone. ^{1}H NMR spectra were recorded on Bruker AC 80 and AC 200 at 80.1 and 200.1 MHz spectrometers, respectively. ^{13}C NMR spectra were recorded on Bruker AC 200 and AC 250 spectrometers at 50.3 and 62.9 MHz (TMS internal standard) and ^{31}P NMR spectra on a Bruker AC 80 spectrometer at 32.4 MHz (H_3PO_4 85% as external standard). Mass spectra (EI) were obtained with a Nermag R10 010 spectrometer at 70 eV. Melting points were determined with a Reichert apparatus. Elemental analyses were performed by the "Service de Microanalyse de l'Ecole de Chimie de Toulouse" (France).

2,4,6-Tri-isopropylphenyldichlorophosphine (isityldichlorophosphine) was made as previously described by reaction of phosphorus trichloride with isityllithium or isitylmagnesium bromide. It was obtained pure by crystallization from pentane; m.p. 90–100 ° C [17]. NMR (C_6D_6) δ (^{31}P): +165.0 [18]. δ (^{1}H): 1.10 (d, ^3J(HH) 6.9 Hz, 3H, p-CHMe_2); 1.23 (d, ^3J(HH) 6.8 Hz, 6H, o-CHMe_2); 2.65 (sept., ^3J(HH) 6.9 Hz, 1H, p-CHMe$_2$); 4.17 (sept. d, ^3J(HH) 6.8, ^4J(PH) 4.4 Hz, 2H, o-CHMe$_2$); 7.06 (d, ^4J(PH): 3.4 Hz, 2H, arom. H). δ (^{13}C) ($CDCl_3$): 23.68 (p-CHMe_2); 24.77 (o-CHMe_2); 30.69 (d, ^3J(PC) 27.5 Hz, o-CHMe$_2$); 34.55 (p-CHMe$_2$); 122.96 (d, ^3J(PC) 3.6 Hz, m-C Is); 131.73 (d, ^1J(PC) 69.1 Hz, i-C Is); 154.20 (p-C Is); 154.53 (d, ^2J(PC) 22.7 Hz, o-C Is).

Isitylphosphine, IsPH$_2$, was obtained by reduction of isityldichlorophosphine with lithium aluminium hydride as described previously [17].

Synthesis of germaphosphene 1

2,4,6-Tri-isopropylphenyldilithiophosphide (**9**) was prepared by addition of two equivalents of n-butyllithium 1.6 *M* in hexane to a solution of isitylphosphine (0.60 g, 2.5 mmol) in Et$_2$O (30 ml), followed by 1 h stirring at room temperature. The yellow-orange solution of IsPLi$_2$ was added to a suspension of dimesityldifluorogermane (**6**) (0.87 g, 2.5 mmol) in Et$_2$O (20 ml) at −78 ° C. The mixture was allowed to warm to room temperature. After 1 h stirring at 20 ° C, the ^{31}P NMR spectrum of the crude solution indicated the nearly quantitative formation of the expected germaphosphene **1** (δ (^{31}P): 145.3 ppm).

Solutions of **1** can be used for reactions without further purification.

Synthesis of 10

A solution of germaphosphene **1** prepared as described above from 0.60 g (2.50 mmol) of IsPH$_2$, 0.87 g (2.50 mmol) of Mes$_2$GeF$_2$, 2 equivalents of n-butyllithium 1.6 *M* in hexane, and 30 ml of Et$_2$O, was added to a solution of benzaldehyde (0.27 g, 2.50 mmol) in Et$_2$O (5 ml) at −30 ° C. The yellow-orange solution rapidly turned light yellow. After 1 h stirring at room temperature, solvents were evaporated *in vacuo*, and pentane (20 ml) was added to the residue. Lithium fluoride was removed by filtration; cooling at −20 ° C led to crystallization of **10**: white crystals, m.p.: 64–68 ° C (dec.), 0.83 g, 51% yield. NMR (C_6D_6) δ (^{1}H): 0.97 (d, ^3J(HH) 6.6 Hz, 6H, o-CHMe_2); 1.19 (d, ^3J(HH) 6.8 Hz, 6H, p-CHMe_2); 1.24 (d, ^3J(HH) 6.7 Hz, 6H, o-CHMe_2); 1.99 (s, 3H, p-Me); 2.14 (s, 3H, p-Me); 2.38 (s, 6H, o-Me); 2.73 (s, 6H, o-Me); 2.73 (sept., ^3J(HH) 6.6 Hz, 1H, p-CHMe$_2$); 3.26 (qqd, ^3J(HH) 6.7 and 6.8

Hz, ^4J(PH) 1.8 Hz, 2H, *o*-C*H*Me$_2$); 6.55 (d, ^2J(PH) 2.2 Hz, 1H, OCH); 6.56 (s, 2H, arom. Mes); 6.60 (s, 2H, arom. Mes); 7.04 (d, ^4J(PH) 2.4 Hz, arom. Is), 6.90–7.78 (m, 5H, Ph). δ (^{13}C): 20.96 (*p*-Me); 21.22 (*p*-Me); 22.99 (*o*-Me); 23.00 (d, ^4J(PC) 8.1 Hz, *o*-CH*Me*$_2$); 23.98 (*o*-Me); 24.17 (d, ^4J(PC) 5.3 Hz, *o*-CH*Me*$_2$); 25.38 (*p*-CH*Me*$_2$); 34.50 (*p*-*C*HMe$_2$); 35.12 (d, ^3J(PC) 8.2 Hz, *o*-*C*HMe$_2$); 85.55 (d, ^1J(PC) 15.0 Hz, OCH); 121.73 (*m*-C Is); 126.76 (d, ^3J(PC) 7.8 Hz, *o*-C Ph); 127.39 and 128.56 (*m*-C and *p*-C Ph); 129.51 (*m*-C Mes); 129.81 (*m*-C Mes); 131.73, 134.84, 139.50 (d, *J*(PC) 10.0 Hz); 139.72, 139.98, 142.51 and 144.50 (*o*- and *p*-C Mes, *i*-Ph, Mes and Ar); 147.14 (d, ^3J(PC) 18.4 Hz, *o*-C Mes); 148 (*p*-C Is); 151.17 (d, ^2J(PC) 8.2 Hz, *o*-C Is). δ (^{31}P) 45.6. MS (EI, 70 eV, ^{74}Ge): 652 (*M*, 5), 546 (Mes$_2$Ge=PIs, 4), 426 (MesGe=PIs − 1, 3), 311 (Mes$_2$Ge − 1, 3), 233 (IsP + 1, 1), 192 (MesGe − 1, 4), 106 (PhCHO, 19) 43 (^{i}Pr, 100)

Synthesis of 11

To a solution of germaphosphene **2** (1.35 g, 2.30 mmol) in Et$_2$O (20 ml) was added a solution of benzaldehyde (0.24 g, 2.30 mmol) in Et$_2$O. The initially orange solution rapidly turned yellow. After evaporation of Et$_2$O, crude **11** was recrystallized from pentane to give 1.05 g of yellow crystals (78% yield); m.p.: 143–146 °C (dec.). NMR (CDCl$_3$) δ (^{1}H): 1.30 (s, 18H, *o*-^{t}Bu); 1.38 (s, 9H, *p*-^{t}Bu); 2.00 (s, 6H, *o*-Me); 2.24 (s, 3H, *p*-Me); 2.28 (s, 3H, *p*-Me); 2.51 (s, 6H, *o*-Me); 5.62 (d, ^2J(PH) 1.5 Hz, 1H, OCH); 6.64 (s, 2H, arom. Mes); 6.81 (s, 2H, arom. Mes); 7.03–7.44 (m, 5H, Ph); 7.18 (d, ^4J(PH) 3.0 Hz, 2H, arom. Ar). δ (^{13}C): 21.17, 23.02, 23.38 and 23.52 (*o*- and *p*-Me); 31.55 (*p*-C*Me*$_3$); 33.76 (d, ^3J(PC) 7.6 Hz, *o*-C*Me*$_3$); 34.47 (*p*-*C*Me$_3$); 39.27 (*o*-*C*Me$_3$); 89.37 (d, ^1J(PC) 18.2 Hz, OCH); 122.64 (*m*-C Ar); 126.81, 127.89, 127.99, 128.93, 129.20 and 129.49 (*o*-, *m*- and *p*-Ph, *m*-C Mes); 130.89, 135.46, 139.04, 139.64, 139.88 (d, *J*(PC) 11.6 Hz); 142.06, 144.27 (*o*- and *p*-C Mes, *i*-Ph, Mes and Ar); 145.26 (d, ^3J(PC) 18.7 Hz, *o*-C Mes); 148.45 (*p*-C Ar); 154.21 (d, ^2J(PC) 5.3 Hz, *o*-C Ar). δ (^{31}P): +82.5. MS (EI, 70 eV, ^{74}Ge): 588 (Mes$_2$Ge=PAr, 4), 531 (Mes$_2$Ge=PAr − ^{t}Bu, 2), 468 (MesGe=PAr − H, 1), 329 (Mes$_2$Ge=O + 1, 1), 313 (Mes$_2$Ge + 1, 15), 312 (Mes$_2$Ge, 10), 106 (PhCHO, 91), 105 (PhCO, 100).

Synthesis of 13

A Et$_2$O/hexane solution containing 2.50 mmol of germaphosphene **1**, prepared as described above, was added to a solution of α-phenyl-*N*-tert-butylnitrone (0.44 g, 2.50 mmol) in Et$_2$O (15 ml) at −30 °C. The mixture was allowed to warm to room temperature then stirred for 1 h. The solvents evaporated off *in vacuo* and replaced by 20 ml of pentane. LiF was removed by filtration; crystallization in pentane at low temperature afforded 0.62 g (35% yield) of **13b**; m.p.: 178–180 °C. NMR (C$_6$D$_6$) δ (^{1}H): 0.77 (d, ^3J(HH) 6.7 Hz, 6H, *o*-CH*Me*$_2$); 1.01 (d, ^3J(HH) 6.9 Hz, 3H, *p*-CH*Me*); 1.03 (d, ^3J(HH) 6.9 Hz, 3H, *p*-CH*Me*); 1.16 (s, 9H, ^{t}Bu); 1.50 (d, ^3J(HH) 6.7 Hz, 6H, *o*-CH*Me*$_2$); 1.99 (s, 3H, *p*-Me); 2.09 (s, 3H, *p*-Me); 2.21–2.73 (very broad s, 6H, *o*-Me); 2.51 (sept., ^3J(HH) 6.9 Hz, 1H, *p*-C*H*Me$_2$); 3.03 (s, 6H, *o*-Me); 4.21 (sept, ^3J(HH) 6.7 Hz, 2H, *o*-C*H*Me$_2$); 5.19 (d, ^2J(PH) 4.0 Hz, 1H, NCH); 6.54 (broad s, 2H, arom. Mes); 6.70–7.22 (m, 5H, Ph); 6.80 (s, 2H, arom. Mes); 7.03 (d, ^4J(PH) 2.3 Hz, 2H, arom. Is). δ (^{13}C): 20.94 (*p*-Me), 20.99 (*p*-Me), 23.76 (d, ^4J(PC) 3.5 Hz, *o*-CH*Me*$_2$); 25.06, 25.17, 25.36 and 26.12 (*p*-CH*Me*$_2$ and *o*-Me); 26.27 (C*Me*$_3$); 33.70 (*p*-*C*HMe$_2$); 34.20 (d, ^3J(PC) 10.3 Hz, *o*-*C*HMe$_2$);

62.66 (d, ^3J(PC) 7.9 Hz, CMe$_3$); 72.03 (d, ^1J(PC) 10.3 Hz, CHP); 122.53 (d, ^3J(PC) 3.9 Hz, *m*-C Is); 126.95, 127.01, 128.00 (*o*, *m*- and *p*-C Ph); 130.00 (*m*-C Mes); 130.04 (*m*-C Mes); 123.97, 124.92, 136.08, 136.48, 136.59, 138.75, 139.16, 144.02 (*o*- and *p*-C Mes, *i*-Ph, Mes and Is); 142.94 (d, ^2J(PC) 18.0 Hz, *o*-C Is); 150.57 (d, ^4J(PC) 1.4 Hz, *p*-C Is); 157.05 (d, ^3J(PC) 15.3 Hz, *o*-C Mes). δ (^{31}P): −18.8.

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