Electronic Supplementary Information for the paper entitled:

A Polychloride-enabled Synthesis of $[NEt_3Me][PCl_6]$ Serving as a Potential PCl_3-Storage and PCl_5-Reagent

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Part 1: Synthetic procedures

1. General Information

1.1. Materials

$[NEt_3Me][Cl_3]$ has been prepared by the standard procedure.[1] All commercially available reagents were used as received unless mentioned otherwise. Solvents were dried over molecular sieves (3 Å) and degassed by three freeze-pump-thaw cycles. All reactions with air- and moisture-sensitive compounds were performed under an argon atmosphere using standard Schlenk techniques.

1.2. Physical Measurements

The NMR spectra were recorded on the following spectrometers: *JEOL ECS 400* (^{1}H: 399.7 MHz, ^{31}P: 161.8 MHz) and *JEOL JNM-ECA400II* (^{1}H: 400.5 MHz, ^{31}P: 162.1 MHz). The NMR samples were measured at room temperature unless otherwise stated. Chemical shifts (δ) are given relative to the signals of the external standards TMS (^{1}H, ^{13}C), 85% phosphoric acid (^{31}P). MestReNova 14.1.1 was used to process the NMR spectra.[2] Electrospray ionization mass spectrometry (ESI-MS) was carried out with the ESI-MSD TOF unit of an *Agilent 6210 TOF LC/MS* system. The measurements were performed in acetonitrile. Raman spectra were recorded at room temperature on a Bruker MultiRAM II equipped with a low-temperature Ge detector (1064 nm, up to 450 mW, resolution 4 cm^{-1}).

1.3. Crystal Structure Determination

Single crystal X-ray diffraction data were collected on a *Bruker D8 Venture* with Mo K_α radiation. Absorption corrections were carried out by the multiscan method.[3,4] Structure solution and refinement were performed with the SHELX program package.[5,6] The representation of molecular structures was done using the program DIAMOND 4.2.2[7] Some remaining crystallographic problems are commented on in the respective *.cif* file.

1.4. Precautions

White phosphorus is toxic and extremely pyrophoric. Special safety precautions must be considered: work in pairs, available and ready to use fire extinguisher and sand. Traces of white phosphorus can be quenched by an aqueous $CuSO_4$ solution.

$[NEt_3Me][Cl_3]$ releases chlorine in contact with water. Traces of the ionic liquid are quenched with $Na_2S_2O_3$ solution. Teflon cannulas are used to avoid reactions with metal of cannulas. Avoid contact of $[NEt_3Me][Cl_3]$ with acetone, as this could react to chloroacetone.

2. Synthesis of $[NEt_3Me][PCl_6]$ from P_4

Method A: 988 mg (4.44 mmol, 5 equiv., 817 µL) $[NEt_3Me][Cl(Cl_2)]$ was added to a suspension of 55 mg (444 µmol, 0.5 equiv.) P_4 in 15 mL CH_2Cl_2 at −20 °C. While warming up to room temperature a colourless suspension was obtained which was stirred overnight. The suspension was filtered in the cold and the residue was washed two times with cold CH_2Cl_2 to remove the $[NEt_3Me][Cl]$. After drying the residue in vacuum, a colourless powder of $[NEt_3Me][PCl_6]$ is obtained. Yield: 59% (375 mg, 1.04 mmol, based on P_4). Single crystals of $[NEt_3Me][PCl_6]$ suitable for X-ray diffraction were obtained from a concentrated solution in CH_2Cl_2 within several days by slowly cooling to −20 °C.

^{31}P NMR (CD_3CN, rt): −297.7 ppm (s).[Fig. S2]

^{1}H NMR (CD_3CN, rt): 3.23 (q, $^3J_{HH}$ = 7.29 Hz, 6H, $(H_3C)N(CH_2CH_3)$), 2.84 (s, 3H, $(H_3C)N(CH_2CH_3)$), 1.25 ppm (tt, $^3J_{HH}$ = 7.29 Hz, $^3J_{NH}$ = 1.98 Hz, 9H, $(H_3C)N(CH_2CH_3)$).[Fig. S3]

$^{13}C\{^1H\}$ NMR (CD_3CN, rt): 56.6 (s, 3C, $(H_3C)N(CH_2CH_3)$), 47.4 (s, 1C, $(H_3C)N(CH_2CH_3)$), 8.1 ppm (s, 3C, $(H_3C)N(CH_2CH_3)$).[Fig. S4]

Raman (rt): $\tilde{\nu}$ = 2989 (m), 2951 (m), 1458 (w), 685 (m), 360 (vs), 283 (s), 239 cm^{-1} (m).[Fig. S5]

ESI-TOF (negative mode): m/z = 132.8924 ($PCl_2O_2^-$, calculated: 132.90131).[Fig. S6] The molecular ion peak of $[PCl_6]^-$ is not observed due to the moisture and air sensitivity of the compound. Therefore, only the molecular ion peaks for oxidation products are observed.

Quantitative NMR:

Due to the high sensitivity towards air and moisture, no CHN-elemental analysis could be performed. The composition of $[NEt_3Me][PCl_6]$ is shown by quantitative NMR with a calibrated PPh_3-capilary (19.7 µmol) (Figure S9/S10). 32.8 mg of $[NEt_3Me][PCl_6]$ are dissolved in 0.65 mL CD_3CN and the capillary is added. Integration of the ^{31}P NMR (relaxation delay 60 s) gives the ratio 1 : 4.50 for PPh_3 : $[PCl_6]^-$ resulting in 88.9 µmol (21.7 mg) $[PCl_6]^-$. Integration of the ^1H NMR gives the ratio 15 : 14.12 for $P(C_6H_5)_3$: $[(H_3C)N(CH_2CH_3)]^+$ resulting in 92.8 µmol (10.8 mg) $[NEt_3Me]^+$. The product is therefore contaminated with 3.95 µmol of $[Cl]^-$ (140 µg) yielding a purity of $[NEt_3Me][PCl_6]$ of 96%.

Method B: 593 mg (2.66 mmol, 5 equiv., 0.49 mL) $[NEt_3Me][Cl(Cl_2)]$ was added to a suspension of 33 mg (266 µmol, 0.5 equiv.) P_4 in 15 mL CH_2Cl_2 at −20 °C. While warming up to room temperature a colourless suspension was obtained which was stirred overnight. The suspension was concentrated to 3 mL and pentane (15 mL) was added to complete the precipitation of the product. After filtration, the residue was dried in vacuum and a colourless powder of the molar composition $[NEt_3Me]_5[PCl_6]_2[Cl]_3$ is obtained. Yield: 92% (577 mg, 532 µmol, based on P_4). This method can be used for further reactions of $[NEt_3Me][PCl_6]$ due to the higher yield and no hindrance of the reactivity by the Cl^--anions.

3. Synthesis of $[NEt_3Me][PCl_6]$ starting from PCl_3

673 mg (3.02 mmol, 1 equiv., 0.56 mL) $[NEt_3Me][Cl(Cl_2)]$ was added to a solution of 415 mg (3.02 mmol, 1 equiv.) PCl_3 in 8 mL CH_2Cl_2 at room temperature. After stirring for 72 h at rt all volatile substances were removed. The residue was dried in vacuum and $[NEt_3Me][PCl_6]$ was obtained as a colourless powder. Yield: 57% (617 mg, 1.71 mmol).

4. Synthesis of $PCl(OPh)_4$ starting from $[NEt_3Me][PCl_6]$

20.0 mg (55.6 µmol, 1 equiv.) $[NEt_3Me][PCl_6]$ and 20.9 mg (222 µmol, 5 equiv.) phenol were dissolved in 0.7 mL CD_2Cl_2 at room temperature. Directly after the addition the $^{31}P\{^1H\}$ and ^1H NMR measurements show full conversion of the starting materials. After removing all volatiles, $PCl(OPh)_4$ is separated from the formed ionic liquid $[NEt_3Me][Cl(HCl)_n]$ by extraction with pentane. Subsequent removal of all volatiles from the extract afforded a colourless solid. The NMR shifts match with the reporting literature.[8,9]

$PCl(OPh)_4$:

$^{31}P\{^1H\}$ NMR (CD_2Cl_2, rt): −23.7 ppm (s).[Fig. S13]

^1H NMR (CD_2Cl_2, rt): 7.57 – 7.54 (m, 8H, $(PCl(OC_6H_5))$), 7.51 – 7.47 (m, 4H, $(PCl(OC_6H_5))$), 7.28 – 7.26 ppm (m, 8H, $(PCl(OC_6H_5))$).[Fig. S14]

$[NEt_3Me](Cl(HCl)_n]$:

^1H NMR (CD_2Cl_2, rt): 11.44 (s, 1.5H, $[Cl(HCl)_{1.5}]^-$, 3.40 (q, $^3J_{HH}$ = 7.28 Hz, 6H, $(H_3C)N(CH_2CH_3)$), 3.03 (s, 3H, $(H_3C)N(CH_2CH_3)$), 1.36 ppm (tt, $^3J_{HH}$ = 7.28 Hz, $^3J_{NH}$ = 1.93 Hz, 9H, $(H_3C)N(CH_2CH_3)$).[Fig. S14]

5. Synthesis of adamantyl acid chloride starting from $[NEt_3Me][PCl_6]$

30 mg (83.4 µmol, 1 equiv.) $[NEt_3Me][PCl_6]$ and 15.0 mg (83.4 µmol, 1 equiv.) adamantyl carbonyl acid were dissolved in 0.7 mL $CDCl_3$ at room temperature. Directly after the addition the $^{31}P\{^1H\}$ and ^1H NMR measurements show full conversion of the starting materials. The NMR shifts match with the reporting literature.[9,10]

$POCl_3$:

$^{31}P\{^1H\}$ NMR ($CDCl_3$, rt): −4.7 ppm (s).Fig. S16

AdC(O)Cl:

^1H NMR ($CDCl_3$, rt): 2.02 (m, 3H, C_{tert}), 1.91 (m, 6H, C_{sec}), 1.65 ppm (m, 6H, C_{sec}).Fig. S17

$[NEt_3Me](Cl(HCl)_n]$:

^1H NMR ($CDCl_3$, rt): 12.63 (s, 0.5H, $[Cl(\mathit{H}Cl)_{0.5}]^-$, 3.48 (q, $^3J_{HH}$ = 7.30 Hz, 6H, $(H_3C)N(C\mathit{H}_2CH_3)$), 3.11 (s, 3H, $(\mathit{H}_3C)N(CH_2CH_3)$), 1.32 ppm (t, $^3J_{HH}$ = 7.16 Hz, 9H, $(H_3C)N(CH_2C\mathit{H}_3)$).Fig. S17

6. Synthesis of hexachlorophosphazene starting from $[NEt_3Me][PCl_6]$

138 mg (385 µmol, 3 equiv.) $[NEt_3Me][PCl_6]$ and 20.6 mg (385 µmol, 3 equiv.) ammonium chloride were suspended in 8 mL chlorobenzene and heated under reflux for 2 hours. After removing all volatiles, the hexachlorophosphazene can be separated from the ionic liquid $[NEt_3Me](Cl(HCl)_n]$ by extraction with pentane and subsequent removal of all volatiles as colourless solid. The NMR shifts match with the reporting literature.[9,11]

$(N{=}PCl_2)_3$:

$^{31}P\{^1H\}$ NMR ($CDCl_3$, rt): 20.0 ppm (s).Fig. S18

$[NEt_3Me](Cl(HCl)_n]$:

^1H NMR ($CDCl_3$, rt): 12.83 (s, 0.6H, $[Cl(\mathit{H}Cl)_{0.6}]^-$, 3.37 (q, $^3J_{HH}$ = 7.29 Hz, 6H, $(H_3C)N(C\mathit{H}_2CH_3)$), 3.01 (s, 3H, $(\mathit{H}_3C)N(CH_2CH_3)$), 1.24 ppm (t, $^3J_{HH}$ = 7.21 Hz, 9H, $(H_3C)N(CH_2C\mathit{H}_3)$).Fig. S19

7. Direct synthesis of PCl_3 from P_4 and $[NEt_3Me][Cl_3]$

7.81 g (30.3 mmol, 1 equiv., 6.46 µL) $[NEt_3Me][Cl(Cl_2)_{1.5}]$ and 938 mg (7.57 mmol, 0.25 equiv.) P_4 are mixed at −20 °C. The suspension is heated in a 120 °C oil bath and PCl_3 collected during the reaction by trap-to-trap condensation at 10^{-3} mbar (reaction setup see: Fig. S23) Yield: 74% (3.07 g, 22.3 mmol, based on P_4).

PCl_3:

$^{31}P\{^1H\}$ NMR (neat, rt): 217.6 ppm (s).Fig. S22

8. Release of PCl_3 from $[NEt_3Me][PCl_6]$ in dichloromethane

2.13 g (6.42 µmol) $[NEt_3Me][PCl_6]$ are suspended in 25 mL CH_2Cl_2 and the rection mixture is heated to reflux for 2.5 hours. The resulting orange suspension is cooled down to room temperature. Afterwards, the formed PCl_3 along with CH_2Cl_2 are separated from the residue by trap-to-trap condensation. PCl_3 forms azeotropic mixtures with chlorinated solvents ($CHCl_3$, CH_2Cl_2, $C_2H_4Cl_2$). PCl_3 is received in a yield of 50% (443 mg, 3.23 mmol, determined by quantitative ^{31}P NMR) as a stock-solution in CH_2Cl_2. To obtain pure PCl_3 from $[NEt_3Me][PCl_6]$, the reaction can be performed neat starting from P_4 (see 8.). The orange solid after condensation contains $[NEt_3Me][Cl]$ and red phosphorus (P_{red}).Fig. S20 By washing with CH_2Cl_2

P_{red} is separated and analysed by Raman spectroscopy which matches with the reporting literature.[12]

P_{red}:

Raman (rt): $\tilde{\nu}$ = 599 (m), 503 (m), 456 (w), 384 (s), 356 (s), 213 cm^{-1} (w).Fig. S21

Part 2: Crystallographic data

Table S1. Crystal data and structure determination parameters.

	$[NEt_3Me][PCl_6]$
Empirical formula	C_7Cl_6NP
Formula weight	341.75
Temperature/K	100
Crystal system	cubic
Space group	Pm-3m
a/Å	7.2009(9)
b/Å	7.2009(9)
c/Å	7.2009(9)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	373.4(1)
Z	1
ρ_{calc}/g cm^{-3}	1.520
μ/mm^{-1}	1.226
F(000)	166
Crystal size/mm^3	0.256 × 0.114 × 0.093
Radiation	Mo K_α (λ = 0.71073)
Θ range for data collection/°	5.658 to 49.878
Index ranges	-8 ≤ h ≤ 8, -7 ≤ k ≤ 8, -8 ≤ l ≤ 8
Reflections collected	3228
Independent reflections	95 [R_{int} = 0.0690, R_{sigma} = 0.0128]
Data/restraints/parameters	95/0/17
Goodness-of-fit on F^2	1.233
Final R indexes [I>=2σ (I)]	R_1 = 0.0552, wR_2 = 0.1497
Final R indexes [all data]	R_1 = 0.0663, wR_2 = 0.1698
Largest diff. peak/hole / e Å^{-3}	0.31/-0.41
Diffractometer	Bruker D8 Venture
CCDC access code	2379260

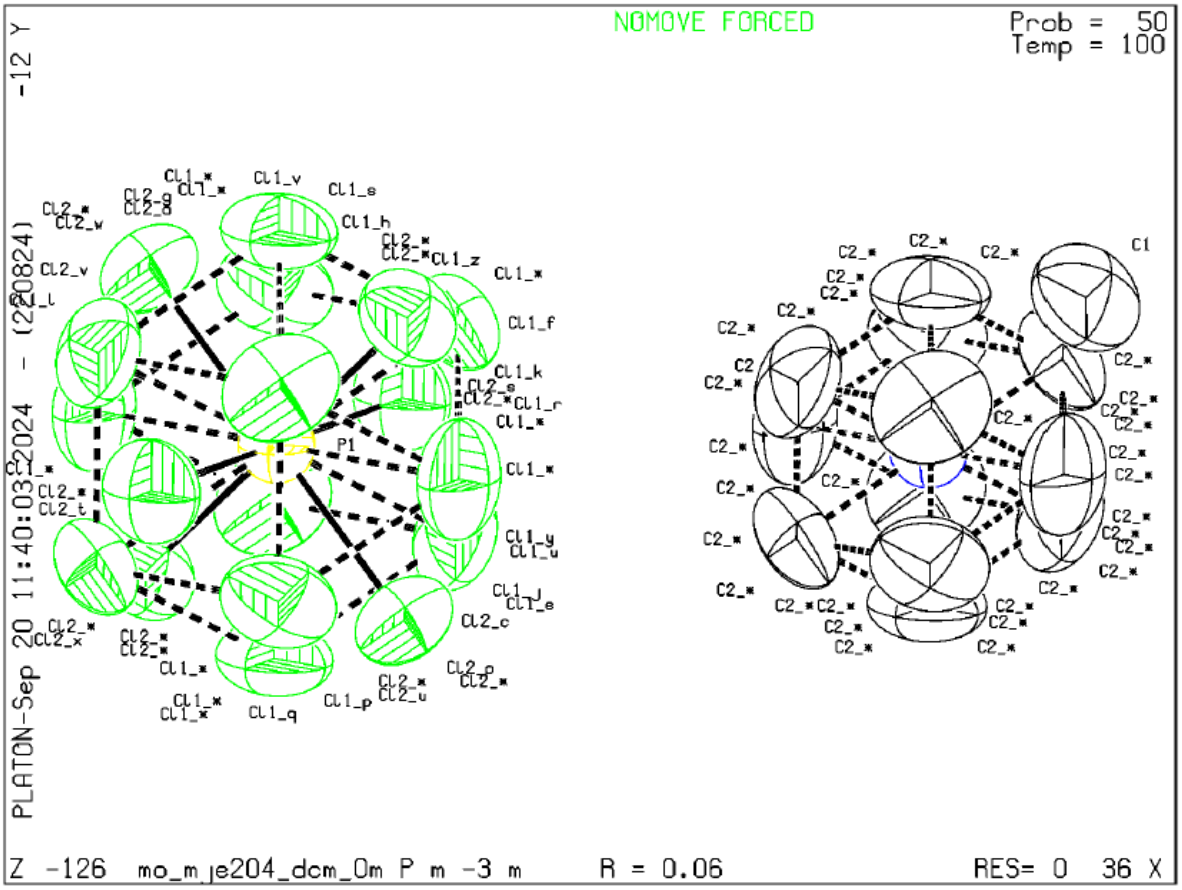

Figure S1. Ellipsoid representation (50% probability) of $[NEt_3Me][PCl_6]$.

Table S2. Selected bond lengths (Å) $[NEt_3Me][PCl_6]$.

Cl1−Cl1	2.11(2)
Cl1−P1	2.11(2)
Cl2−P1	2.119(3)
N1−C2	1.55(2)
C1−C2	1.81(2)

Explanation of the refinement of the structure of $[NEt_3Me][PCl_6]$

Within the molecular structure of $[NEt_3Me][PCl_6]$ both the anion and the cation are heavily disordered. Whilst the cation disorder is a well-documented phenomenon, the anion disorder is most likely caused by very low intermolecular interactions. The comparably large sizes of anion and cation, in combination with a very delocalized charge of the anion, results in a pseudo-binary CsCl type packing that is only caused by coulombic interactions without preferential orientation of the anion.

No twinning was found. Neither reciprocal space analysis, nor LEPAGE, nor attempts for manual twin law refinements were successful and the list of most disagreeable reflections does not indicate a wrong refinement model. We have remeasured further crystals with Cu radiation, however, no improvement on the refinement could be obtained – most likely due to the generally observed low crystal quality.

With regard to the $[NEt_3Me]^+$ cation, the statistical disorder of ethyl and methyl fragments, superimposed by the (CH_2) and (CH_3) carbon disorder renders any H-treatment purely cosmetic. Therefore, hydrogen atoms where not treated in the structure solution.

The anion has not been treated with individual part assignment as the refinement is not stable upon assigning PART -1 to Cl1. Manual change of the Cl1 position from Wyckoff 12j and subsequent part assignment yields stable refinement and proper part assignment, however, the associated model contains 36 positions of Cl1. Since no physical meaning can be attributed to such an (anisotropic) model, we omitted the part assignment and employed the presented refinement.

Consideration of the disorder of the $[PCl_6]^-$ anion

Within the molecular structure of $[NEt_3Me][PCl_6]$ there are only two symmetry inequivalent Cl positions (Cl1 and Cl2). Due to the symmetry of the crystal system there is a total of 18 different Cl positions in the unit cell (12 times Cl1 and 6 times Cl2. These 18 Cl positions result in 4 different orientations for the $[PCl_6]^-$ anions. Within the main orientation (Figure S2 A) the 6 Cl2 positions are occupied with an occupation number of 0.625. The other three orientations (Figure S2 B,C,D) consist of 4 Cl1 and 2 Cl2 atoms with an occupation number of 0.125 each. This results in a total occupation number of 0.75 for Cl2 and 0.125 for Cl1. A representation of the color-coded superposition of the four different orientations is shown in Figure S3. For all different orientations only 90° Cl-P-Cl angles and equal P-Cl distances (*R*(P1-Cl1) = 2.11(2) Å, *R*(P1-Cl2) = 2.119(3) Å), within the margin of error, resulting in an octahedral geometry.

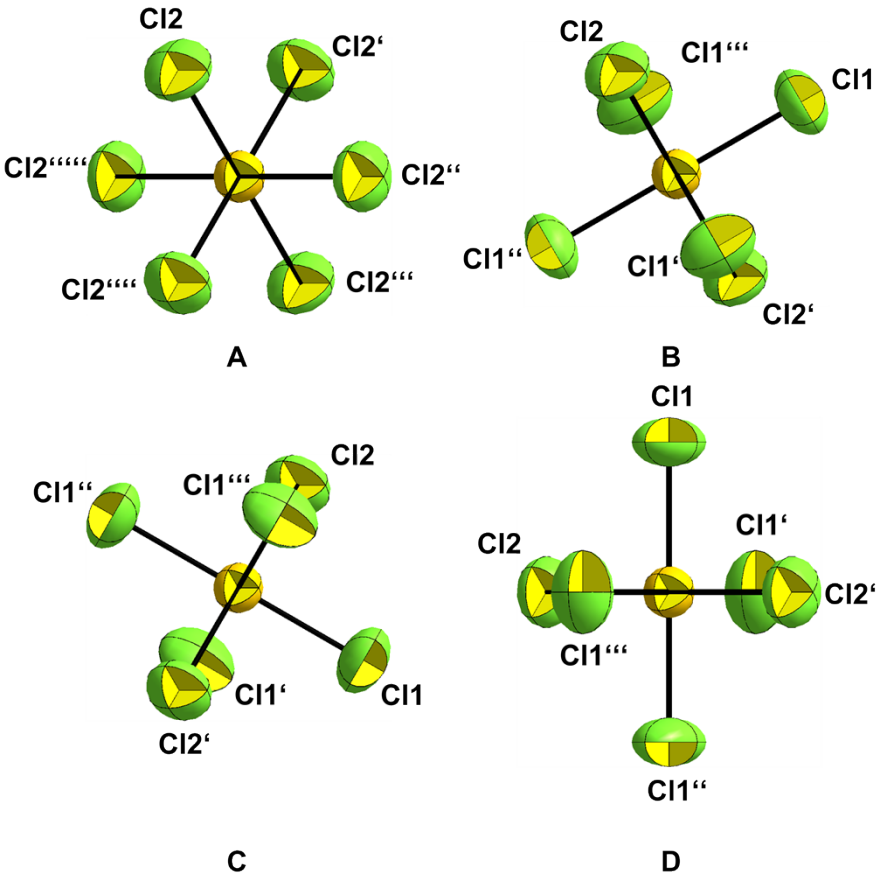

Figure S2. Representation of the four different orientations of the $[PCl_6]$ anion within the molecular structure of $[NEt_3Me][PCl_6]$. Orientation **A** has an occupation number of 0.625 while B, C and D have an occupation number of 0.125. The different geometries are shown with a viewing direction towards the (1,1,1) plane.

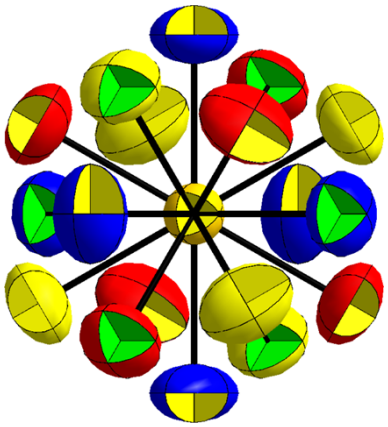

Figure S3. Color-coded superposition of the 4 different orientations of the $[PCl_6]^-$ anion within the molecular structure of $[NEt_3Me][PCl_6]$. **A** green inner octants, **B** yellow octants, **C** red octants, **D** blue octants.

Part 3: Spectral data

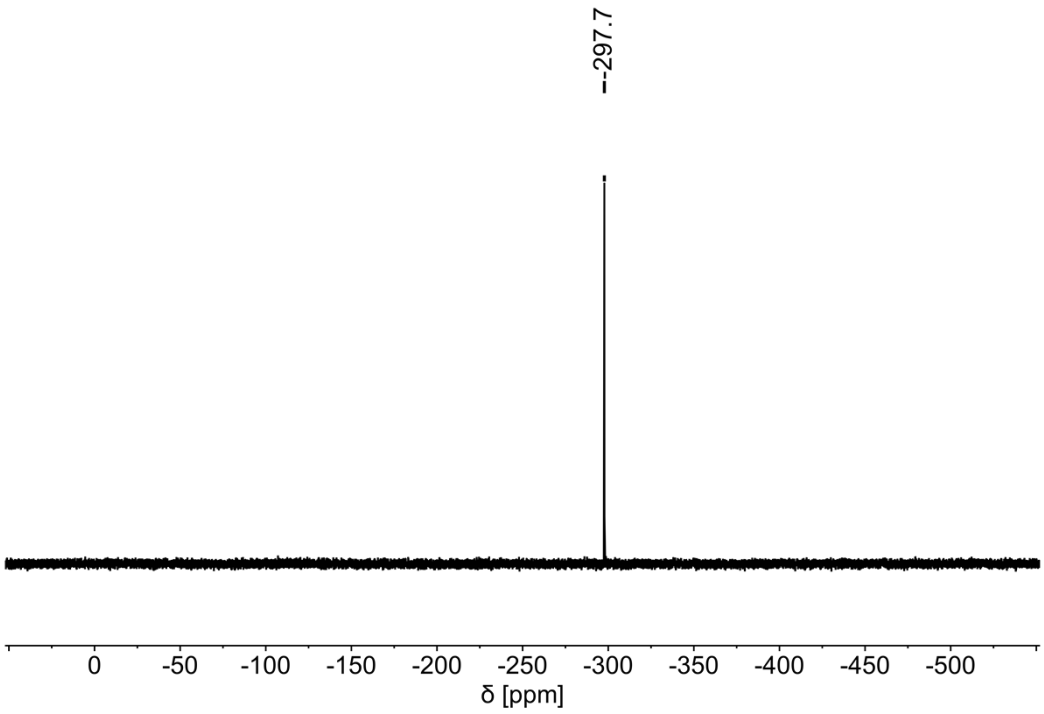

Figure S4. ^{31}P NMR spectrum ($NCCD_3$) of $[NEt_3Me][PCl_6]$.

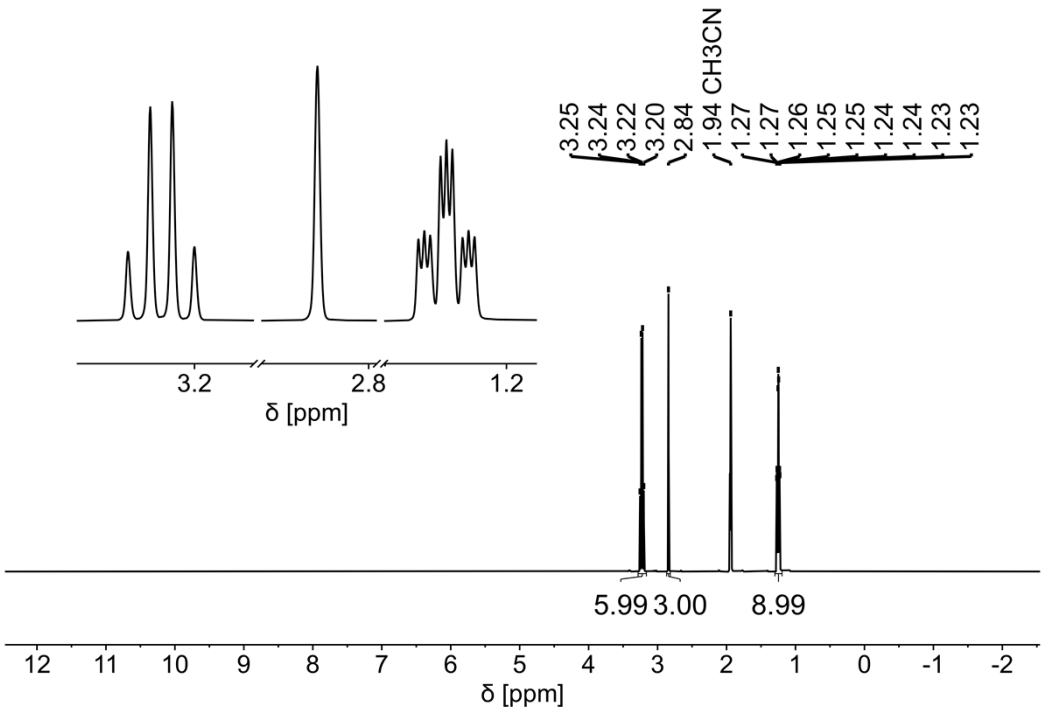

Figure S5. ^{1}H NMR spectrum ($NCCD_3$) of $[NEt_3Me][PCl_6]$.

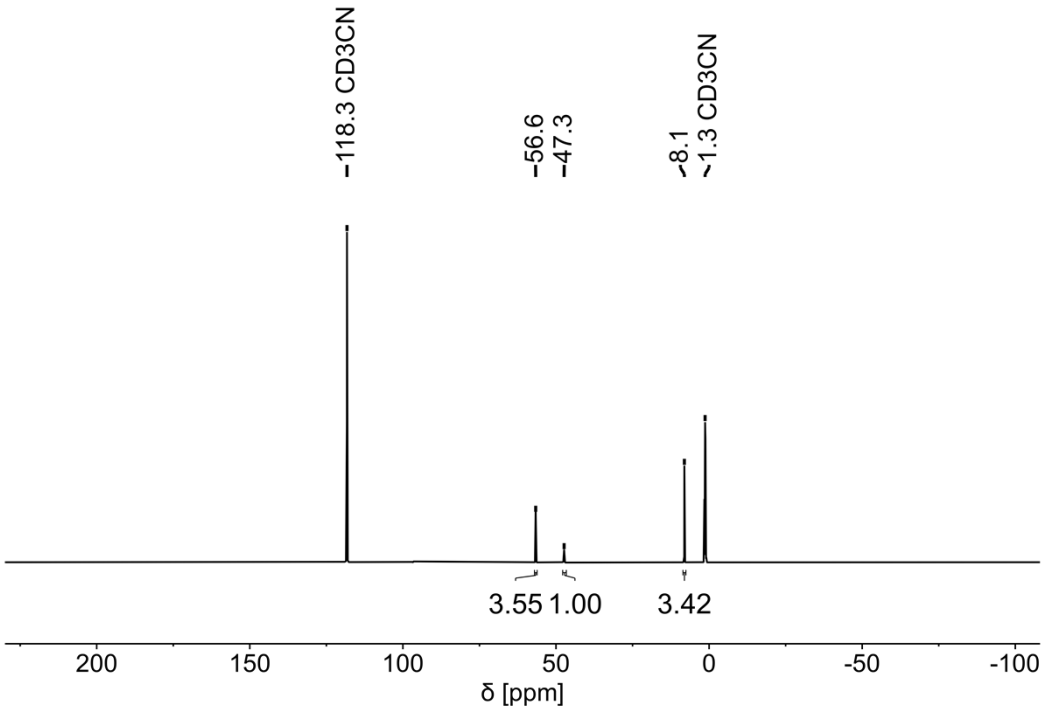

Figure S6. $^{13}C\{^{1}H\}$ NMR spectrum ($NCCD_3$) of $[NEt_3Me][PCl_6]$.

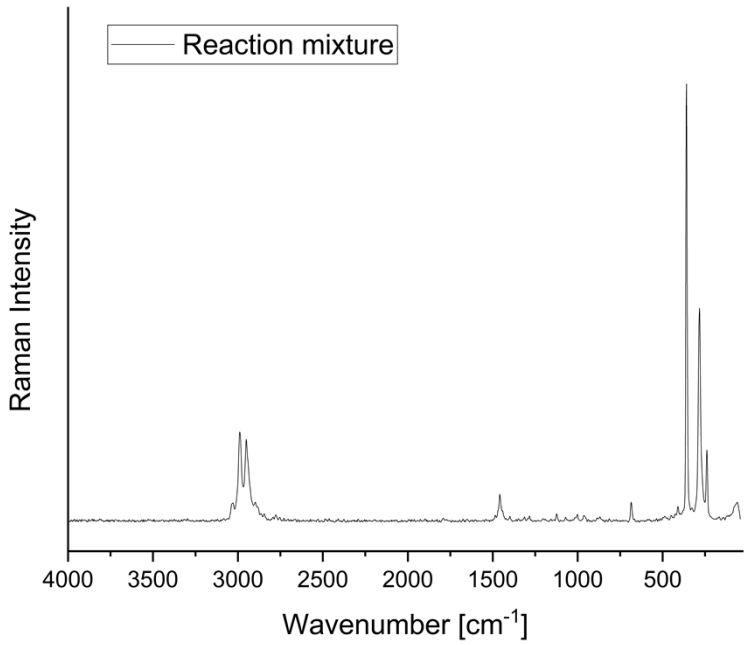

Figure S7. Experimental Raman spectrum of $[NEt_3Me][PCl_6]$ recorded at room temperature.

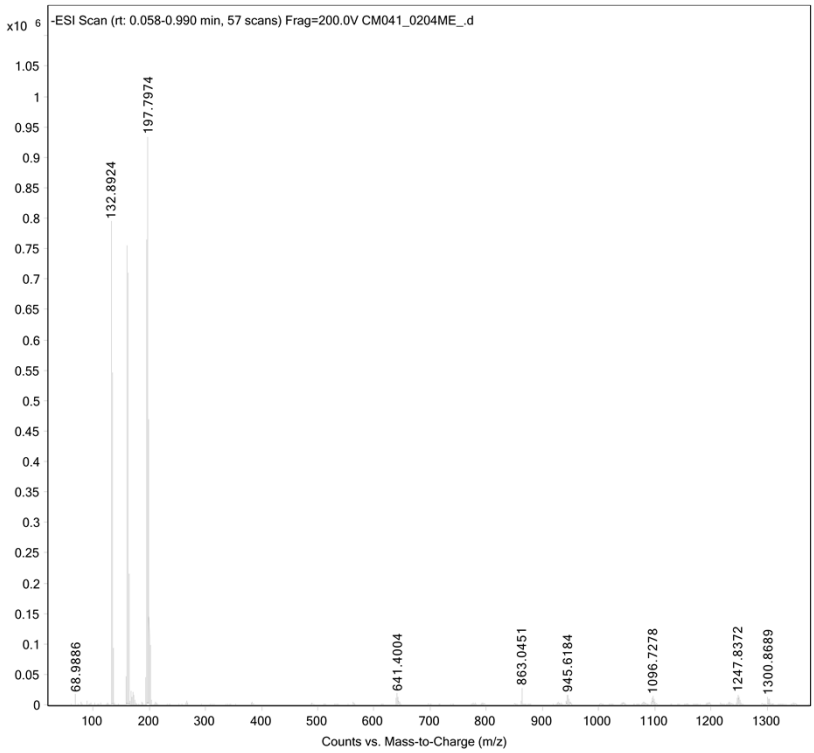

Figure S8. ESI$^-$ MS spectrum of $[NEt_3Me][PCl_6]$.

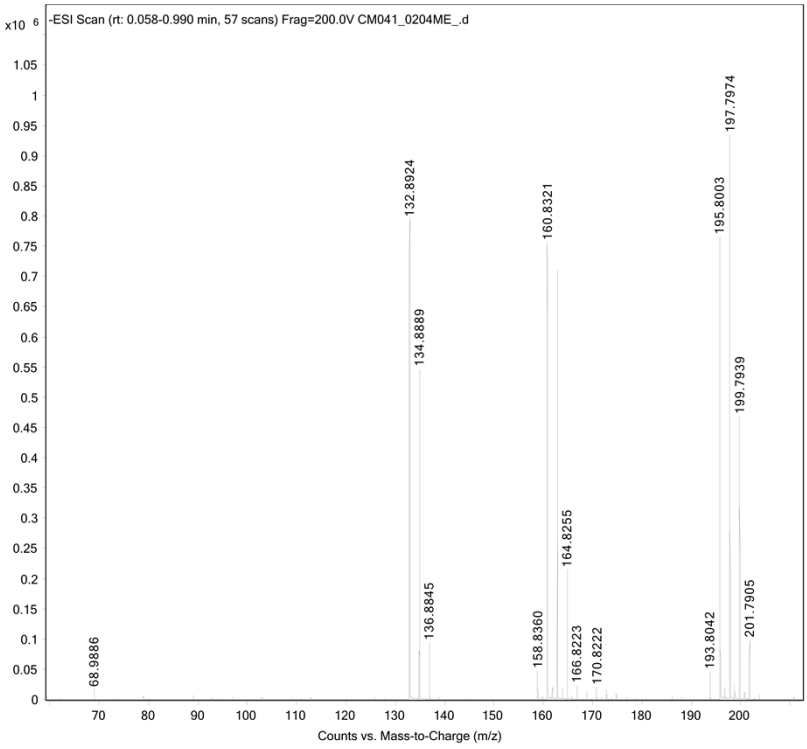

Figure S9. ESI$^-$ MS spectrum (zoom) of $[NEt_3Me][PCl_6]$.

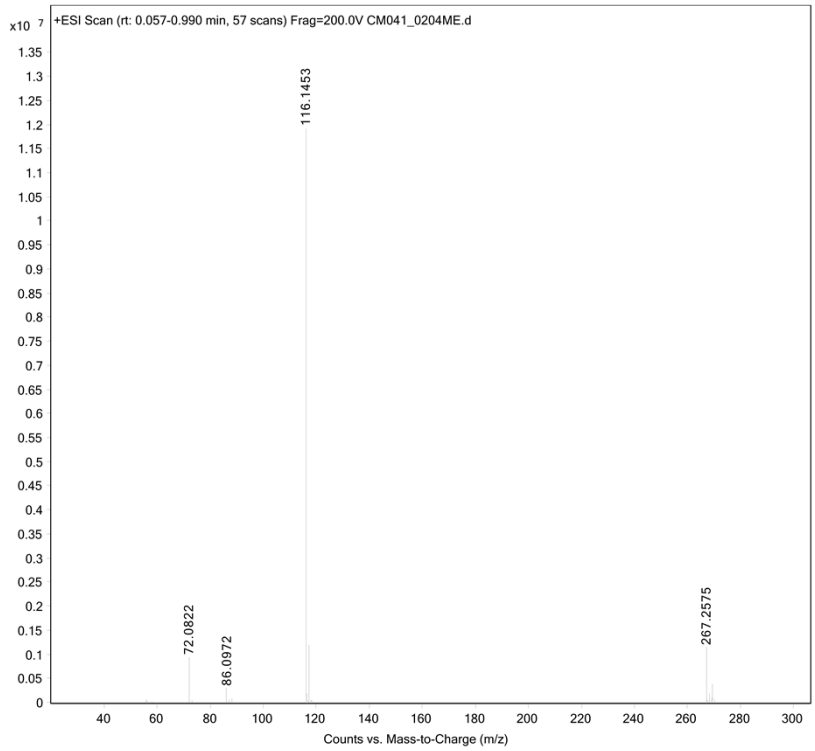

Figure S10. ESI^+ MS spectrum of $[NEt_3Me][PCl_6]$.

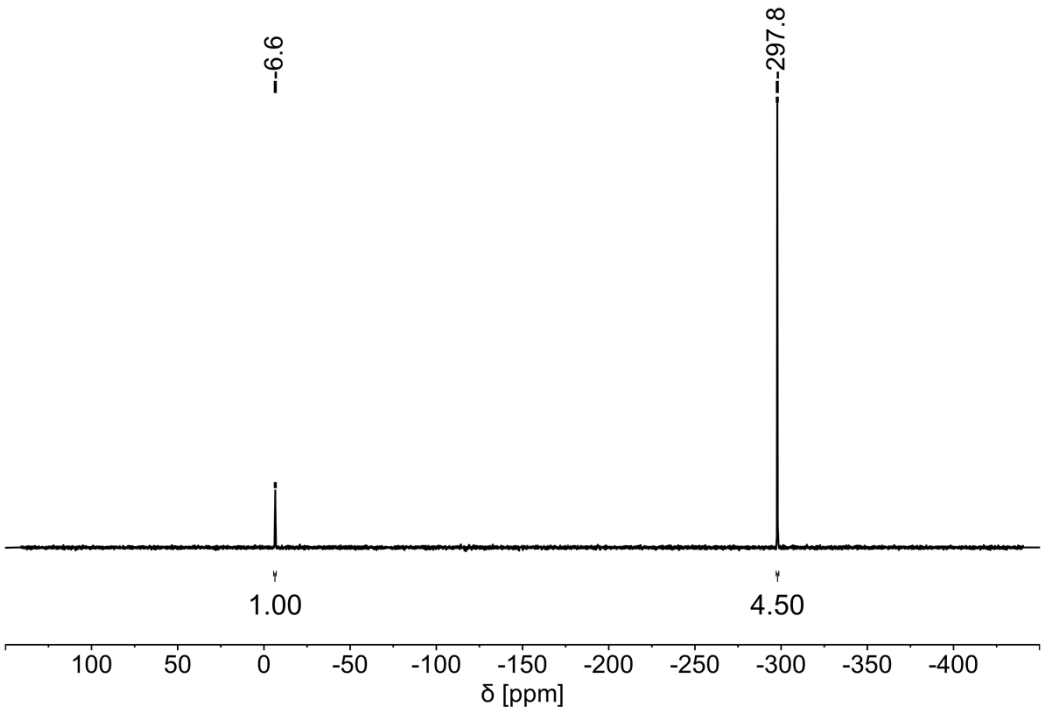

Figure S11. Quantitative ^{31}P NMR spectrum ($NCCD_3$) of $[NEt_3Me][PCl_6]$ with a PPh_3-capilary (19.7 µmol).

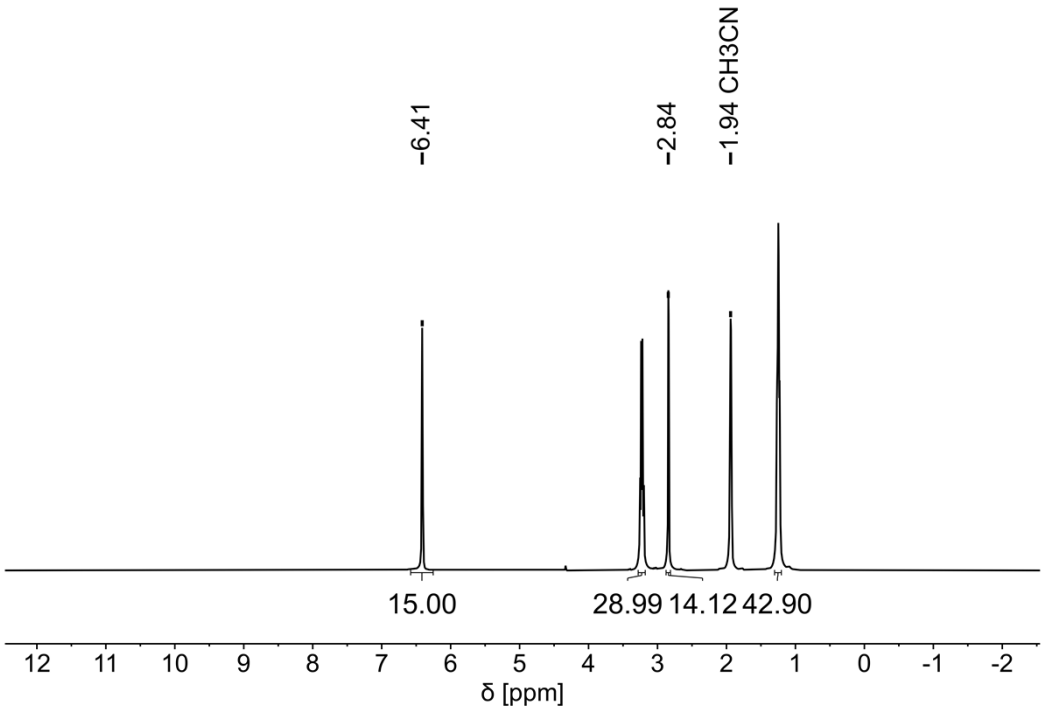

Figure S12. Quantitative ^{1}H NMR spectrum ($NCCD_3$) of $[NEt_3Me][PCl_6]$ with a PPh_3-capilary (19.7 µmol).

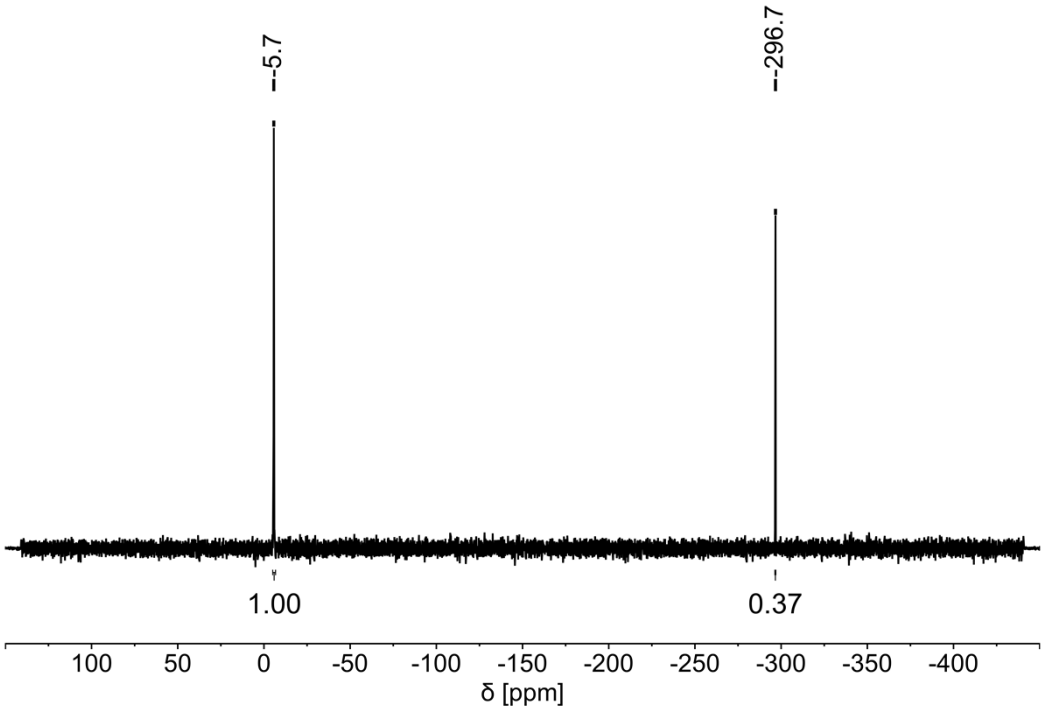

Figure S13. Quantitative ^{31}P NMR spectrum (CD_2Cl_2) of the CH_2Cl_2 washing solution with a PPh_3-capilary (19.7 µmol).

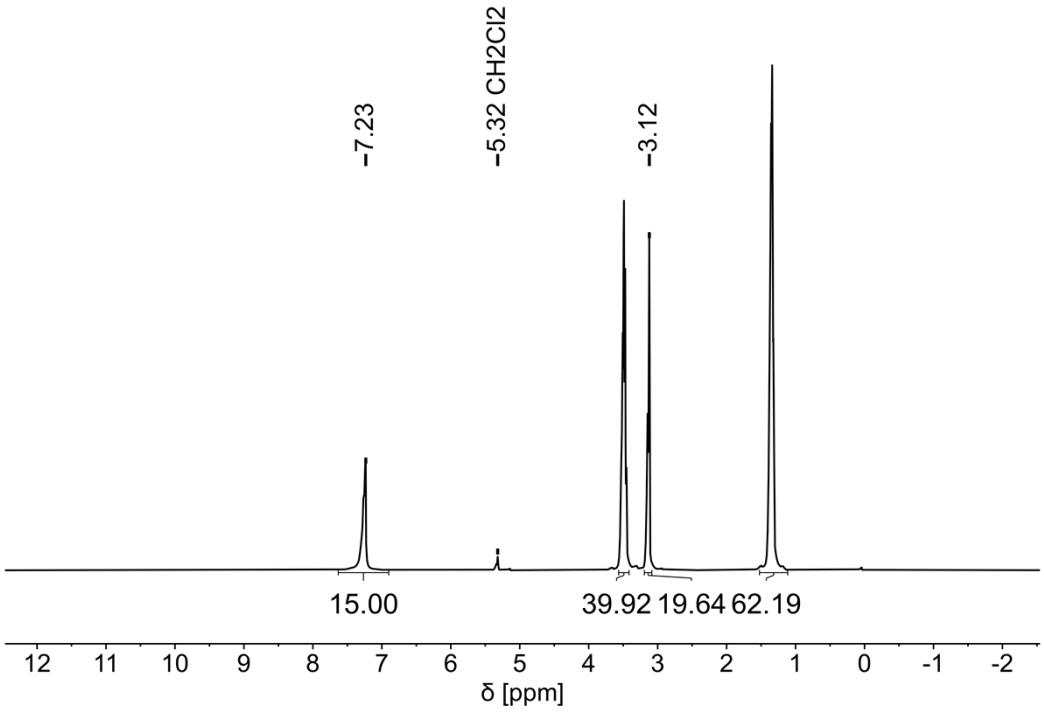

Figure S14. Quantitative ^{1}H NMR spectrum (CD_2Cl_2) of the CH_2Cl_2 washing solution with a PPh_3-capilary (19.7 µmol).

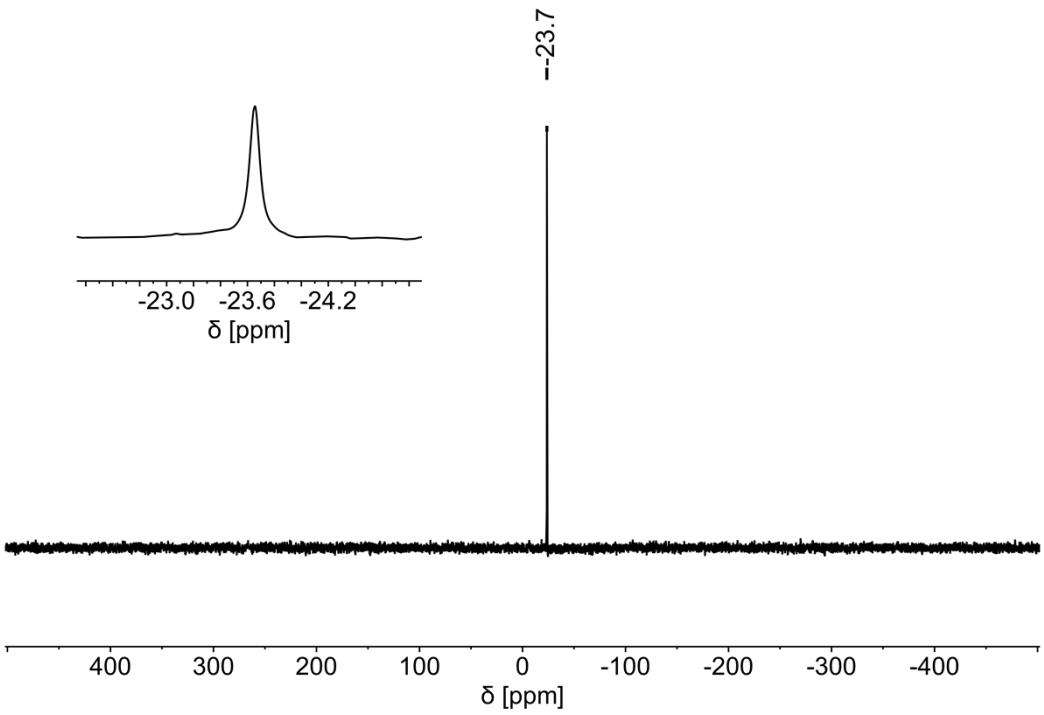

Figure S15. $^{31}P\{^{1}H\}$ NMR spectrum (CD_2Cl_2) of $PCl(OPh)_4$ synthesized from $[NEt_3Me][PCl_6]$ and phenol.

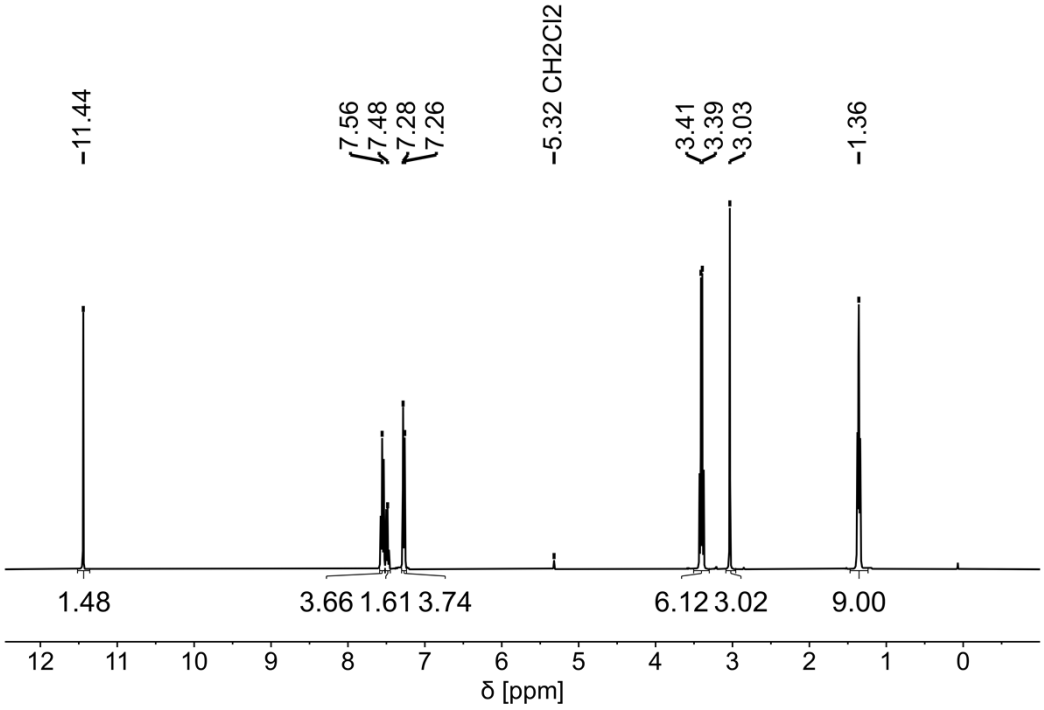

Figure S16. ^{1}H NMR spectrum (CD_2Cl_2) of $PCl(OPh)_4$ and $[NEt_3Me](Cl(HCl)_n]$ synthesized from $[NEt_3Me][PCl_6]$ and phenol.

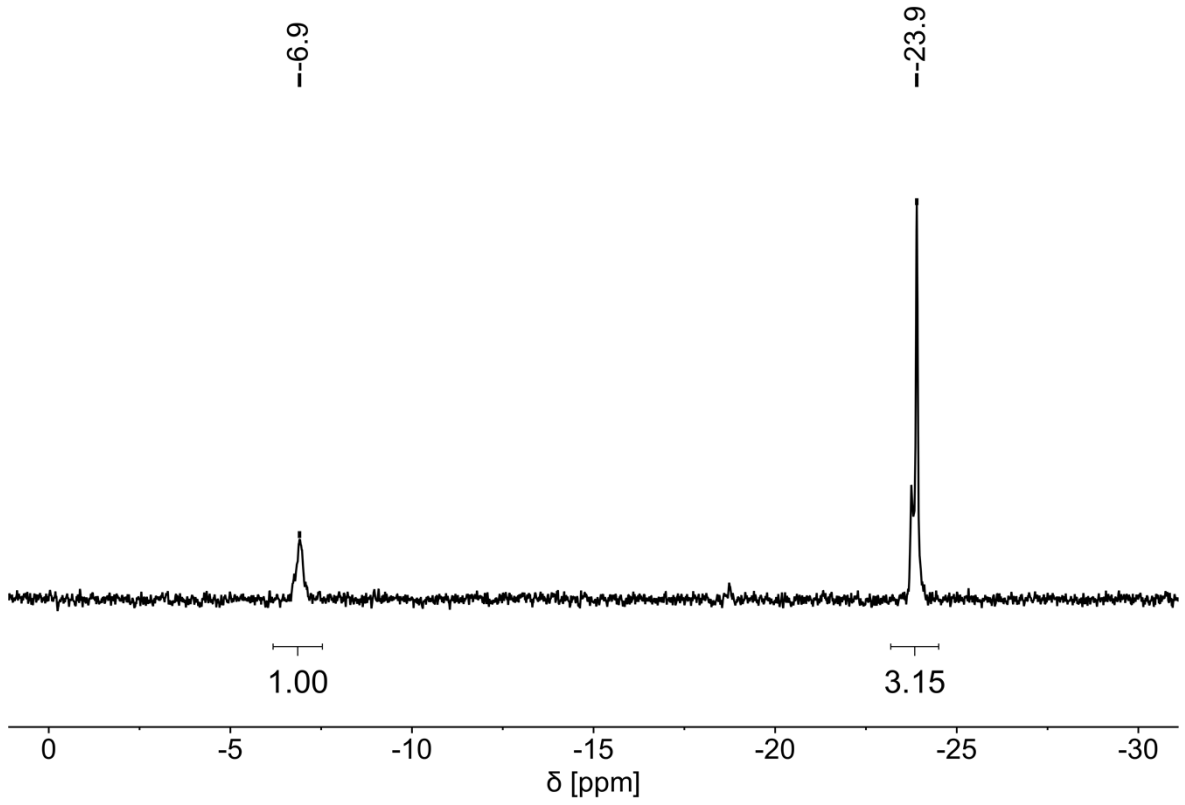

Figure S17. Quantitative ^{31}P NMR spectrum (CD_2Cl_2) of $PCl(OPh)_4$ synthesized from $[NEt_3Me][PCl_6]$ and phenol with a PPh_3-capilary (12.1 µmol).

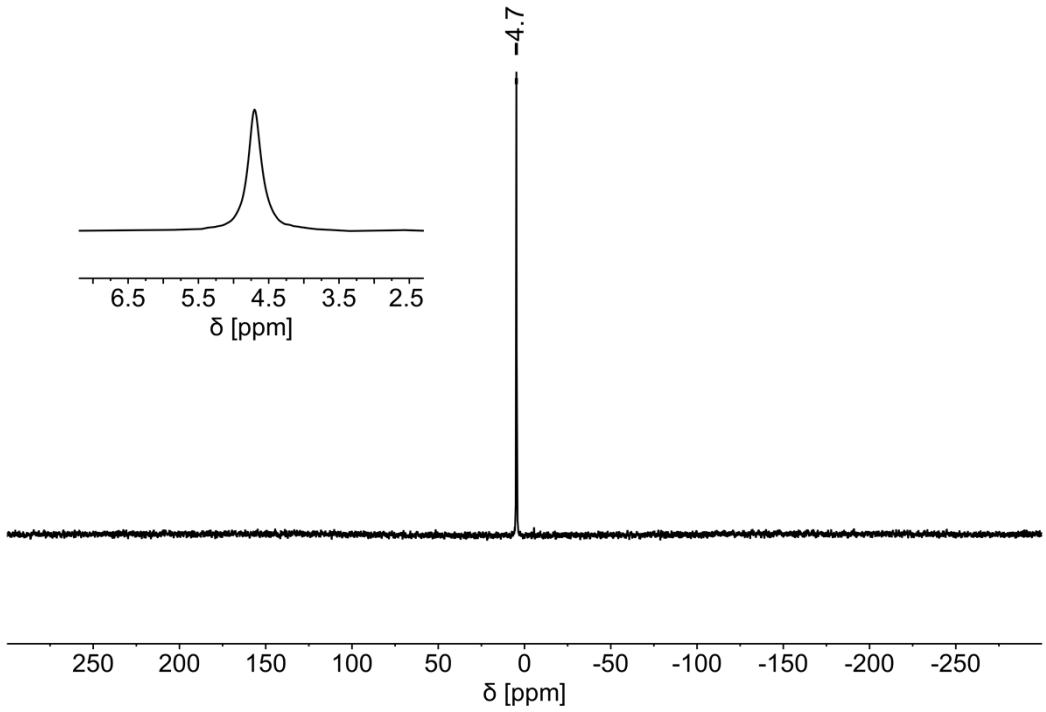

Figure S18. $^{31}P\{^{1}H\}$ NMR spectrum (CD_2Cl_2) of $POCl_3$ synthesized from $[NEt_3Me][PCl_6]$ and adamantyl carboxylic acid.

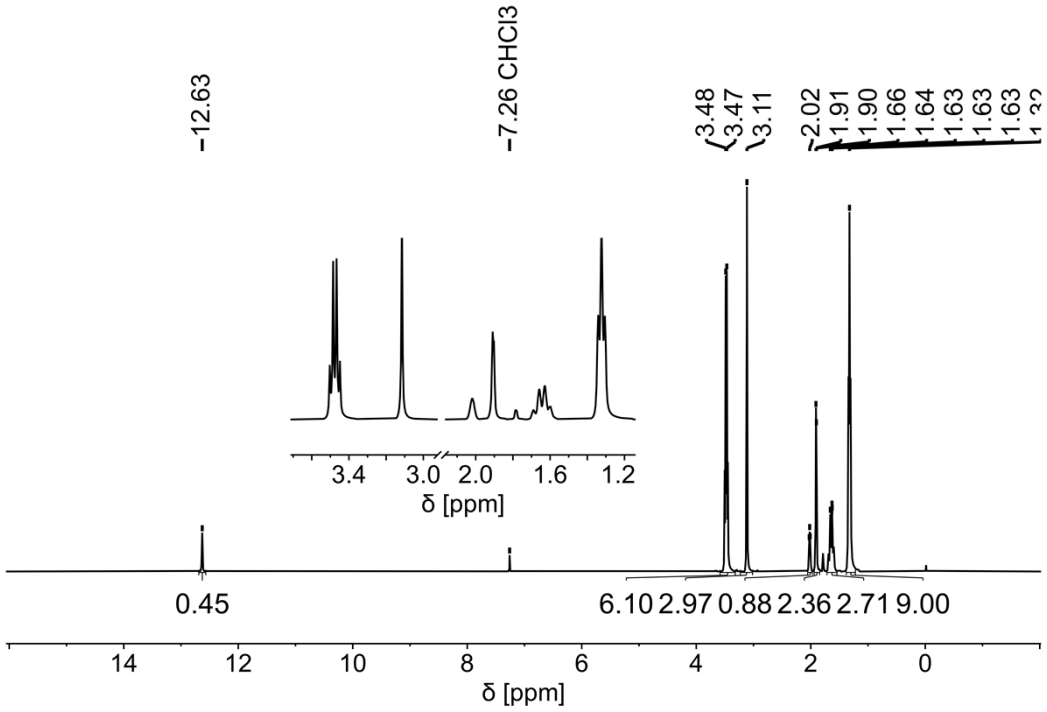

Figure S19. ^{1}H NMR spectrum (CD_2Cl_2) of $POCl_3$ and $[NEt_3Me][Cl(HCl)_n]$ synthesized from $[NEt_3Me][PCl_6]$ and adamantyl carboxylic acid.

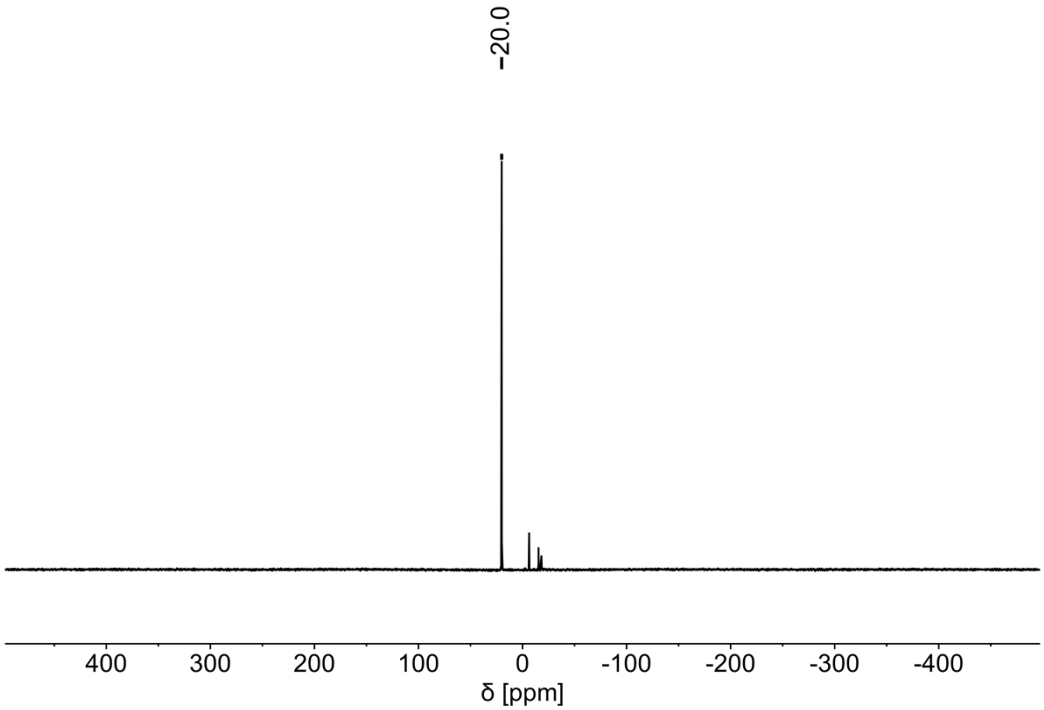

Figure S20. $^{31}P\{^{1}H\}$ NMR spectrum (CD_2Cl_2) of hexaphosphabenzene synthesized from $[NEt_3Me][PCl_6]$ and NH_4Cl.

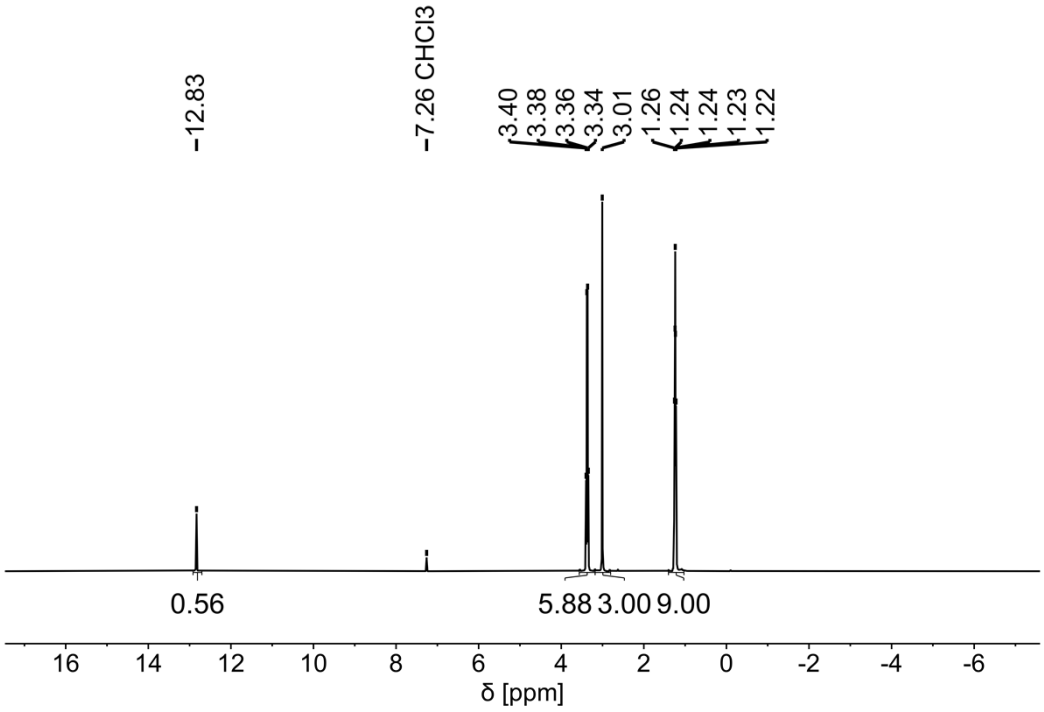

Figure S21. ^{1}H NMR spectrum (CD_2Cl_2) of $[NEt_3Me](Cl(HCl)_n]$ synthesized in the reaction between $[NEt_3Me][PCl_6]$ and NH_4Cl.

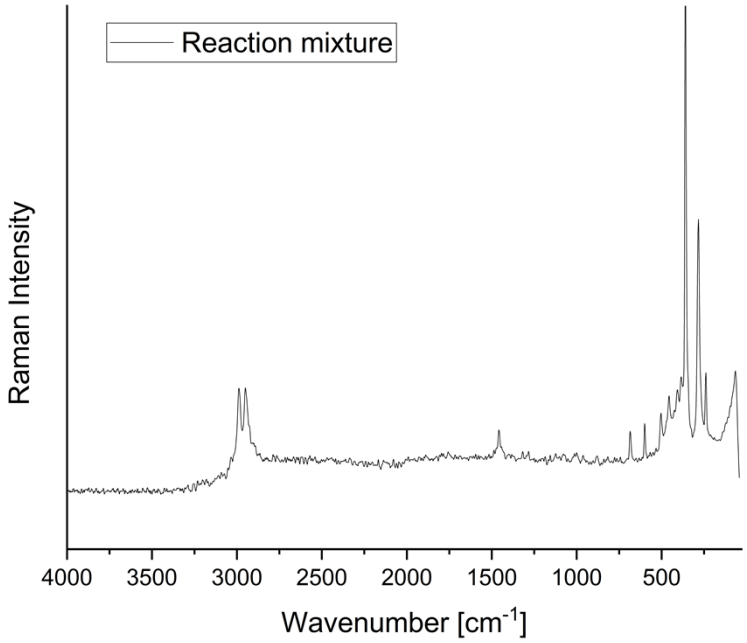

Figure S22. Experimental Raman spectrum of the mixture of $[NEt_3Me][PCl_6]$ and red phosphorus recorded at room temperature.

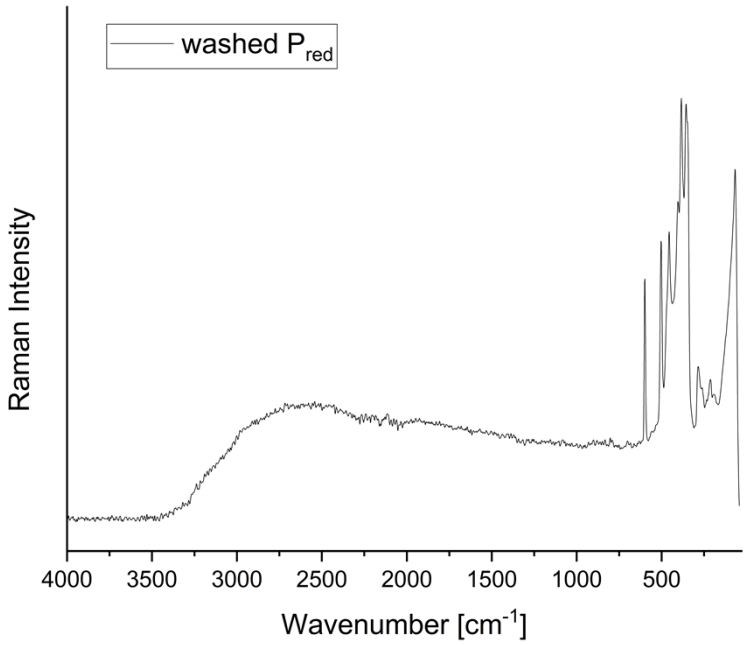

Figure S23. Experimental Raman spectrum of the isolated red phosphorus (P_{red}) recorded at room temperature.

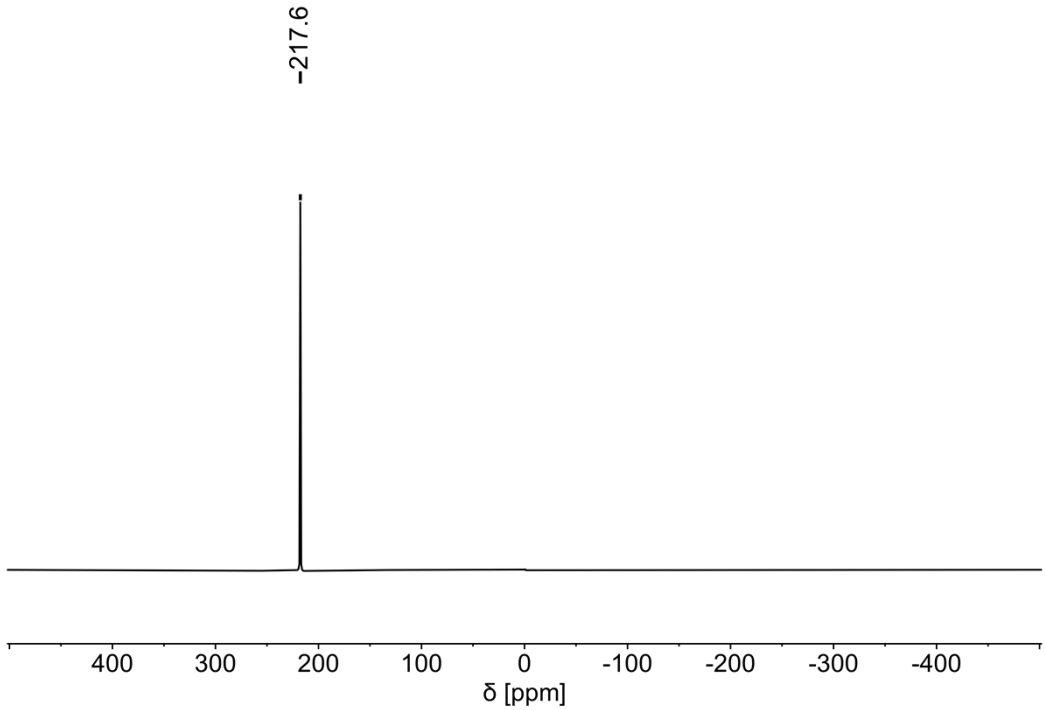

Figure S24. ^{31}P NMR spectrum (neat) of PCl_3 directly synthesized from P_4 and $[NEt_3Me][Cl_3]$.

Figure S25. Experimental setup for the direct synthesis of PCl_3 directly from P_4 and $[NEt_3Me][Cl_3]$.

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