Supporting Information for:

Selenoamides with two reactive sites: synthesis, structures, and dual reactivity of (selenocarbamoyl)phosphines

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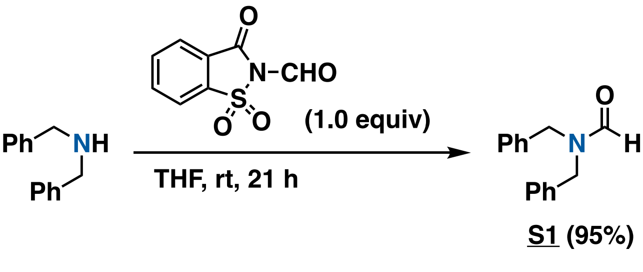
Contents

1. Experimental section

General experimental section

For operations involving air- and/or moisture-sensitive compounds, a glove box (Miwa Manufacturing Co., Ltd. DBO-1KH-GUK2) with an argon atmosphere was used. THF and Et_2O (anhydrous) were purchased from Kanto Chemical and passed through a Kayama Oxygen solvent-purification system prior to use. CH_2Cl_2 and toluene (anhydrous) were purchased from Kanto Chemical and used as received. $CDCl_3$ was passed through a small column of neutral alumina prior to use. Et_3N was purchased from commercial sources and distilled successively from KOH and CaH_2. Other chemicals were purchased from common commercial sources and used as received. Silica-gel column chromatography was performed using Kanto silica gel N60. Preparative gel-permeation liquid chromatography (GPLC) was performed using LC9210 NEXT with a JAI gel 2HR column (Japan Analytical Industry) and AcOEt or $CHCl_3$ as the solvents. ^1H NMR spectra were recorded on a JEOL ECZ-400 or a Bruker ADVANCE-III, and the ^1H chemical shifts are referenced to the residual proton signal of $CDCl_3$ (δ 7.26). $^{13}C\{^1H\}$ NMR spectra were recorded on a JEOL ECZ-400, and the chemical shifts are given relative to the signal of $CDCl_3$ (δ 77.00). $^{11}B\{^1H\}$, $^{19}F\{^1H\}$, $^{31}P\{^1H\}$, and $^{77}Se\{^1H\}$ NMR spectra were recorded on a JEOL ECZ-400, and the chemical shifts of ^{11}B, ^{19}F, ^{31}P, and ^{77}Se are referenced to the external standards of $BF_3 \cdot OEt_2$ (δ 0.0), C_6H_5F (δ −113), H_3PO_4 (δ 0.0), and Ph_2Se_2 (δ 480), respectively. The chemical-shift assignment was performed with the aid of DEPT, COSY, HMQC, and HMBC NMR experiments. IR spectra were recorded on a JASCO FT/IR-4100. UV–vis spectra were recorded on a JASCO V-650 UV–vis spectrometer. High-resolution ESI-TOF mass spectra were measured on a Bruker micrOTOF II instrument. Elemental analysis was conducted on a Thermo Scientific FlashSmart CHNS at the Comprehensive Analysis Center for Science (Saitama University). Melting points (m.p.) were measured using an OptiMelt instrument (Stanford Research Systems) (uncorrected).

Synthesis of *N,N*-dibenzylformamide (S1)

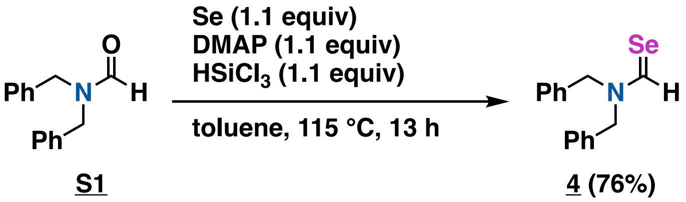


Scheme S1. Synthesis of *N,N*-dibenzylformamide (**S1**).

Briefly, *N*-formyl saccharin (4.17 g, 19.8 mmol) was placed in a flame-dried two-necked flask (200 mL). After the flask was evacuated and backfilled with nitrogen, THF (80 mL) and dibenzylamine (3.77 mL, 19.8 mmol) were added at room temperature. The resulting mixture was stirred at room temperature for 21 h before sat. aq. $NaHCO_3$ was added. After separation of the layers, the aqueous layer was extracted with AcOEt (3×20 mL). The combined organic layers were washed with brine, dried over Na_2SO_4, and filtered. The filtrate was concentrated *in vacuo*, and the crude product was purified by flash column chromatography on silica gel (hexane/AcOEt = 3:1) to give **S1** as colorless crystals. Yield 4.23 g (18.8 mmol, 95%).

S1: colorless crystals. ^{1}**H NMR** (400 MHz, $CDCl_3$): δ 4.26 (s, 2H), 4.42 (s, 2H), 7.16-7.21 (m, 4H), 7.27-7.40 (m, 6H), 8.42 (s, 1H). The NMR data matched previously reported values.[1]

Synthesis of *N,N*-dibenzylselenoformamide (4)

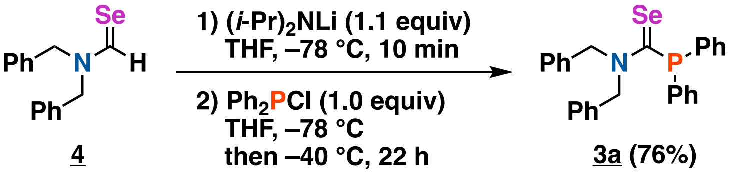


Scheme S2. Synthesis of *N,N*-dibenzylselenoformamide (**4**).

The selenation was performed according to a slightly modified Shibahara–Murai method.[2] Briefly, *N,N*-dibenzylformamide (1.23 g, 5.45 mmol) was placed in a flame-dried two-necked flask (30 mL). After the flask was evacuated and backfilled with nitrogen, toluene (5.5 mL), selenium powder (474 mg, 6.00 mmol), *N,N*-dimethylaminopyridine (709 mg, 6.00 mmol), and $HSiCl_3$ (0.60 mL, 6.0 mmol) were added at room temperature. The resulting mixture was stirred at 115 °C (oil bath) for 13 h, before sat. aq. $NaHCO_3$ was added. The obtained reaction mixture was filtered through Celite on a glass filter (eluent: CH_2Cl_2). The two layers were separated and the aqueous layer was extracted with CH_2Cl_2 (3×10 mL). The combined organic layers were washed with brine, dried over Na_2SO_4, and filtered. The filtrate was concentrated *in vacuo*, and the crude product was purified by flash column chromatography on silica gel (hexane/AcOEt = 5:1) to give **4** as yellow crystals. Yield 1.19 g (4.12 mmol, 76%).

4: yellow crystals. ^{1}**H NMR** (400 MHz, $CDCl_3$): δ 4.52 (s, 2H), 5.10 (s, 2H), 7.13-7.17 (m, 2H), 7.31-7.42 (m, 8H), 11.1 (s, 1H); ^{77}**Se{^{1}H} NMR** (76 MHz, $CDCl_3$): 571. The ^{1}H NMR data matched those reported previously.[2]

Synthesis of (selenocarbamoyl)phosphine 3a

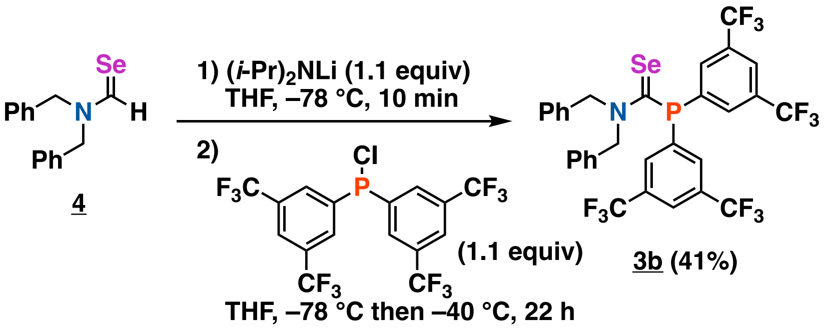


Scheme S3. Synthesis of (selenocarbamoyl)phosphine **3a**.

A THF (7.5 mL) solution of (i-Pr)$_2$NH (0.51 mL, 3.6 mmol) in a flame-dried two-necked flask (100 mL) cooled to at −78 °C was treated drop-wise with n-BuLi (1.51 M in hexane, 2.2 mL, 3.30 mmol), before the mixture was stirred at the same temperature for 30 min. Then, a THF (22.5 mL) solution of selenoformamide **4** (865 mg, 3.00 mmol), which was prepared in another flame-dried two-necked flask (100 mL), was added dropwise at −78 °C for 15 min via a cannula. After stirring at the same temperature for 10 min, a THF (20 mL) solution of chlorodiphenylphosphine (0.56 mL, 3.0 mmol) was added dropwise at −78 °C for 20 min via a cannula. The resulting mixture was stirred at −40 °C for 22 h, followed by the addition of degassed water. The following workup was performed under nitrogen atmosphere. After separation of the layers, the aqueous layer was extracted with Et_2O (4×10 mL) using Schlenk tubes. The combined organic layers were washed with brine, dried over Na_2SO_4, and filtered glass filter using Schlenk tubes. The filtrate was concentrated *in vacuo*, and the crude product was purified by flash column chromatography on silica gel (hexane/AcOEt = 9:1) to give **3a** as bright-orange crystals. Yield 1.21 g (2.57 mmol, 86%).

3a: bright-orange crystals; m.p. 102.2–106.1 °C. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 4.93 (s, 1H), 4.94 (s, 1H), 5.46 (s, 2H), 7.14-7.18 (m, 2H), 7.28-7.46 (m, 14H), 7.50-7.56 (m, 4H); **^{13}C{^{1}H} NMR** (100 MHz, $CDCl_3$): δ 57.0 (d, $^3J_{P\text{–}C}$ = 26.8 Hz), 58.5, 127.50, 127.53, 127.8, 128.2, 128.4, 128.45, 128.54, 128.6, 128.9, 129.9, 133.8, 134.8, 135.0, 135.24, 135.28, 217.1 (d, $^1J_{P\text{–}C}$ = 42.7 Hz); **^{31}P{^{1}H} NMR** (162 MHz, $CDCl_3$): 18.8; **^{77}Se{^{1}H} NMR** (76 MHz, $CDCl_3$): 873 (d, $^2J_{P\text{–}Se}$ = 13.0 Hz); **IR** (KBr); 3050, 3023, 2957, 2916, 1602, 1583, 1494, 1432, 1410, 1314, 1194, 1079, 1025, 974, 863, 743, 697, 612 cm^{-1}; **UV-vis** (THF, 298 K) λ_{max} 495 nm (ε = 134); **HRMS** (ESI-TOF) *m/z* 496.0692 [M+Na]$^+$ (calcd for $C_{27}H_{24}NNaPSe$, 496.0705); **Anal.** calcd. for $C_{27}H_{24}NPSe$: C, 68.64; H, 5.12; N, 2.97. Found: C, 68.47; H, 5.10; N, 2.92%.

Synthesis of (selenocarbamoyl)phosphine 3b

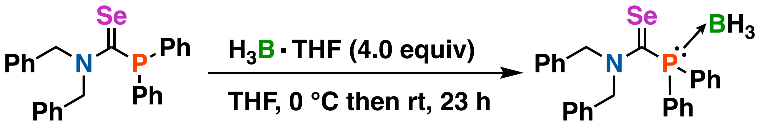


Scheme S4. Synthesis of (selenocarbamoyl)phosphine **3b**.

A THF (2.5 mL) solution of (i-Pr)$_2$NH (0.17 mL, 1.2 mmol) in a flame-dried two-necked flask (100 mL) cooled to at −78 °C was treated drop-wise with n-BuLi (1.63 M in hexane, 0.67 mL, 1.1 mmol), before the mixture was stirred at the same temperature for 30 min. Then, a THF (7.5 mL) solution of selenoformamide **4** (288 mg, 1.00 mmol), which was prepared in another flame-dried two-necked flask (100 mL), was added dropwise at −78 °C for 10 min via a cannula. After stirring at the same temperature for 10 min, a THF (3.3 mL) solution of bis[3,5-bis(trifluoromethyl)phenyl]chlorophosphine (561 mg, 1.14 mmol) was added dropwise at −78 °C for 2 min via a cannula. The resulting mixture was stirred at −40 °C for 22 h, followed by the addition of degassed water. After separation of the layers, the aqueous layer was extracted with AcOEt (2×10 mL). The combined organic layers were washed with brine, dried over Na$_2$SO$_4$, and filtered. The filtrate was concentrated *in vacuo*, and the crude product was purified by flash column chromatography on silica gel (hexane/AcOEt = 19:1) to give **3b** as reddish orange crystals. Yield 305 mg (0.410 mmol, 41%).

3b: reddish orange crystals; m.p. 106.3–108.6 °C. **^{1}H NMR** (400 MHz, CDCl$_3$): δ 4.95 (s, 1H), 4.96 (s, 1H), 5.45 (s, 2H), 7.12-7.15 (br m, 2H), 7.28-7.32 (br m, 2H), 7.33-7.40 (br m, 6H), 7.86 (br d, J = 7.6 Hz, 4H), 7.94 (br, 2H); **^{13}C{^{1}H} NMR** (100 MHz, CDCl$_3$): δ 57.4 (d, $^3J_{\text{P–C}}$ = 28.1 Hz), 59.6, 122.90 (q, $^1J_{\text{C–F}}$ = 271 Hz), 124.27-124.37 (m), 127.0, 128.3, 128.5, 128.7, 129.1, 129.2, 131.89 (q, $^2J_{\text{C–F}}$ = 33.4 Hz), 131.97 (q, $^2J_{\text{C–F}}$ = 33.4 Hz), 133.1, 134.2, 134.3, 134.6, 137.5, 137.6, 210.0 (d, $^1J_{\text{P–C}}$ = 37.4 Hz); **^{19}F{^{1}H} NMR** (376 MHz, CDCl$_3$): –63; **^{31}P{^{1}H} NMR** (126 MHz, CDCl$_3$): 13.0; **^{77}Se{^{1}H} NMR** (76 MHz, CDCl$_3$): 968 (d, $^2J_{\text{P–Se}}$ = 13.0 Hz); **IR** (KBr); 3025, 2951, 2922, 1615, 1600, 1494, 1454, 1447, 1414, 1353, 1277, 1175, 1126, 983, 919, 891, 843, 747, 700, 682, 614, 547 cm^{-1}; **HRMS** (ESI-TOF) m/z 768.0200 [M+Na]$^+$ (calcd for $C_{31}H_{20}F_{12}NNaPSe$, 768.0207).

Synthesis of phosphine borane 6



3a

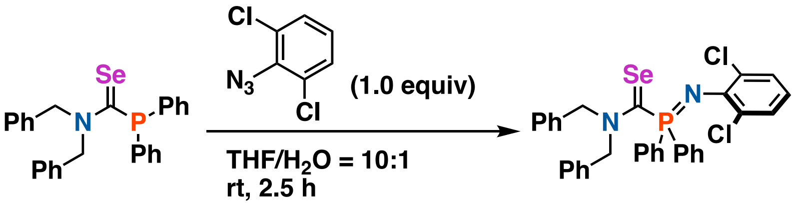
6 (70%)

Scheme S5. Synthesis of phosphine borane **6**.

An evacuated and nitrogen-backfilled flame-dried two-necked flask (100 mL) was charged with (selenocarbamoyl)phosphine **3a** (236 mg, 0.500 mmol), THF (50 mL), and then borane-THF complex (2.00 mL, 2.00 mmol). The resulting mixture was stirred at room temperature for 23 h. After TLC monitoring (hexane/AcOEt = 9:1), the obtained reaction mixture was concentrated *in vacuo*, and the crude product was purified by recrystallization from benzene/*n*-pentane to give **6** as red crystals. Yield 169 mg (0.348 mmol, 70%).

6: red crystals; m.p. 177.6 °C (decomp.). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 1.03-1.85 (br, 3H), 4.81 (s, 2H), 5.34 (s, 2H), 7.01-7.04 (m, 2H), 7.23-7.25 (m, 2H), 7.28-7.37 (m, 6H), 7.43 (td, J = 7.8, 2.7 Hz, 4H), 7.52 (td, J = 7.3, 1.4 Hz, 2H), 7.74-7.79 (m, 4H); **^{13}C{^{1}H} NMR** (100 MHz, $CDCl_3$): δ 58.8, 59.8 (d, $J_{\mathrm{P-C}}$ = 26.8 Hz), 128.05, 128.09, 128.25, 128.29, 128.5, 128.61, 128.64, 128.7, 129.3, 129.9, 131.8, 133.35, 133.42, 133.5, 133.9, 203.3 (d, $^{1}J_{\mathrm{P-C}}$ = 26.9 Hz); **^{11}B{^{1}H} NMR** (128 MHz, $CDCl_3$): –36.9; **^{31}P{^{1}H} NMR** (162 MHz, $CDCl_3$): 42.7; **^{77}Se{^{1}H} NMR** (76 MHz, $CDCl_3$): 1040; **IR** (KBr); 3069, 3028, 2937, 2410, 2384, 2353, 2261, 1601, 1495, 1436, 1422, 1353, 1311, 1214, 1197, 1138, 1102, 1056, 1029, 981, 915, 860, 743, 698, 593, 567, 531, 508 cm^{-1}.

Synthesis of iminophosphorane 7



3a

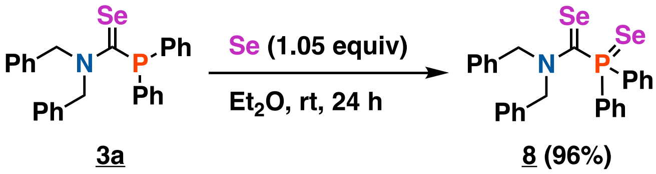
7 (98%)

Scheme S6. Synthesis of iminophosphorane **7**.

2,6-Dichlorophenylazide (775.0 mg, 4.120 mmol) was placed in a two-necked flask (30 mL). After the flask was evacuated and backfilled with nitrogen, THF (16 mL), water (16 mL), and then (selenocarbamoyl)phosphine **3a** (1.947 g, 4.120 mmol) were added at room temperature. The resulting mixture was stirred at the same temperature for 2.5 h. After separation of the layers, the aqueous layer was extracted with AcOEt (3×10 mL). The combined organic layers were washed with water then brine, dried over $MgSO_4$, and filtered. The filtrate was concentrated *in vacuo* to give sufficiently pure **7** as ruby red crystals. Yield 2.557 g (4.404 mmol, 98%).

7: ruby-red crystals; m.p. 170.1–171.5 °C. **^{1}H NMR** (400 MHz, $CDCl_3$): 5.33 (s, 2H), 5.74 (s, 2H), 6.47 (d, J = 7.3 Hz, 2H), 6.58 (td, J = 7.8, 1.8 Hz, 1H), 7.05 (br t, J = 7.8 Hz, 2H), 7.12 (br d, J = 8.2 Hz, 2H), 7.13 (br t, J = 7.3 Hz, 1H), 7.34-7.38 (m, 5H), 7.40 (td, J = 7.8, 3.2 Hz, 4H), 7.51 (td, J = 7.6, 1.4 Hz, 2H), 8.04 (d, J = 8.2 Hz, 2H), 8.07 (d, J = 7.8 Hz, 2H); **^{13}C{^{1}H} NMR** (100 MHz, $CDCl_3$): δ 58.4 (d, $^3J_{P\text{–}C}$ = 37.6 Hz), 119.4, 127.1, 127.2, 127.5, 127.9, 128.0, 128.1, 128.3, 128.5, 129.01, 129.08, 130.1, 130.6, 130.7, 131.5, 132.6, 132.7, 134.0, 143.7, 204.7 (d, $^1J_{P\text{–}C}$ = 67.2 Hz); **^{31}P{^{1}H} NMR** (126 MHz, $CDCl_3$): 9.8; **^{77}Se{^{1}H} NMR** (76 MHz, $CDCl_3$): 946 (d, $^2J_{P\text{–}Se}$ = 8.7 Hz); **IR** (KBr); 3057, 2979, 1570, 1461, 1422, 1338, 1216, 1114, 1073, 851, 769, 754, 736, 695, 580, 556, 541 cm^{-1}; **HRMS** (ESI-TOF) *m/z* 655.0346 $[M+Na]^+$ (calcd for $C_{33}H_{27}Cl_2N_2NaPSe$, 655.0350); **Anal.** calcd. for $C_{33}H_{27}Cl_2N_2PSe$: C, 62.67; H, 4.30; N, 4.43. Found: C, 62.66; H, 4.27; N, 4.34%.

Synthesis of (selenocarbamoyl)phosphine selenide 8

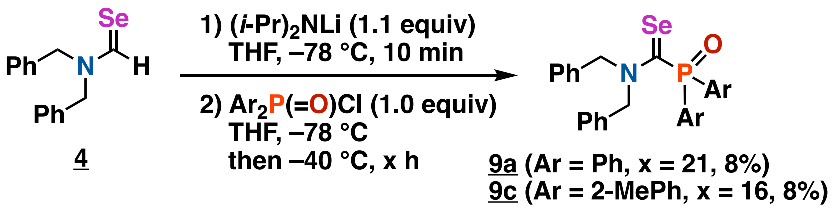


Scheme S7. Synthesis of (selenocarbamoyl)phosphine selenide **8**.

An evacuated and nitrogen-backfilled flame-dried two-necked flask (200 mL) was charged with Et_2O (58 mL), (selenocarbamoyl)phosphine **3a** (472.4 mg, 1.000 mmol), and then elemental selenium (82.9 mg, 1.05 mmol). The resulting mixture was stirred at room temperature for 24 h. After TLC monitoring (hexane/AcOEt = 9:1), the obtained reaction mixture was filtered through Celite on a glass filter (eluent: Et_2O). The filtrate was concentrated *in vacuo*, and the crude product was purified by flash column chromatography on silica gel (hexane/AcOEt = 12:1) to give **8** as reddish-purple crystals. Yield 531.6 mg (0.9641 mmol, 96%).

8: reddish-purple crystals; m.p. 169.2 °C (decomp.). **^{1}H NMR** (400 MHz, $CDCl_3$, 40 °C): 5.05 (s, 2H), 5.29 (s, 2H), 7.04-7.09 (m, 2H), 7.24-7.30 (m, 5H), 7.30-7.36 (m, 3H), 7.42 (td, J = 7.6, 3.2 Hz, 4H), 7.49 (td, J = 7.3, 1.8 Hz, 2H), 7.94 (d, J = 7.8 Hz, 2H), 7.97 (d, J = 7.8 Hz, 2H); **^{13}C{^{1}H} NMR** (100 MHz, $CDCl_3$): δ 58.7, 59.1 (d, $^{3}J_{\text{P–C}}$ = 9.4 Hz), 128.0, 128.1, 128.2, 128.5, 128.6, 131.7, 131.88, 131.91, 132.5, 132.7, 132.8, 133.2, 134.0, 200.2 (d, $^{1}J_{\text{P–C}}$ = 48.4 Hz); **^{31}P{^{1}H} NMR** (126 MHz, $CDCl_3$): 47.1 (t, $^{1}J_{\text{P–Se}}$ = 741 Hz); **^{77}Se{^{1}H} NMR** (76 MHz, $CDCl_3$): –254 (d, $J_{\text{P–Se}}$ = 742 Hz), 1035; **IR** (KBr); 3053, 3031, 2930, 1495, 1462, 1450, 1437, 1402, 1352, 1308, 1226, 1197, 1129, 1089, 1026, 999, 864, 747, 696, 621, 600, 569, 542, 506 cm^{-1}; **HRMS** (ESI-TOF) *m/z* 575.9874 $[M+Na]^+$ (calcd for $C_{27}H_{24}N_2NaPSe_2$, 575.9879); **Anal.** calcd. for $C_{27}H_{24}NPSe_2$: C, 58.81; H, 4.87; N, 2.54. Found: C, 58.75; H, 4.45; N, 2.42%.

General procedure for the preparation of (selenocarbamoyl)phosphine oxides 9a and 9c



Scheme S8. Synthesis of (selenocarbamoyl)phosphine oxides **9a** and **9c**.

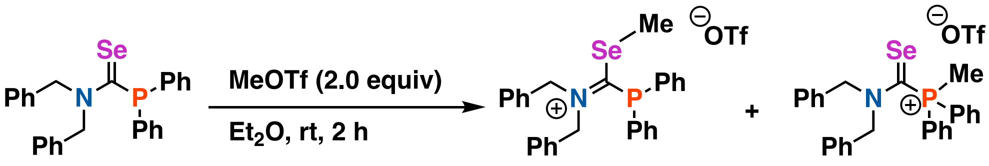
A THF (0.48 M) solution of (i-Pr)$_2$NH (1.2 equiv) in a flame-dried two-necked flask at –78 °C was treated dropwise with n-BuLi (1.55 M in hexane, 1.1 equiv) and subsequently stirred at the same temperature for 30 min. Then, a THF (0.13 M) solution of selenoformamide **4** (1.0 equiv), which was prepared in another flame-dried two-necked flask, was added dropwise at –78 °C for 20 min via a cannula. After stirring at the same temperature for 10 min, a THF (0.30 M) solution of diphenylphosphinic chloride or bis(2-methylphenyl)phosphinic chloride (1.0 equiv) was added dropwise at –78 °C for 10 min via a cannula. The resulting mixture was stirred at –40 °C for the indicated time, before water was added. After separation of the layers, the aqueous layer was extracted with AcOEt (3×10 mL). The combined organic layers were washed with brine, dried over Na$_2$SO$_4$, and filtered. The filtrate was concentrated *in vacuo*, and the crude product was purified by flash column chromatography on silica gel (hexane/AcOEt = 9:1) and then GPLC (AcOEt) to give (selenocarbamoyl)phosphine oxides **9a** or **9c** as pink crystals.

(a) (selenocarbamoyl)phosphine oxide **9a**.
Reagents: (i-Pr)$_2$NH (0.34 mL, 2.4 mmol), n-BuLi (1.27 mL, 2.20 mmol), **4** (577 mg, 2.00 mmol), Ph$_2$P(=O)Cl (74.0 mg, 0.300 mmol), THF (total 4.2 mL). Reaction time: 16 h. Yield: 11.7 mg (0.023 mmol, 8%)
9a: pink crystals; 151.6–152.0 °C. **^{1}H NMR** (400 MHz, CDCl$_3$, *only signals corresponding to the major conformer of the rotameric mixture are shown*): 5.26 (s, 2H), 5.37 (s, 2H), 7.08-7.12 (m, 2H), 7.19-7.23 (m, 2H), 7.24-7.28 (m, 3H), 7.29-7.35 (m, 3H), 7.48 (td, J = 7.3, 3.2 Hz, 4H), 7.54-7.60 (m, 2H), 7.95-8.00 (m, 4H); **^{13}C{^{1}H} NMR** (100 MHz, CDCl$_3$): δ 57.8 (d, $^3J_{\text{P–C}}$ = 5.4 Hz), 58.4 (d, $^3J_{\text{P–C}}$ = 2.7 Hz), 127.97, 128.0, 128.1, 128.2, 128.7, 128.8, 131.4, 132.0, 132.1, 132.2, 132.3, 132.5, 133.9, 134.0, 203.7 (d, $^1J_{\text{P–C}}$ = 73.9 Hz); **^{31}P{^{1}H} NMR** (126 MHz, CDCl$_3$): 35.1; **^{77}Se{^{1}H} NMR** (76 MHz, CDCl$_3$): 983 (d, $J_{\text{P–Se}}$ = 8.6 Hz); **IR** (KBr); 3033, 2937, 2917, 1493, 1468, 1451, 1436, 1422, 1217, 1179, 1115, 861, 748, 727, 702, 555 cm^{-1}; **HRMS** (ESI-TOF) m/z 512.0658 [M+Na]$^+$ (calcd for C$_{27}$H$_{24}$NNaOPSe, 512.0654).

(b) (selenocarbamoyl)phosphine oxide **9c**.
Reagents: (i-Pr)$_2$NH (0.05 mL, 0.36 mmol), n-BuLi (0.19 mL, 0.33 mmol), **4** (86.5 mg, 0.300 mmol), (2-MePh)$_2$P(=O)Cl (0.38 mL, 2.0 mmol), THF (total 26.7 mL). Reaction time: 21 h. Yield: 80.3 mg (0.164 mmol, 8%)
9c: pink crystals; 174.8-176.8 °C. **^{1}H NMR** (400 MHz, CDCl$_3$): 2.68 (br s, 6H), 5.29 (br s, 2H), 5.38 (br s, 2H), 7.17 (br td, J = 7.1, 2.3 Hz, 2H), 7.27-7.55 (m, 16H); **^{13}C{^{1}H} NMR** (100 MHz, CDCl$_3$): δ 22.29, 22.33, 58.3 (d, $^3J_{\text{P–C}}$ = 2.7 Hz), 58.6 (d, $^3J_{\text{P–C}}$ = 5.4 Hz), 125.0, 125.1, 128.1, 128.2, 128.6, 128.68, 128.72, 128.77, 132.0, 132.1,

132.4, 132.5, 134.0, 134.4, 143.3, 143.4, 203.9 (d, $^{1}J_{\mathrm{P-C}}$ = 72.5 Hz); **^{31}P{^{1}H} NMR** (126 MHz, $CDCl_3$): 45.5; **^{77}Se{^{1}H} NMR** (76 MHz, $CDCl_3$): 959 (d, $^{2}J_{\mathrm{P-Se}}$ = 21.7 Hz); **IR** (KBr); 3035, 2959, 2924, 1593, 1496, 1450, 1422, 1353, 1282, 1209, 1177, 1140, 1119, 1078, 1031, 983, 940, 806, 761, 750, 699, 615, 570, 514 cm^{-1}; **HRMS** (ESI-TOF) *m/z* 540.0957 [M+Na]$^{+}$ (calcd for $C_{29}H_{28}NONaPSe$, 540.0968).

Methylation of (selenocarbamoyl)phosphine 3a by MeOTf



3a

10 (95%*)

11 (5%*)

*Estimated by ^{31}P{^{1}H} NMR spectroscopy

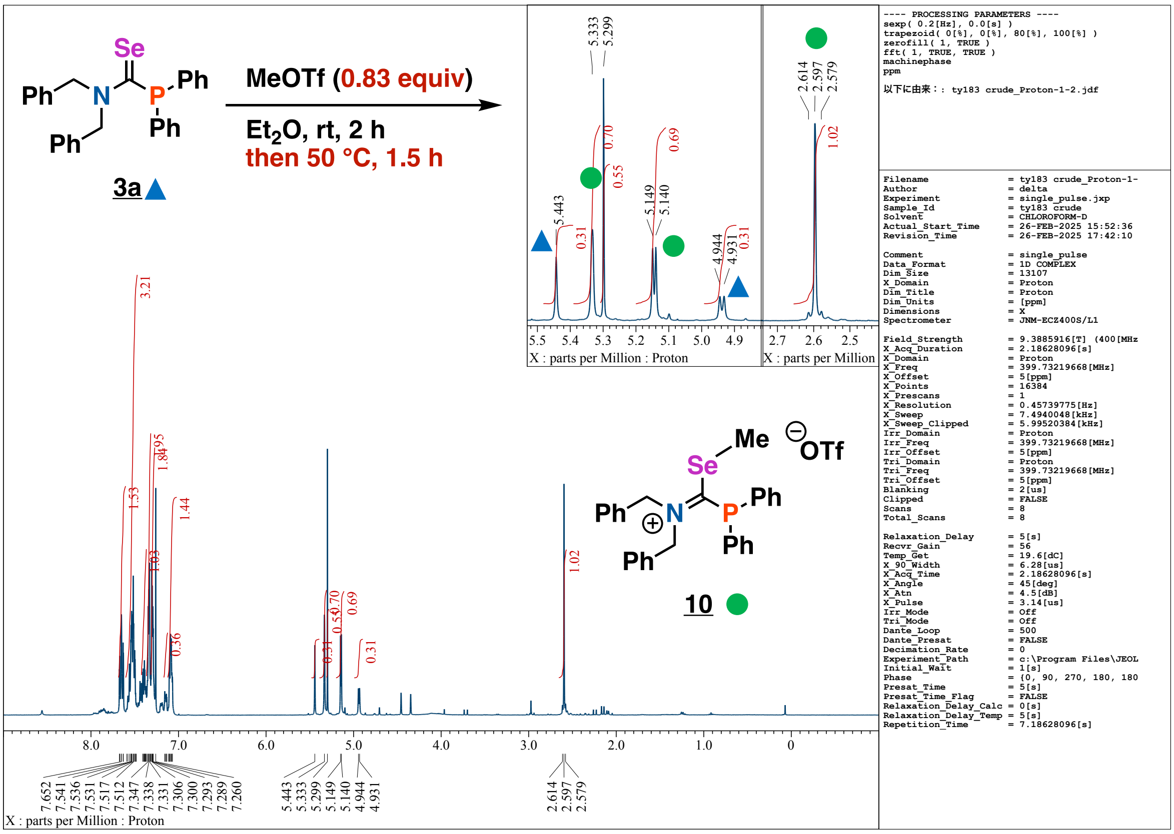
Scheme S9. Methylation of (selenocarbamoyl)phosphine **3a** by MeOTf.

An evacuated and nitrogen-backfilled flame-dried test tube (20 mL) was charged with (selenocarbamoyl)phosphine **3a** (47.1 mg, 0.100 mmol), Et_2O (10 mL), and then. MeOTf (22 µL, 0.20 mmol). The resulting mixture was stirred at room temperature for 2 h. The resulting mixture was concentrated *in vacuo* to give **10** as yellow oil. The NMR yields of **10** and phosphonium salt **11** were determined as 95% and 5%, by the ^{31}P{^{1}H} NMR spectrum (**Figure S50**), respectively. Yield 69.5 mg (0.101 mmol, quant.).

10: yellow amorphous solid. **^{1}H NMR** (400 MHz, $CDCl_3$): 2.60 (s, 3H), 5.15 (s, 1H), 5.16 (s, 1H), 5.34 (s, 2H), 7.06-7.12 (m, 4H), 7.28-7.31 (m, 3H), 7.32-7.35 (m, 3H), 7.49-7.56 (m, 6H), 7.63-7.68 (m, 4H); **^{13}C{^{1}H} NMR** (100 MHz, $CDCl_3$): δ 17.9, 63.0 (d, $^{3}J_{\mathrm{P-C}}$ = 22.8 Hz), 63.7, 120.8 (q, $^{1}J_{\mathrm{F-C}}$ = 321.0 Hz), 127.55, 127.61, 127.8, 128.2, 129.25, 129.3, 129.4, 129.8, 129.9, 130.2, 131.4, 132.0, 135.0, 135.2, 208.5 (d, $^{2}J_{\mathrm{P-Se}}$ = 68.5 Hz); **^{31}P{^{1}H} NMR** (126 MHz, $CDCl_3$): 19.5; **^{77}Se{^{1}H} NMR** (76 MHz, $CDCl_3$): 512; **HRMS** (ESI-TOF) *m/z* 488.1044 [M–OTf]$^{+}$ (calcd for $C_{28}H_{27}NPSe$, 488.1042).

Reactions using other methylation agents: The methylation of **3a** by methyl iodide (2.0 equiv) was examined in Et_2O for 4 h but no reaction was observed even at 50 °C. When Me_3OBF_4 was used, **3a** consumed and selenoiminium salt **10** and phosphonium salt **11** were formed in approximately 98% and <2% NMR yields, respectively.

Reactions with MeOTf at the elevated temperature: To consider the possibility of thermodynamic control, we also examined the reaction of (selenocarbamoyl)phosphine **3a** with MeOTf (0.83 equiv) in Et_2O at room temperature for 2 h and then the resulting mixture was heated at 50 °C for 1.5 h. However, no significant amount of phosphonium salt **11** was observed in the ^{31}P{^{1}H} NMR spectrum of the crude mixture (**Figure S1**). In the ^{1}H NMR spectrum (**Figure S2**), the crude mixture was more complicated than that of Scheme S9, and the starting material **3a** and selenoiminium salt **10** were observed (**3a**:**10** = 69:31), respectively. Furthermore, we also conducted the reaction of **3a** with MeOTf (2.0 equiv) at 50 °C for 2 h. As expected, the composition of the crude mixture was the similar to that obtained in Scheme S9; the NMR yields of **10** and **11** were determined as 95% and 5%, respectively.



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fft( 1, TRUE, TRUE )
machinephase
ppm

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Author                    = delta
Experiment                = single_pulse.jxp
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Revision_Time             = 26-FEB-2025 17:42:10

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Dim_Title                 = Proton
Dim_Units                 = [ppm]
Dimensions                = X
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Tri_Mode                  = Off
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Dante_Presat              = FALSE
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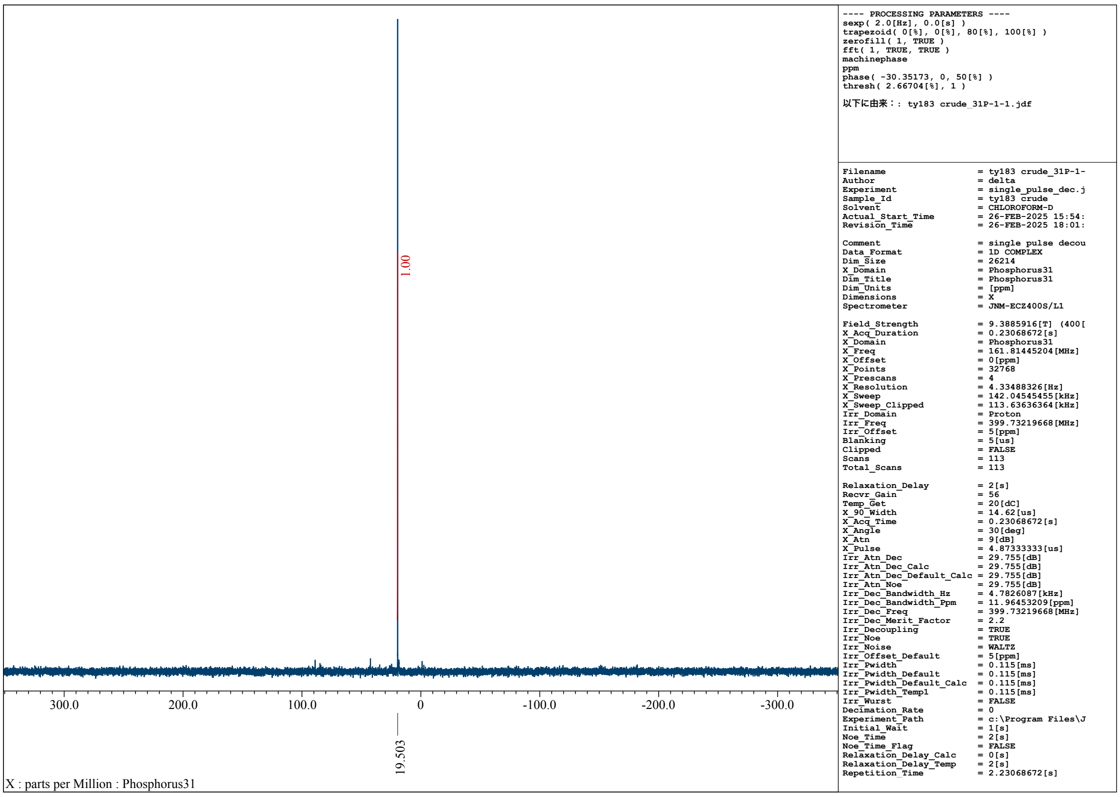
Figure S1. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of the crude mixture of the reaction of **3a** with MeOTf (0.83 equiv) at 50 °C.



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machinephase
ppm
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以下に由来：: ty183 crude_31P-1-1.jdf

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Revision_Time             = 26-FEB-2025 18:01:

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Dim_Title                 = Phosphorus31
Dim_Units                 = [ppm]
Dimensions                = X
Spectrometer              = JNM-ECZ400S/L1

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Irr_Atn_Dec_Calc          = 29.755[dB]
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Irr_Atn_Noe               = 29.755[dB]
Irr_Dec_Bandwidth_Hz      = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm     = 11.96453209[ppm]
Irr_Dec_Freq              = 399.73219668[MHz]
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Irr_Noe                   = TRUE
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Relaxation_Delay_Temp     = 2[s]
Repetition_Time           = 2.23068672[s]
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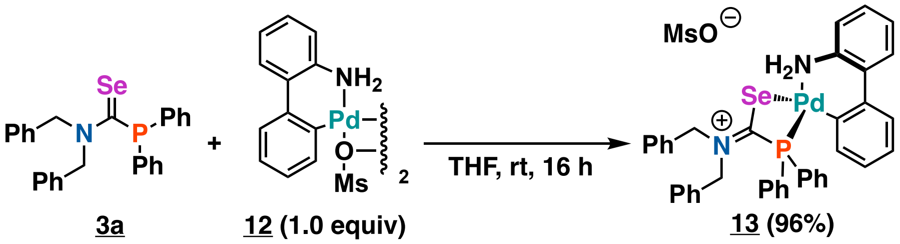
Figure S2. $^{31}P\{^1H\}$ NMR spectrum (162 MHz, $CDCl_3$) of the crude mixture of the reaction of **3a** with MeOTf (0.83 equiv) at 50 °C.

Synthesis of palladium complex 13

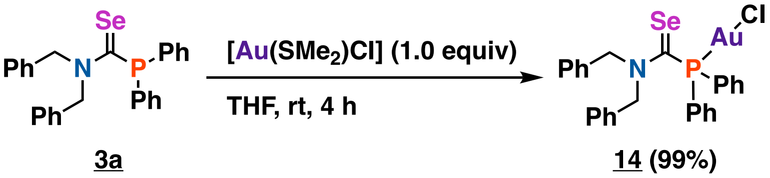


Scheme S10. Synthesis of palladium complex **13**.

An evacuated and nitrogen-backfilled flame-dried Schlenk tube (25 mL) was charged with μ-OMs dimer **14**[3] (185 mg, 0.250 mmol), (selenocarbamoyl)phosphine **3a** (236.2 mg, 0.500 mmol), and then THF (3.6 mL). The resulting mixture was stirred at room temperature for 16 h. After TLC monitoring (hexane/AcOEt = 9:1), the filtrate was concentrated *in vacuo*, and the crude product was washed with dry pentane under nitrogen atmosphere to give **13** as pale-yellow crystals. Yield 392.7 mg (0.4663 mmol, 93%).

13: pale-yellow crystals; m.p. 156.4-161.7 °C. **^{1}H NMR** (400 MHz, $CDCl_3$, 40 °C): 2.89 (s, 3H), 4.42 (s, 2H), 4.91 (s, 2H), 6.33 (d, J = 7.3 Hz, 2H), 6.60 (t, J = 7.8 Hz, 1H), 6.73 (t, J = 7.8 Hz, 1H), 7.04 (t, J = 7.3 Hz, 1H), 7.10 (t, J = 7.8 Hz, 2H), 7.20 (quint, J = 7.3 Hz, 2H), 7.23-7.30 (m, 3H), 7.32-7.43 (m, 9H), 7.46-7.52 (m, 2H), 7.61 (d, J = 7.8 Hz, 2H), 7.64 (d, J = 7.8 Hz, 2H), 7.87 (d, J = 7.8 Hz, 1H) (*two protons of the amino group are not detected clearly because they may be broadly observed*); **^{13}C{^{1}H} NMR** (100 MHz, $CDCl_3$): δ 39.8, 55.0, 58.8 (d, $J_{P\text{–}C}$ = 4.0 Hz), 121.5, 121.7, 122.1, 125.1, 126.0, 126.6, 126.8, 127.2, 127.3, 127.8, 128.0, 128.9, 129.0, 129.3, 129.5, 129.8, 130.0, 131.6, 132.8, 133.0, 133.1, 135.7 (d, $J_{P\text{–}C}$ = 13.4 Hz), 136.6 (d, $J_{P\text{–}C}$ = 4.0 Hz), 138.6, 139.1, 151.5 (d, $J_{P\text{–}C}$ = 14.8 Hz), 206.3; **^{31}P{^{1}H} NMR** (126 MHz, $CDCl_3$): 16.8; **^{77}Se{^{1}H} NMR** (76 MHz, $CDCl_3$): 836; **IR** (KBr); 3193, 3061, 2976, 2932, 1517, 1496, 1438, 1422, 1214, 1192, 1040, 745, 694, 553, 523 cm^{-1}; **HRMS** (ESI-TOF) *m/z* 747.0678 $[M\text{–}OMs]^+$ (calcd for $C_{39}H_{34}N_2PPdSe$, 747.0673).

Synthesis of gold complex 14

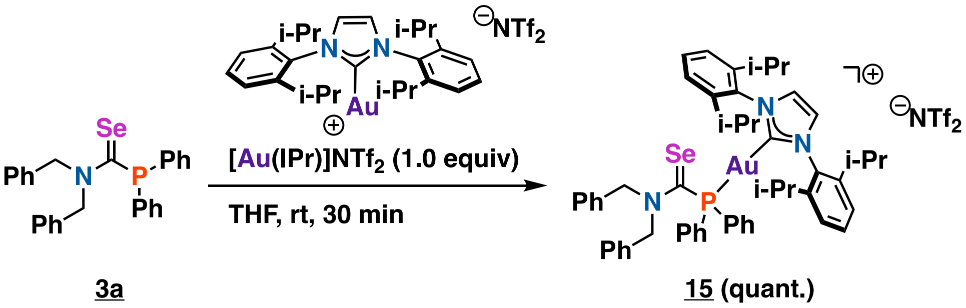


Scheme S11. Synthesis of gold complex **14**.

An evacuated and nitrogen-backfilled flame-dried two-necked flask (5 mL) was charged with chloro(dimethylsulfide)gold (59.2 mg, 0.200 mmol), (selenocarbamoyl)phosphine **3a** (94.0 mg, 0.200 mmol), and then THF (1.33 mL). The resulting mixture was stirred at room temperature for 4 h. After TLC monitoring (hexane/AcOEt = 9:1), the filtrate was concentrated *in vacuo* to give **14** as ruby-reddish crystals. Yield 140 mg (0.199 mmol, 99%).

14: ruby-reddish crystals; m.p. 183.4 °C (decomp.). **^{1}H NMR** (400 MHz, $CDCl_3$): 5.018 (s, 1H), 5.023 (s, 1H), 5.37 (s, 2H), 7.07-7.11 (m, 2H), 7.24-7.26 (m, 2H), 7.33-7.38 (m, 6H), 7.46 (td, J = 7.8, 2.7 Hz, 4H), 7.53-7.58 (m, 2H), 7.69-7.71 (m, 2H), 7.72-7.74 (m, 2H); **^{13}C{^{1}H} NMR** (100 MHz, $CDCl_3$): δ 58.7 (d, $J_{P\text{–}C}$ = 16.1 Hz), 59.4, 127.4, 128.4, 128.6, 128.8, 129.0, 129.1, 129.2, 129.3, 132.2, 132.6, 133.8, 135.0, 135.1, 199.5 (d, $^1J_{P\text{–}C}$ = 32.2 Hz); **^{31}P{^{1}H} NMR** (126 MHz, $CDCl_3$): 46.2; **^{77}Se{^{1}H} NMR** (76 MHz, $CDCl_3$): 1063; **IR** (KBr); 3057, 3026, 2950, 1493, 1449, 1437, 1418, 1342, 1202, 1192, 1097, 1078, 980, 853, 757, 747, 698, 683, 557 cm^{-1}; **HRMS** (ESI-TOF) *m/z* 728.0058 $[M+Na]^+$ (calcd for $C_{27}H_{24}AuClNNaPSe$, 728.0057).

Synthesis of cationic gold complex 15



Scheme S12. Synthesis of cationic gold complex **15**.

An evacuated and nitrogen-backfilled flame-dried two-necked flask (5 mL) was charged with [Au(IPr)]NTf$_2$,[4] which was prepared by the reported procedure, (86.5 mg, 0.100 mmol), (selenocarbamoyl)phosphine **3a** (47.3 mg, 0.100 mmol), and then THF (0.67 mL). The resulting mixture was stirred at room temperature for 30 min. After TLC monitoring (hexane/AcOEt = 9:1), the filtrate was concentrated *in vacuo* to give **15** as brownish amorphous solid. Yield 133 mg (0.099 mmol, 99%).

15: brownish amorphous solid; m.p. 162.0 °C (decomp.). **^{1}H NMR** (400 MHz, CDCl$_3$): 1.10 (d, J = 6.9 Hz, 12H), 1.23 (d, J = 6.9 Hz, 12H), 2.50 (septet, J = 6.9 Hz, 4H), 4.55 (s, 2H), 5.14 (s, 2H), 6.28 (d, J = 7.3 Hz, 2H), 7.03-7.11 (m, 8H), 7.17 (t, J = 7.3 Hz, 1H), 7.24-7.27 (m, 4H), 7.29-7.31 (m, 6H), 7.35 (tt, J = 7.3, 2.7 Hz, 1H), 7.45 (br td, J = 7.6, 1.4 Hz, 2H), 7.50 (s, 2H), 7.56 (t, J = 7.8 Hz, 2H); **^{13}C{^{1}H} NMR** (100 MHz, CDCl$_3$): δ 23.9, 24.5, 28.7, 59.0 (d, $^3J_{P–C}$ = 10.8 Hz), 60.3, 119.9 (q, $J_{C–F}$ = 322 Hz), 124.3, 124.98, 125.00, 126.1, 126.4, 126.7, 128.0, 128.5, 128.8, 129.1, 129.6, 129.7, 130.9, 131.1, 132.5, 132.8, 133.3, 133.4, 133.6, 186.0 (d, $^2J_{P–C}$ = 124.9 Hz), 198.8 (d, $^1J_{P–C}$ = 20.2 Hz); **^{19}F{^{1}H} NMR** (376 MHz, CDCl$_3$): –79; **^{31}P{^{1}H} NMR** (126 MHz, CDCl$_3$): 61.9 (br); **^{77}Se{^{1}H} NMR** (76 MHz, CDCl$_3$): no signal; **IR** (KBr); 2964, 2928, 2871, 1497, 1455, 1438, 1355, 1334, 1226, 1196, 1139, 1058, 740, 690, 618 cm^{-1}; **HRMS** (ESI-TOF) *m/z* 1058.3361 [M–NTf$_2$]$^+$ (calcd for $C_{54}H_{60}AuN_3PSe$, 1058.3356).

2. X-ray crystallographic analysis

X-ray crystallographic analysis of 3a.

Single crystals of **3a** were grown in their AcOEt-hexane solution. A bright-orange crystal of **3a** was mounted on a loop. All measurements were made on a XtaLAB mini II (Rigaku Co.) with graphite monochromated Mo-Kα radiation (λ = 0.71073 Å) at –173 °C. Crystallographic and experimental data are listed in **Table S1**. The structures were solved and refined against all F_2 values using Shelx-2018[5] implemented through Olex2 v1.5. Nonhydrogen atoms were refined anisotropically. Hydrogen atoms were idealized using the riding models.

Table S1. Crystal data and structure refinement for **3a**.

Empirical formula	$C_{27}H_{24}NPSe$	
Formula weight	472.40	
Temperature	99.99(10) K	
wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	*Ia*	
Unit cell dimensions	a = 9.8139(7) Å	α = 90°
	b = 17.3013(11) Å	β = 98.701(6)°
	c = 26.7081(18) Å	γ = 90°
Volume	4482.7(5) Å^3	
Z	8	
D_{calc}	1.400 g/cm^3	
Absorption coefficient	1.761 mm^{-1}	
F(000)	1936.0	
Crystal size	0.11 x 0.09 x 0.03 mm^3	
Theta range for data collection	2.354 to 24.994°	
Index ranges	$-11 \leq h \leq 11$, $-20 \leq k \leq 20$, $-31 \leq l \leq 31$	
Reflections collected	17101	
Independent reflections	6923 [R_{int} = 0.0852]	
Max. and min. transmission	0.865 and 1.000	
Completeness to theta = 50.0°	99.7%	
data / restraints / parameters	6923 / 225 / 541	
Goodness-of-fit on F^2	1.025	
Final R induces (I>2σ(I))	$R1$ = 0.0619, w$R2$ = 0.1309	
R induces (all data)	$R1$ = 0.1012, w$R2$ = 0.1480	
Largest diff. peak and hole	0.90 and −0.49 e. Å^{-3}	

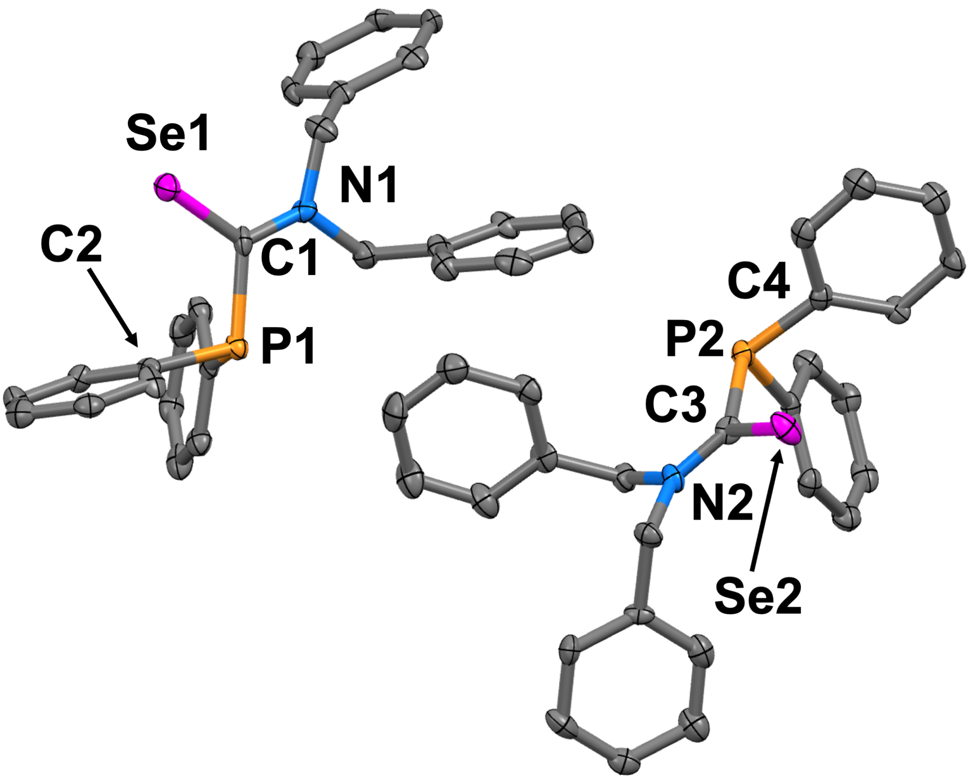


Figure S3. Molecular structure of **3a** with thermal ellipsoids at 50% probability (CCDC 2420890). Hydrogen atoms are omitted for clarity. Selected bond lengths [Å] and bond angles [°]: C1–Se1 1.836(12), C3–Se2 1.830(11), C1–P1 1.864(12), C3–P2 1.875(12), C1–N1 1.312(14), C3–N2 1.310(14); Se1–C1–P1–C2 0.5(8), Se2–C3–P2–C4 3.6(8).

X-ray crystallographic analysis of 3b.

Single crystals of **3b** were grown in their Et$_2$O-hexane solution. A reddish-orange crystal of **3b** was mounted on a loop. All measurements were made on a XtaLAB mini II (Rigaku Co.) with graphite monochromated Mo-Kα radiation (λ = 0.71073 Å) at –173 °C. Crystallographic and experimental data are listed in **Table S2**. The structures were solved and refined against all F_2 values using Shelx-2018[5] implemented through Olex2 v1.5. Nonhydrogen atoms were refined anisotropically. Hydrogen atoms were idealized using the riding models.

Table S2. Crystal data and structure refinement for **3b**.

Empirical formula	$C_{31}H_{20}F_{12}NPSe$	
Formula weight	744.41	
Temperature	99.97(18) K	
wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	$P2_12_12_1$	
Unit cell dimensions	a = 6.8359(5) Å	α = 90°

	b = 12.3127(10) Å	β = 90°
	c = 35.205(4) Å	γ = 90°
Volume	2963.1(5) Å^3	
Z	4	
D_{calc}	1.669 g/cm^3	
Absorption coefficient	1.421 mm^{-1}	
F(000)	1480.0	
Crystal size	0.20 x 0.16 x 0.05 mm^3	
Theta range for data collection	2.314 to 24.99°	
Index ranges	$-7 \le h \le 8$, $-14 \le k \le 14$, $-41 \le l \le 41$	
Reflections collected	15390	
Independent reflections	5123 [R_{int} = 0.0745]	
Max. and min. transmission	0.726 and 1.000	
Completeness to theta = 50.0°	99.9%	
data / restraints / parameters	5123 / 0 / 415	
Goodness-of-fit on F^2	0.981	
Final R induces (I>2σ(I))	R1 = 0.0520, wR2 = 0.0784	
R induces (all data)	R1 = 0.0929, wR2 = 0.0885	
Largest diff. peak and hole	0.42 and −0.42 e. Å^{-3}	

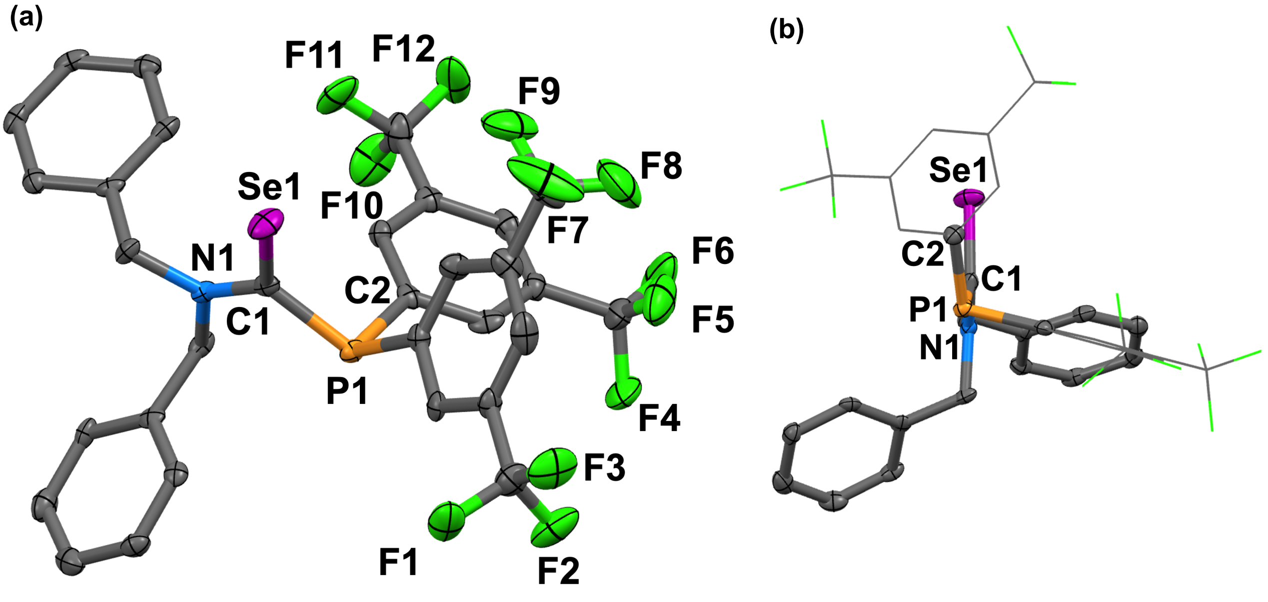


Figure S4. Molecular structure of **3b** with thermal ellipsoids at 50% probability (CCDC 2420891). Hydrogen atoms are omitted for clarity. (a) Drawn by ellipsoid style. (b) Side view. Selected bond lengths [Å] and bond angles [°]: C1–Se1 1.827(7), C1–P1 1.854(7), C1–N1 1.340(8); Se1–C1–P1–C2 1.6(4).

X-ray crystallographic analysis of 7.

Single crystals of **7** were grown in their AcOEt-hexane solution. A ruby-red crystal of **7** was mounted on a loop. All measurements were made on a XtaLAB mini II (Rigaku Co.) with graphite monochromated Mo-Kα radiation (λ = 0.71073 Å) at –173 °C. Crystallographic and experimental data are listed in **Table S3**. The structures were solved and refined against all F_2 values using Shelx-2018[5] implemented through Olex2 v1.5. Nonhydrogen atoms were refined anisotropically. Hydrogen atoms were idealized using the riding models.

Table S3. Crystal data and structure refinement for **7**.

Empirical formula	$C_{33}H_{27}Cl_2N_2PSe$	
Formula weight	632.39	
Temperature	101(3) K	
wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/n$	
Unit cell dimensions	a = 16.6851(3) Å	α = 90°
	b = 9.8964(2) Å	β = 105.983(2)°
	c = 18.0914(4) Å	γ = 90°
Volume	2871.82(10) Å^3	
Z	4	
D_{calc}	1.463 g/cm^3	
Absorption coefficient	1.576 mm^{-1}	
F(000)	1288.0	
Crystal size	0.25 x 0.21 x 0.12 mm^3	
Theta range for data collection	2.368 to 30.753°	
Index ranges	$-23 \leq h \leq 23$, $-12 \leq k \leq 14$, $-22 \leq l \leq 25$	
Reflections collected	31657	
Independent reflections	8607 [R_{int} = 0.0263]	
Max. and min. transmission	0.859 and 1.000	
Completeness to theta = 50.5°	99.9%	
data / restraints / parameters	8607 / 0 / 352	
Goodness-of-fit on F^2	1.019	
Final R induces (I>2σ(I))	$R1$ = 0.0309, w$R2$ = 0.0682	
R induces (all data)	$R1$ = 0.0460, w$R2$ = 0.0726	
Largest diff. peak and hole	0.62 and −0.33 e. Å^{-3}	

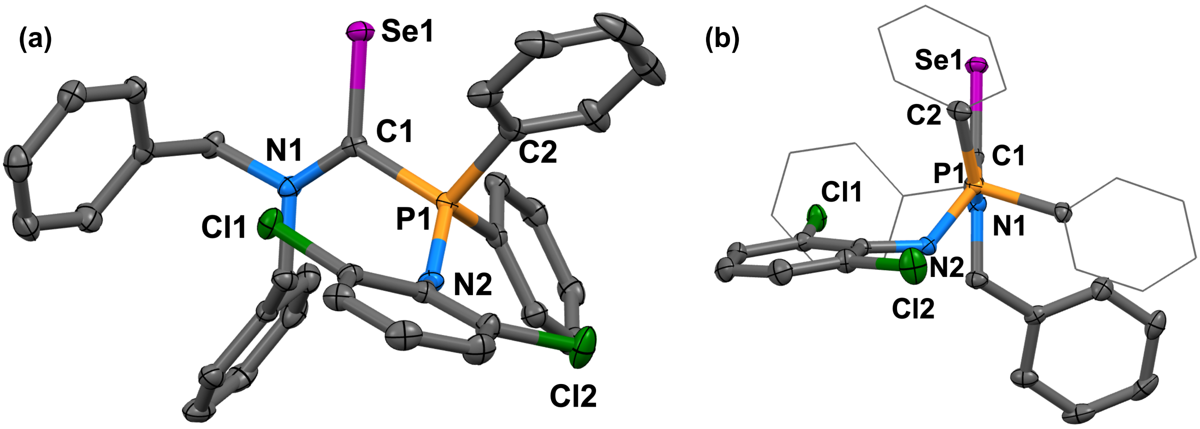


Figure S5. Molecular structure of **7** with thermal ellipsoids at 50% probability (CCDC 2420892). Hydrogen atoms are omitted for clarity. (a) Drawn by ellipsoid style. (b) Side view. Selected bond lengths [Å] and bond angles [°]: C1–Se1 1.8214(14), C1–P1 1.8704(15), C1–N1 1.3312(19), P1–N2 1.5581(13); Se1–C1–P1–N2 137.70(8), Se1–C1–P1–C2 11.06(10).

X-ray crystallographic analysis of 8.

Single crystals of **8** were grown in their AcOEt-hexane solution. A reddish-purple crystal of **8** was mounted on a loop. All measurements were made on a XtaLAB mini II (Rigaku Co.) with graphite monochromated Mo-Kα radiation (λ = 0.71073 Å) at –173 °C. Crystallographic and experimental data are listed in **Table S4**. The structures were solved and refined against all F_2 values using Shelx-2018[5] implemented through Olex2 v1.5. Nonhydrogen atoms were refined anisotropically. Hydrogen atoms were idealized using the riding models.

Table S4. Crystal data and structure refinement for **8**.

Empirical formula	$C_{27}H_{24}NPSe_2$	
Formula weight	551.36	
Temperature	100.00(10) K	
wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	*C*2/*c*	
Unit cell dimensions	a = 16.0502(5) Å	α = 90°
	b = 10.1153(4) Å	β = 103.173(3)°
	c = 29.8261(10) Å	γ = 90°
Volume	4714.9(3) Å^3	
Z	8	
D_{calc}	1.553 g/cm^3	
Absorption coefficient	3.220 mm^{-1}	
F(000)	2208.0	
Crystal size	0.22 x 0.20 x 0.11 mm^3	

Theta range for data collection	2.388 to 30.69°
Index ranges	$-22 \leq h \leq 21$, $-14 \leq k \leq 13$, $-41 \leq l \leq 42$
Reflections collected	252589
Independent reflections	7063 [$R_{\mathrm{int}} = 0.0263$]
Max. and min. transmission	0.627 and 1.000
Completeness to theta = 50.5°	99.9%
data / restraints / parameters	7063 / 0 / 280
Goodness-of-fit on F^2	1.073
Final R induces (I>2σ(I))	$R1 = 0.0280$, w$R2 = 0.0666$
R induces (all data)	$R1 = 0.0401$, w$R2 = 0.0697$
Largest diff. peak and hole	0.68 and −0.36 e. Å^{-3}

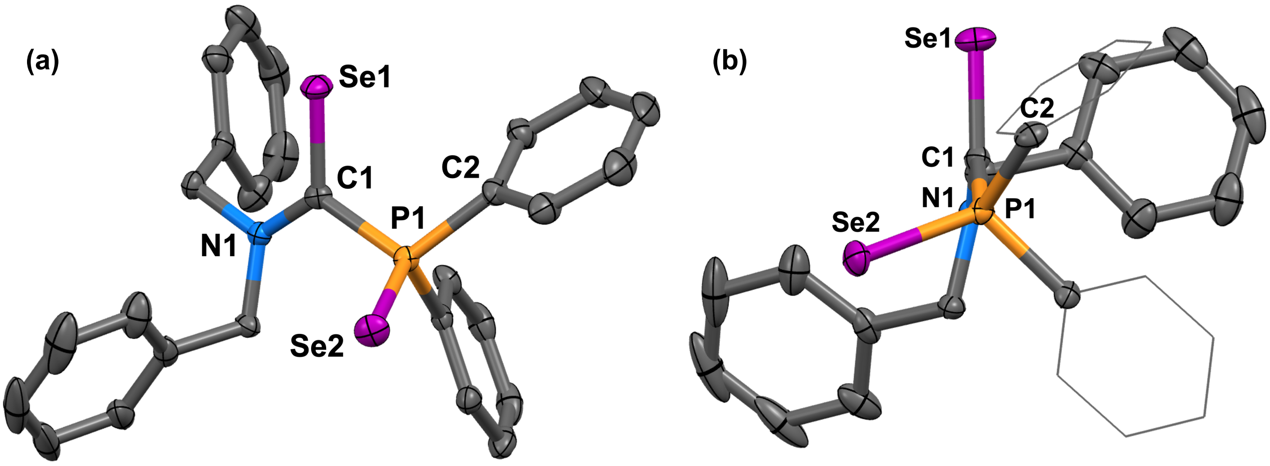


Figure S6. Molecular structure of **8** with thermal ellipsoids at 50% probability (CCDC 2420893). Hydrogen atoms are omitted for clarity. (a) Drawn by ellipsoid style. (b) Side view. Selected bond lengths [Å] and bond angles [°]: C1–Se1 1.8065(15), C1–P1 1.8515(15), C1–N1 1.3348(19), P1–Se2 2.1041(4); Se1–C1–P1–Se2 101.61(8), Se1–C1–P1–C2 25.49(11).

X-ray crystallographic analysis of 9a.

Single crystals of **9a** were grown in their AcOEt-hexane solution. A ruby-red crystal of **9a** was mounted on a loop. All measurements were made on a XtaLAB mini II (Rigaku Co.) with graphite monochromated Mo-Kα radiation (λ = 0.71073 Å) at –173 °C. Crystallographic and experimental data are listed in **Table S5**. The structures were solved and refined against all F_2 values using Shelx-2018[5] implemented through Olex2 v1.5. Nonhydrogen atoms were refined anisotropically. Hydrogen atoms were idealized using the riding models.

Table S5. Crystal data and structure refinement for **9a**.

Empirical formula	$C_{27}H_{24}NOPSe$
Formula weight	488.40

Temperature	100.00(10) K	
wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	*Pbca*	
Unit cell dimensions	a = 17.4280(6) Å	α = 90°
	b = 9.6355(3) Å	β = 90°
	c = 26.6337(6) Å	γ = 90°
Volume	4472.5(2) Å^3	
Z	8	
D_{calc}	1.451 g/cm^3	
Absorption coefficient	1.770 mm^{-1}	
F(000)	2000.0	
Crystal size	0.18 x 0.10 x 0.08 mm^3	
Theta range for data collection	2.337 to 30.64°	
Index ranges	$-23 \le h \le 24$, $-13 \le k \le 13$, $-37 \le l \le 38$	
Reflections collected	24587	
Independent reflections	6686 [R_{int} = 0.0248]	
Max. and min. transmission	0.748 and 1.000	
Completeness to theta = 50.5°	99.9%	
data / restraints / parameters	6686 / 0 / 280	
Goodness-of-fit on F^2	1.045	
Final R induces (I>2σ(I))	$R1$ = 0.0315, w$R2$ = 0.0779	
R induces (all data)	$R1$ = 0.0492, w$R2$ = 0.0830	
Largest diff. peak and hole	0.47 and −0.46 e. Å^{-3}	

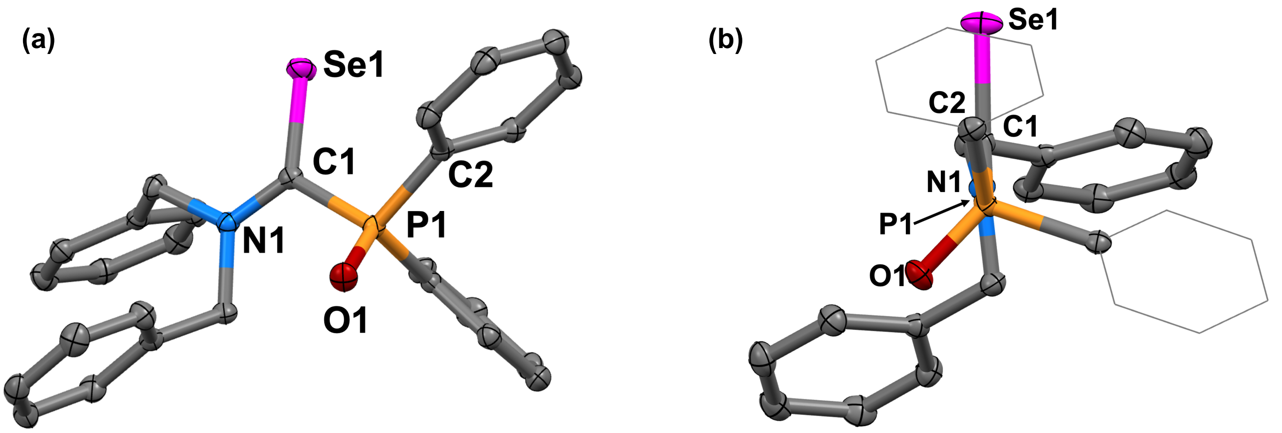


Figure S7. Molecular structure of **9c** with thermal ellipsoids at 50% probability (CCDC 2420894). Hydrogen atoms are omitted for clarity. (a) Drawn by ellipsoid style. (b) Side view. Selected bond lengths [Å] and bond angles [°]: C1–Se1 1.8163(15), C1–P1 1.8516(15), C1–N1 1.332(2), P1–O1 1.4840(11); Se1–C1–P1–O1 130.77(8), Se1–C1–P1–C2 7.45(11).

X-ray crystallographic analysis of 9c.

Single crystals of **9c** were grown in their AcOEt-hexane solution. A ruby-red crystal of **9c** was mounted on a loop. All measurements were made on a XtaLAB mini II (Rigaku Co.) with graphite monochromated Mo-Kα radiation (λ = 0.71073 Å) at –173 °C. Crystallographic and experimental data are listed in **Table S6**. The structures were solved and refined against all F_2 values using Shelx-2018[5] implemented through Olex2 v1.5. Nonhydrogen atoms were refined anisotropically. Hydrogen atoms were idealized using the riding models.

Table S6. Crystal data and structure refinement for **9c**.

Empirical formula	$C_{29}H_{28}NOPSe$	
Formula weight	516.45	
Temperature	100.4(9) K	
wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/n$	
Unit cell dimensions	a = 10.9052(2) Å	α = 90°
	b = 17.7869(4) Å	β = 98.305(2)°
	c = 12.6940(3) Å	γ = 90°
Volume	2436.43(9) Å^3	
Z	4	
D_{calc}	1.408 g/cm^3	
Absorption coefficient	1.629 mm^{-1}	
F(000)	1064.0	
Crystal size	0.22 x 0.17 x 0.11 mm^3	
Theta range for data collection	2.208 to 30.692°	
Index ranges	$-15 \leq h \leq 15$, $-24 \leq k \leq 25$, $-17 \leq l \leq 18$	
Reflections collected	25222	
Independent reflections	7284 [R_{int} = 0.0267]	
Max. and min. transmission	0.833 and 1.000	
Completeness to theta = 50.5°	99.9%	
data / restraints / parameters	7284 / 0 / 300	
Goodness-of-fit on F^2	1.035	
Final R induces (I>2σ(I))	$R1$ = 0.0334, w$R2$ = 0.0716	
R induces (all data)	$R1$ = 0.0497, w$R2$ = 0.0757	
Largest diff. peak and hole	0.47 and −0.35 e. Å^{-3}	

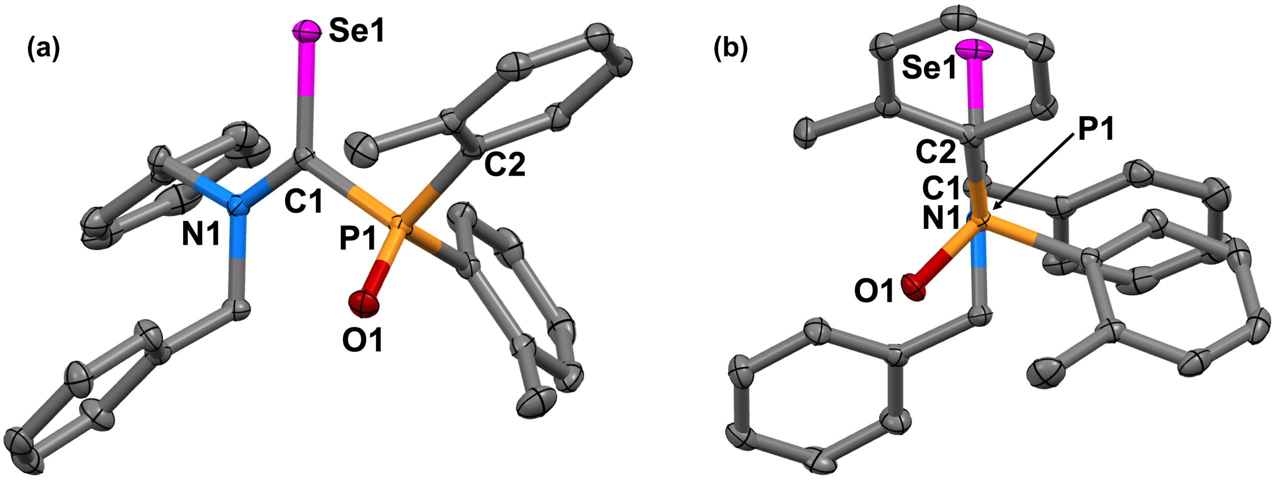


Figure S8. Molecular structure of **9c** with thermal ellipsoids at 50% probability (CCDC 2420895). Hydrogen atoms are omitted for clarity. (a) Drawn by ellipsoid style. (b) Side view. Selected bond lengths [Å] and bond angles [°]: C1–Se1 1.8151(15), C1–P1 1.8617(15), C1–N1 1.3332(18), P1–O1 1.4862(11); Se1–C1–P1–O1 129.25(8), Se1–C1–P1–C2 5.01(10).

X-ray crystallographic analysis of 13.

Single crystals of **13** were grown in their CH_2Cl_2-*n*-pentane solution. A pale-yellow crystal of **13** was mounted on a loop. All measurements were made on a XtaLAB mini II (Rigaku Co.) with graphite monochromated Mo-Kα radiation (λ = 0.71073 Å) at –173 °C. Crystallographic and experimental data are listed in **Table S7**. The structures were solved and refined against all F_2 values using Shelx-2018[5] implemented through Olex2 v1.5. Nonhydrogen atoms were refined anisotropically. Hydrogen atoms were idealized using the riding models.

Table S7. Crystal data and structure refinement for **13**.

Empirical formula	$C_{41}H_{39}Cl_2N_2O_3PPdSSe$	
Formula weight	927.03	
Temperature	100.0(2) K	
wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/n$	
Unit cell dimensions	a = 12.1825(5) Å	α = 90°
	b = 15.4090(6) Å	β = 105.099(4)°
	c = 21.4921(8) Å	γ = 90°
Volume	3895.2(3) Å^3	
Z	4	
D_{calc}	1.581 g/cm^3	
Absorption coefficient	1.684 mm^{-1}	
F(000)	1872.0	

Crystal size	0.22 x 0.21 x 0.11 mm^3
Theta range for data collection	2.178 to 30.76°
Index ranges	$-17 \leq h \leq 17$, $-16 \leq k \leq 21$, $-30 \leq l \leq 30$
Reflections collected	37587
Independent reflections	11683 [$R_{\mathrm{int}} = 0.0353$]
Max. and min. transmission	0.724 and 1.000
Completeness to theta = 50.5°	99.9%
data / restraints / parameters	11683 / 2 / 489
Goodness-of-fit on F^2	1.022
Final R induces (I>2σ(I))	$R1 = 0.0429$, w$R2 = 0.0761$
R induces (all data)	$R1 = 0.0673$, w$R2 = 0.0818$
Largest diff. peak and hole	0.94 and −0.88 e. Å^{-3}

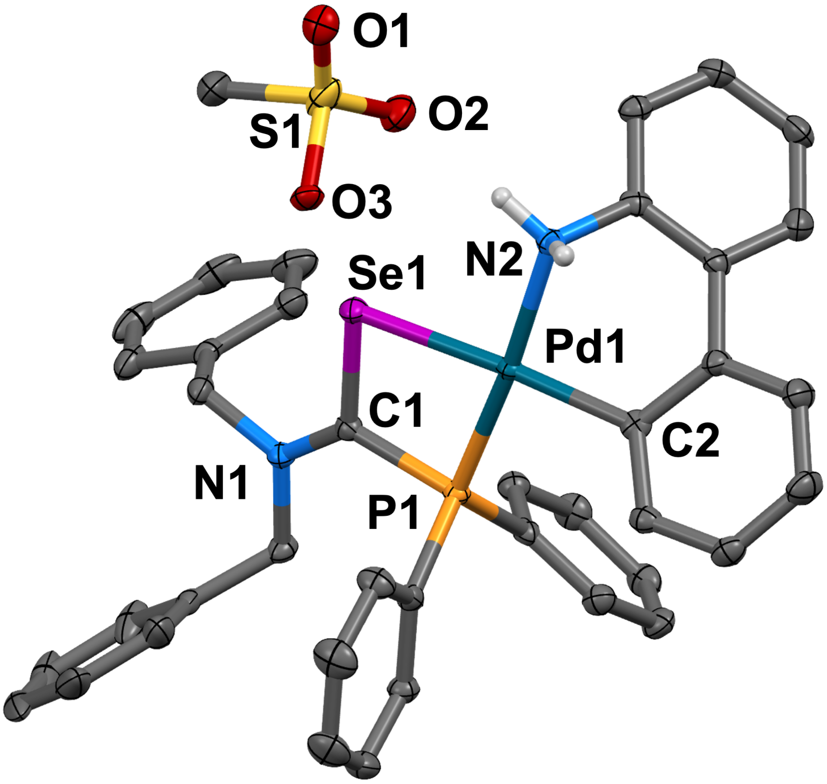


Figure S9. Molecular structure of **13** with thermal ellipsoids at 50% probability (CCDC 2420898). Hydrogen atoms except for the amino group are omitted for clarity. Selected bond lengths [Å] and bond angles [°]: C1–Se1 1.854(2), C1–P1 1.846(3), C1–N1 1.313(3), Se1–Pd1 2.4816(3), P1–Pd1 2.2202(6), N2–Pd1 2.096(2), Pd1–C2 2.023(3); P1–Pd1–Se1: 76.139(19), Se1–Pd1–N2: 98.26(6), P1–Pd1–C2: 99.63(7), N2–Pd1–C2: 85.93(0), C1–Se1–Pd1: 85.90(8), C1–P1–Pd1: 94.22(8), Se1–C1–P1: 103.54(12).

X-ray crystallographic analysis of 14.

Single crystals of **14** were grown in their CH_2Cl_2-hexane solution. A red crystal of **14** was mounted on a loop. All measurements were made on a XtaLAB mini II (Rigaku Co.) with graphite monochromated Mo-Kα radiation (λ = 0.71073 Å) at –173 °C. Crystallographic and experimental data are listed in **Table S8**. The structures were solved and refined against all F_2 values using Shelx-2018[5] implemented through Olex2 v1.5. Nonhydrogen atoms were refined anisotropically. Hydrogen atoms were idealized using the riding models.

Table S8. Crystal data and structure refinement for **14**.

Empirical formula	$C_{27}H_{24}NPClSeAu$	
Formula weight	704.82	
Temperature	99.9(3) K	
wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	*Pbca*	
Unit cell dimensions	a = 17.2293(7) Å	α = 90°
	b = 14.1326(5) Å	β = 90°
	c = 19.9231(7) Å	γ = 90°
Volume	4851.2(3) $Å^3$	
Z	8	
D_{calc}	1.930 g/cm^3	
Absorption coefficient	7.758 mm^{-1}	
$F(000)$	2704.0	
Crystal size	0.31 x 0.28 x 0.22 mm^3	
Theta range for data collection	2.363 to 24.99°	
Index ranges	$-20 \le h \le 20$, $-16 \le k \le 16$, $-23 \le l \le 23$	
Reflections collected	22371	
Independent reflections	4267 [R_{int} = 0.0603]	
Max. and min. transmission	0.047 and 0.141	
Completeness to theta = 50.0°	99.8%	
data / restraints / parameters	4267 / 0 / 289	
Goodness-of-fit on F^2	1.045	
Final R induces (I>2σ(I))	$R1$ = 0.0309, w$R2$ = 0.0752	
R induces (all data)	$R1$ = 0.0388, w$R2$ = 0.0785	
Largest diff. peak and hole	1.88 and −1.27 e. $Å^{-3}$	

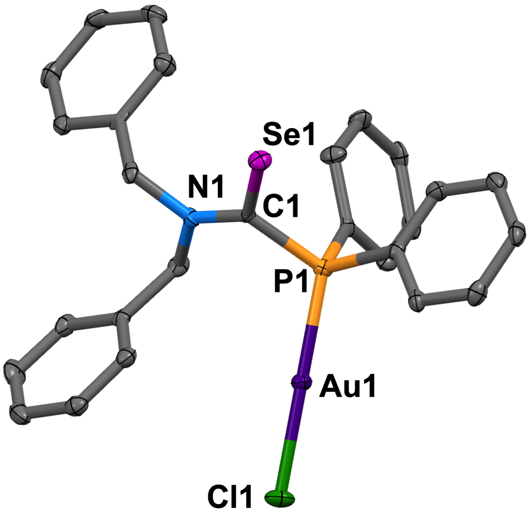


Figure S10. Molecular structure of **14** with thermal ellipsoids at 50% probability (CCDC 2420896). Hydrogen atoms except for the amino group are omitted for clarity. Selected bond lengths [Å] and bond angles [°]: C1–Se1 1.824(5), C1–P1 1.865(5), C1–N1 1.322(6), P1–Au1 2.2369(12); Se1–C1–N1: 125.4(3), Se1–C1–P1: 116.6(2), N1–C1–P1: 118.0(3), P1–Au1–Cl1: 177.07(4), Se1–C1–P1–Au1: 120.7(2).

3. Computational Analysis

All DFT calculations were carried out using the Gaussian 16 software.[6] Structures were visualized with Gauss View.

Reaction profile of the methylation of (selenocarbamoyl)phosphine 3a by MeOTf

The initial optimization of (selenocarbamoyl)phosphine **3a** was conducted based on its X-ray structure. The geometries of all relevant stationary points were optimized using the Heyd–Scuseria–Ernzerhof screened hybrid density (HSE06)[7] functional with Ahlrich's def2-SVP double-zeta basis set,[8] with the exception of selenium atoms, which were described using Dunning's correction consistent basis set of triple-zeta quality (aug-cc-pVTZ-pp)[9] including ECP. To take into account solvent effects, all structures were optimized in Et_2O employing the IEFPCM solvation model. This level of theory is here on denoted as IEFPCM(Et_2O)/HSE06/def2-SVP/aug-cc-pVTZ-pp(Se). All structures except for transition states were confirmed to be local minima on the potential energy surface by the absence of negative eigenvalues of the Hessian after harmonic frequency analysis at the same level. No symmetrical or internal coordinate constraints were applied. Additional single point computations on the optimized geometries were performed using the M062X[10] combined with the Grimme D3 dispersion correction functional with def2-TZVPP triple-zeta basis set, with the exception of selenium atoms, which were described using aug-cc-pVQZ-pp basis set[11], in Et_2O employing the SMD[12] solvation model. The reported Gibbs free energies (kcal·mol^{-1}) at 298.15K and 1 atm were obtained as the sum of the single point electronic energies and the respective thermal free energy corrections, as obtained from the harmonic frequency calculation.

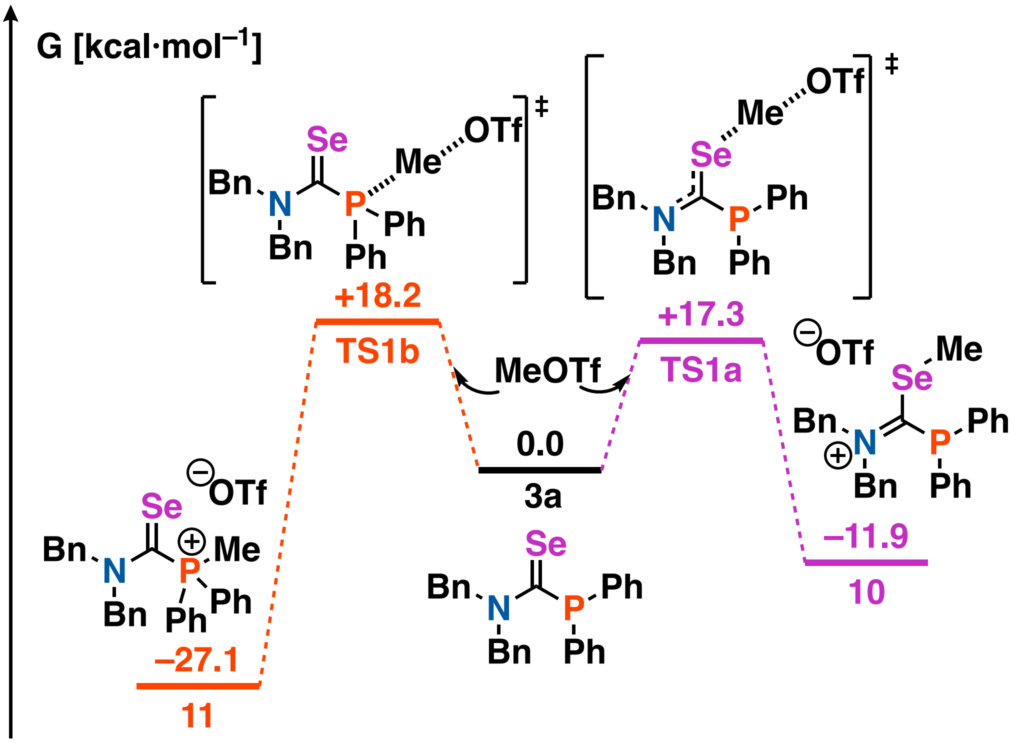


Figure S11. Reaction profile of the methylation of (selenocarbamoyl)phosphine **3a** with MeOTf.

Table S9. Stationary point energies of relevant structures.

	Free energy (kcal·mol^{-1})	Free energy (Hatree)	single point electronic energy (Hatree)	thermal correction (Hatree)
3a	–1137281.8	–1812.3724	–1812.746187	0.373758
3a + MeOTf	–1765657	–2813.7513	–2814.17667477	0.425422
TS1a	–1765639.7	–2813.7236	–2814.14951342	0.425892
10	–1765668.9	–2813.7702	–2814.19934599	0.429159
TS1b	–1765638.8	–2813.7222	–2814.14877412	0.426541
11	–1765684.2	–2813.7945	–2814.22457269	0.43011

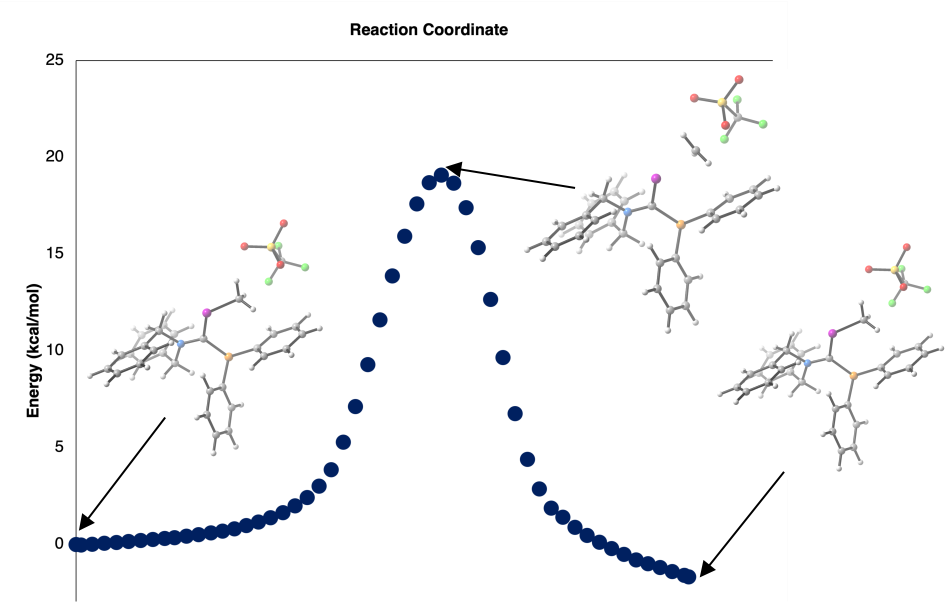


Figure S12. Analysis of total energy along IRC derived from **TS1a**.

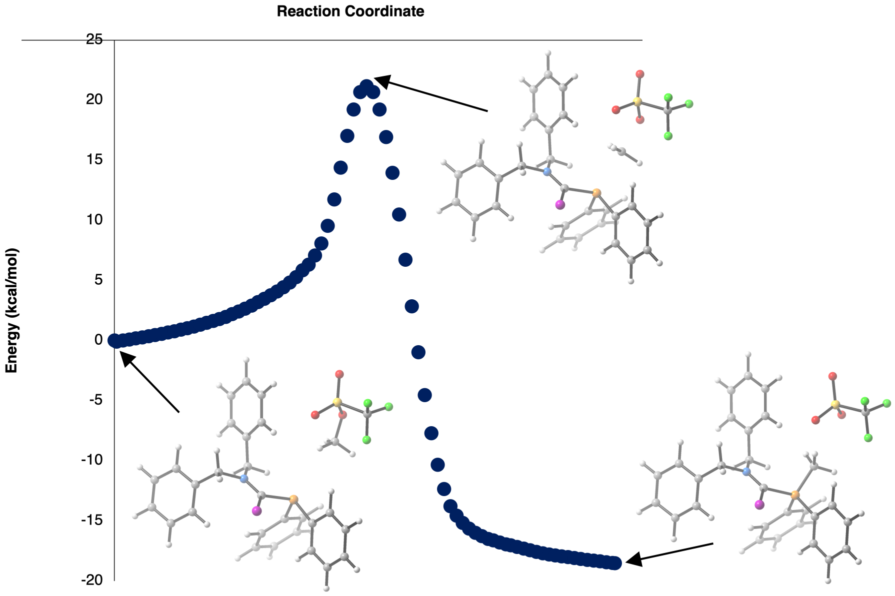
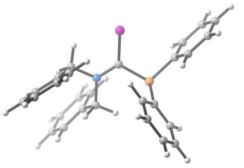


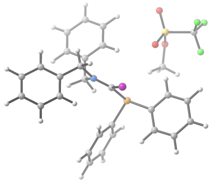
Figure S13. Analysis of total energy along IRC derived from **TS1b**.

3a



Se	0.06765656	-0.322696	-0.0499353
P	0.09665984	-0.1074089	3.14259221
N	2.34451825	-0.2975033	1.58439715
C	-4.3990994	0.14711944	2.0694743
H	-5.462028	0.23402522	1.83094811
C	5.23696076	0.95993621	3.39438824
H	5.71976253	-0.0123349	3.5287337
C	5.24973998	-4.0874477	-0.1370167
H	5.76795613	-5.0394564	-0.2755079
C	-2.4671415	-1.2272984	2.54649156
H	-2.0235907	-2.2166483	2.67821464
C	3.19436074	-2.8174822	-0.0341608
H	2.10403506	-2.770642	-0.1069912
C	0.75972738	-4.1671788	3.75562415
H	1.00871967	-5.0507991	3.16291291
C	1.0054601	-0.2904975	1.51084698
C	5.96521785	2.13205655	3.60150024
H	7.0153365	2.07361618	3.89793057
C	3.91617375	-1.641626	0.21628616
C	5.35585485	3.37313686	3.42362284
H	5.92563264	4.29212017	3.58081862
C	-1.6736853	-0.0807825	2.67773203
C	3.89218157	1.01507268	3.01504302
C	3.19932832	-0.327259	0.39477528
H	3.93022084	0.48978072	0.49258892
H	2.55352109	-0.1133031	-0.4683789
C	5.97639122	-2.9235069	0.10759511
H	7.06721428	-2.9588368	0.16270753
C	0.58037128	-4.276296	5.13457419
H	0.69013574	-5.2459931	5.62609202
C	-2.2660329	1.1807854	2.52695672
H	-1.6594655	2.08209552	2.65407914
C	-3.6187473	1.29474714	2.21232767
H	-4.0671963	2.2833786	2.08798315
C	0.26493856	-3.1432617	5.88534564
H	0.12690434	-3.2216752	6.96645015
C	5.31171517	-1.7090293	0.28529171
H	5.8853377	-0.7978369	0.47855254
C	-3.822603	-1.1118531	2.24341691
H	-4.4330371	-2.0123918	2.14033106
C	3.09711937	-0.2563986	2.83742204
H	3.76950196	-1.1289299	2.8619258
H	2.40397767	-0.3771948	3.68106352
C	0.13627841	-1.9045639	5.25929455
H	-0.0961842	-1.0166869	5.85438824
C	3.85616013	-4.031028	-0.2076498
H	3.28131356	-4.9388082	-0.4070323
C	0.30386449	-1.786727	3.87137138
C	0.61770338	-2.9310218	3.12504887
H	0.7481072	-2.858963	2.0422653
C	3.28709199	2.26594325	2.83947547
H	2.23557694	2.32150423	2.54432933
C	4.0148378	3.43689534	3.04045377
H	3.53117228	4.40654737	2.89968608

3a + MeOTf



Se	0	0	0
P	0	0	3.20236965
N	2.26208048	0	1.65441477
C	-4.489539	0.39609694	2.1433938
H	-5.551212	0.51756257	1.91454457
C	5.05670429	1.22065014	3.67138203
H	5.5743578	0.26120147	3.76157742
C	5.36456902	-3.5148967	-0.296486
H	5.93018203	-4.4281275	-0.4969229
C	-2.6089856	-1.0357614	2.65945275
H	-2.207317	-2.0367809	2.83082182
C	3.24630227	-2.3697575	-0.0743668
H	2.15348722	-2.3817392	-0.11291
C	0.57841718	-4.1104697	3.40096375
H	0.74584322	-4.9431055	2.71339643
C	0.92433284	-0.0308964	1.56677925
C	5.7190891	2.40206235	4.00752219
H	6.75292057	2.36309173	4.35950612
C	3.90927289	-1.1689416	0.21597855
C	5.06591219	3.62813127	3.88789767
H	5.5844137	4.55427277	4.14750664
C	-1.7662205	0.07986266	2.72709904
C	3.73396052	1.25154068	3.21860605
C	3.12704566	0.09324494	0.47609753
H	3.81585107	0.93643137	0.63801617
H	2.47915302	0.33485324	-0.3783295
C	6.03279977	-2.3254046	-0.0113547
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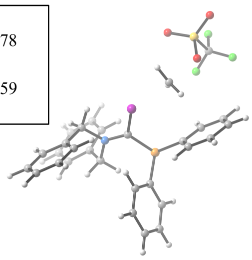

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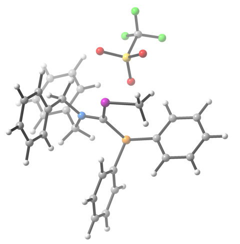

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H	2.99457514	-0.1178518	-2.2651246
C	-4.1089664	-0.3824546	3.49141914
H	-4.8460668	-1.1770244	3.63049791
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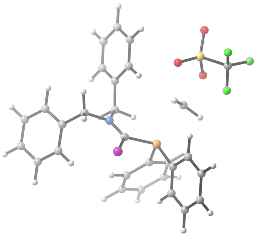

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C	3.12605418	-2.4373883	-0.1278489
H	2.03621869	-2.3837528	-0.204564
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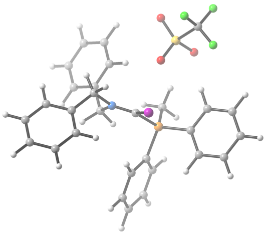

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H	2.00157713	-2.2104643	-0.4871655
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H	0.72193688	-4.8843974	2.77569673
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H	3.91495596	0.88831054	0.56632175
H	2.50911102	0.47103065	-0.4438128
C	5.87022186	-2.4775063	-0.3155256
H	6.95999541	-2.5438733	-0.2724593
C	0.34860272	-4.2474045	4.80750142
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H	-1.5040522	2.46664751	2.64350298
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H	-4.6806883	-1.3600876	2.29413707
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C	3.72503045	-3.4807206	-0.7777042
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C	0.42678399	-2.7560759	2.90863611
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C	3.5942936	2.24947571	2.97959707
H	2.68485159	2.47301441	2.41364982
C	4.38113592	3.30306351	3.44538196
H	4.05603903	4.32584177	3.23990531
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F	-1.7003162	4.5469907	0.77317325
F	0.01753409	5.32235131	-0.2735283
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O	0.76885821	3.04221477	1.50416705
O	-0.2573528	4.31490675	3.35574511
C	0.49083861	1.22722908	4.29901997
H	-0.1792484	1.11405369	5.16306795
H	0.34659754	2.233091	3.873257
H	1.53399183	1.10482811	4.61431234

Analysis of (selenocarbamoyl)phosphine 3a and selenoformamide 4

The initial optimizations of (selenocarbamoyl)phosphine **3a** and selenoformamide **4**[13] were conducted based on their X-ray structures. The geometries of all relevant stationary points were optimized using the TPSSTPSS combined with the Grimme D3 dispersion correction functional with Ahlrich's def2-SVP double-zeta basis set, with the exception of selenium atoms, which were described using def2-TZVP. This level of theory is here on denoted as TPSS-D3/def2-SVP/def2-TZVP(Se, ECP). All structures were confirmed to be local minima on the potential energy surface by the absence of negative eigenvalues of the Hessian after harmonic frequency analysis at the same level. No symmetrical or internal coordinate constraints were applied. Additional single point computations on the optimized geometries were performed using the M062X functional with def2-TZVPP triple-zeta basis set. Natural bond orbital (NBO) analyses were performed using the NBO 7.0 program[14] implemented in Gaussian.

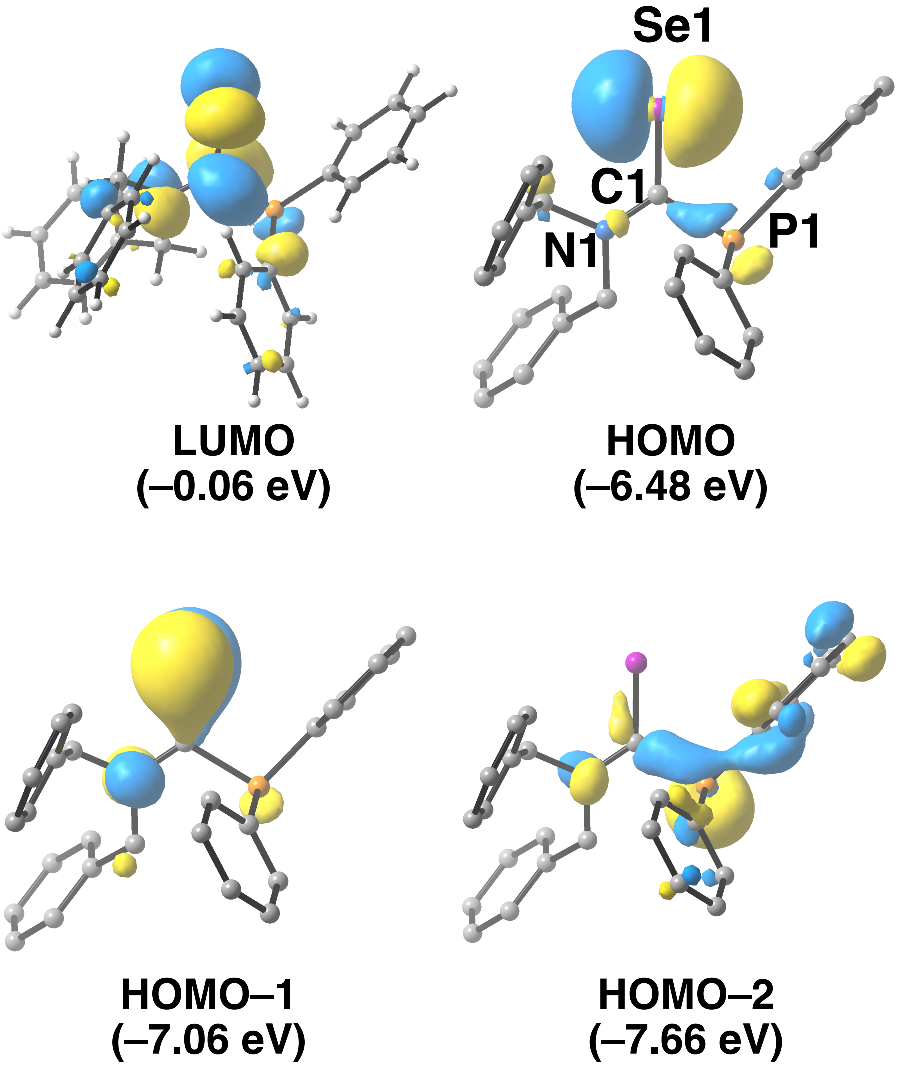
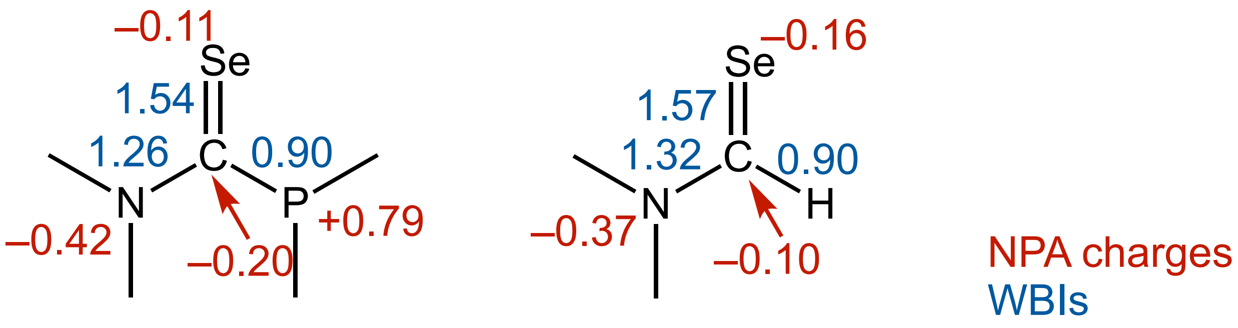


Figure S14. Selected frontier Kohn–Sham orbitals of **3a** (isovalues = 0.04).



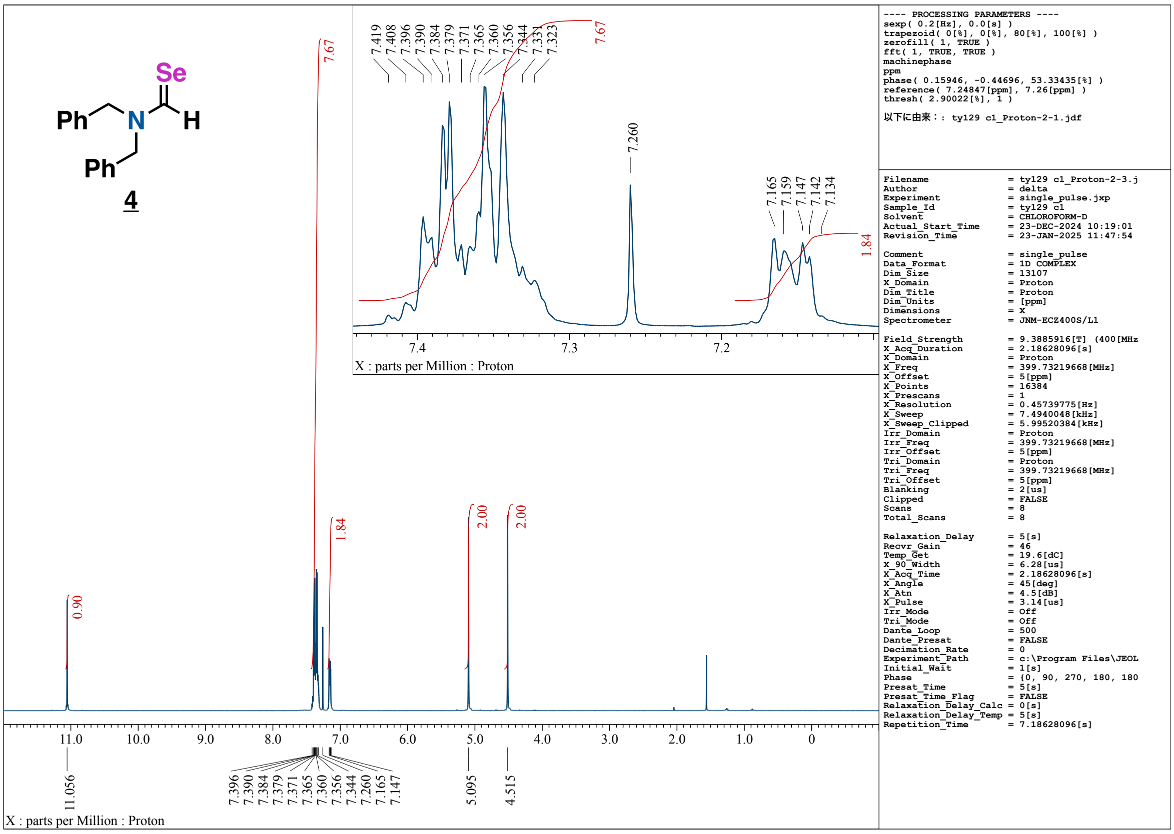
3a **4**

Figure S15. Natural population analysis (NPA) and Wiberg bond indices (WBIs) of **3a** and **4**.

Table S10. Selected results of the NBO analysis of **3a** and **4** (WBI = Wiberg Bond Indices).

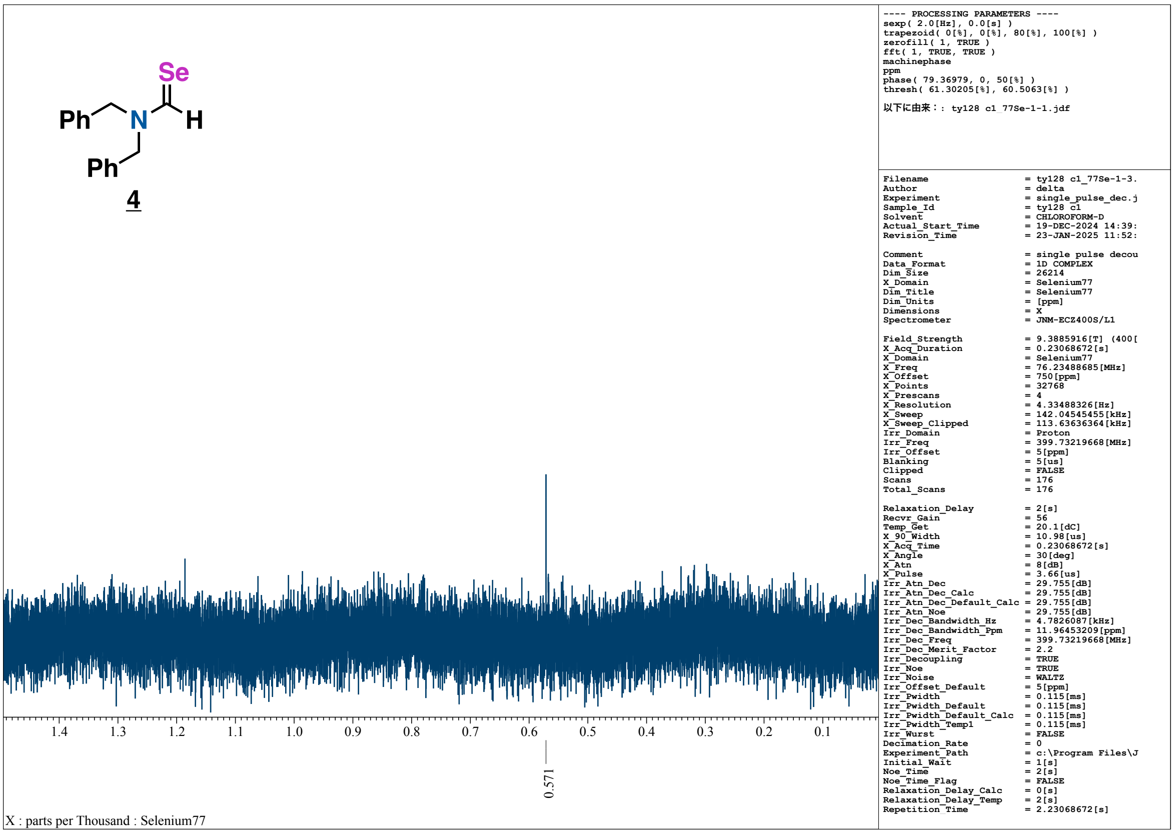
compd.	bond	occupancy	polarization (%)	hybridization	WBI
3a	σ(Se1–C1)	1.96	40.21 (Se1)	s (15.27%) p (84.38%) d (0.33%)	1.544
			59.79 (C1)	s (33.19%) p (66.53%) d (0.21%)	
3a	π(Se1–C1)	1.96	64.95 (Se1)	s (1.63%) p (98.13%) d (0.22%)	
			35.05 (C1)	s (3.2%) p (96.38%) d (0.35%)	
3a	σ(P1–C1)	1.95	37.02 (P1)	s (16.91%) p (82.48%) d (0.60%)	0.9033
			62.98 (C1)	s (31.35%) p (68.45%) d (0.16%)	
3a	σ(N1–C1)	1.99	64.82 (N1)	s (39.15%) p (60.64%) d (0.18%)	1.2593
			35.18 (C1)	s (32.21%) p (67.68%) d (0.08%)	
4	π(Se1–C1)	1.99	69.26 (Se1)	s (0.05%) p (99.71%) d (0.22%)	1.5731
			30.74 (C1)	s (0.13%) p (99.33%) d (0.47%)	
4	σ(C1–N1)	1.99	63.66 (N1)	s (38.10%) p (61.69%) d (0.18%)	1.3181
			36.34 (C1)	s (32.56%) p (67.33%) d (0.07%)	

4. NMR spectra



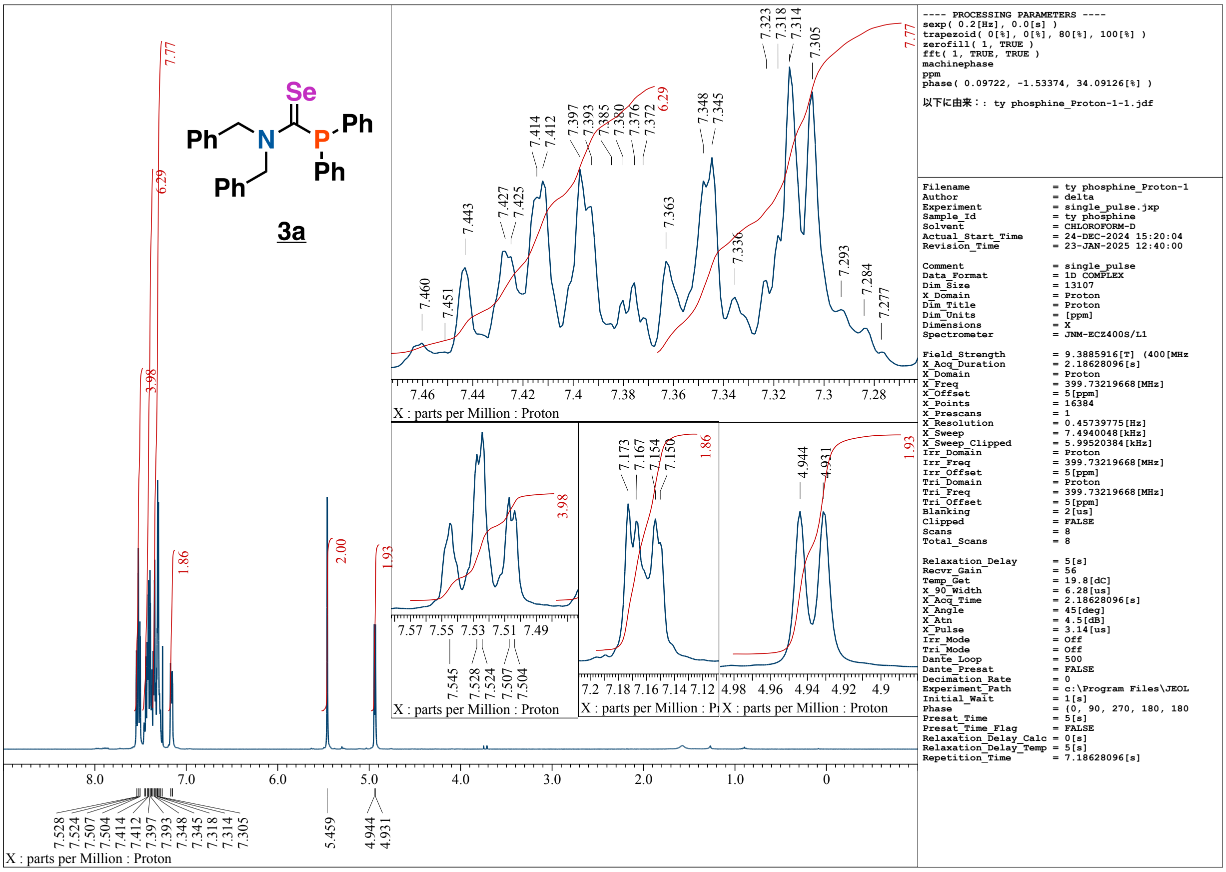
```
---- PROCESSING PARAMETERS ----
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( 0.15946, -0.44696, 53.33435[%] )
reference( 7.24847[ppm], 7.26[ppm] )
thresh( 2.90022[%], 1 )

以下に由来：: ty129 c1_Proton-2-1.jdf

Filename               = ty129 c1_Proton-2-3.j
Author                 = delta
Experiment             = single_pulse.jxp
Sample_Id              = ty129 c1
Solvent                = CHLOROFORM-D
Actual_Start_Time      = 23-DEC-2024 10:19:01
Revision_Time          = 23-JAN-2025 11:47:54

Comment                = single_pulse
Data_Format            = 1D COMPLEX
Dim_Size               = 13107
X_Domain               = Proton
Dim_Title              = Proton
Dim_Units              = [ppm]
Dimensions             = X
Spectrometer           = JNM-ECZ400S/L1

Field_Strength         = 9.3885916[T] (400[MHz
X_Acq_Duration         = 2.18628096[s]
X_Domain               = Proton
X_Freq                 = 399.73219668[MHz]
X_Offset               = 5[ppm]
X_Points               = 16384
X_Prescans             = 1
X_Resolution           = 0.45739775[Hz]
X_Sweep                = 7.4940048[kHz]
X_Sweep_Clipped        = 5.99520384[kHz]
Irr_Domain             = Proton
Irr_Freq               = 399.73219668[MHz]
Irr_Offset             = 5[ppm]
Tri_Domain             = Proton
Tri_Freq               = 399.73219668[MHz]
Tri_Offset             = 5[ppm]
Blanking               = 2[us]
Clipped                = FALSE
Scans                  = 8
Total_Scans            = 8

Relaxation_Delay       = 5[s]
Recvr_Gain             = 46
Temp_Get               = 19.6[dC]
X_90_Width             = 6.28[us]
X_Acq_Time             = 2.18628096[s]
X_Angle                = 45[deg]
X_Atn                  = 4.5[dB]
X_Pulse                = 3.14[us]
Irr_Mode               = Off
Tri_Mode               = Off
Dante_Loop             = 500
Dante_Presat           = FALSE
Decimation_Rate        = 0
Experiment_Path        = c:\Program Files\JEOL
Initial_Wait           = 1[s]
Phase                  = {0, 90, 270, 180, 180
Presat_Time            = 5[s]
Presat_Time_Flag       = FALSE
Relaxation_Delay_Calc  = 0[s]
Relaxation_Delay_Temp  = 5[s]
Repetition_Time        = 7.18628096[s]
```

Figure S16. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **4**.



```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( 79.36979, 0, 50[%] )
thresh( 61.30205[%], 60.5063[%] )

以下に由来：: ty128 c1_77Se-1-1.jdf

Filename                  = ty128 c1_77Se-1-3.
Author                    = delta
Experiment                = single_pulse_dec.j
Sample_Id                 = ty128 c1
Solvent                   = CHLOROFORM-D
Actual_Start_Time         = 19-DEC-2024 14:39:
Revision_Time             = 23-JAN-2025 11:52:

Comment                   = single pulse decou
Data_Format               = 1D COMPLEX
Dim_Size                  = 26214
X_Domain                  = Selenium77
Dim_Title                 = Selenium77
Dim_Units                 = [ppm]
Dimensions                = X
Spectrometer              = JNM-ECZ400S/L1

Field_Strength            = 9.3885916[T] (400[
X_Acq_Duration            = 0.23068672[s]
X_Domain                  = Selenium77
X_Freq                    = 76.23488685[MHz]
X_Offset                  = 750[ppm]
X_Points                  = 32768
X_Prescans                = 4
X_Resolution              = 4.33488326[Hz]
X_Sweep                   = 142.04545455[kHz]
X_Sweep_Clipped           = 113.63636364[kHz]
Irr_Domain                = Proton
Irr_Freq                  = 399.73219668[MHz]
Irr_Offset                = 5[ppm]
Blanking                  = 5[us]
Clipped                   = FALSE
Scans                     = 176
Total_Scans               = 176

Relaxation_Delay          = 2[s]
Recvr_Gain                = 56
Temp_Get                  = 20.1[dC]
X_90_Width                = 10.98[us]
X_Acq_Time                = 0.23068672[s]
X_Angle                   = 30[deg]
X_Atn                     = 8[dB]
X_Pulse                   = 3.66[us]
Irr_Atn_Dec               = 29.755[dB]
Irr_Atn_Dec_Calc          = 29.755[dB]
Irr_Atn_Dec_Default_Calc = 29.755[dB]
Irr_Atn_Noe               = 29.755[dB]
Irr_Dec_Bandwidth_Hz      = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm     = 11.96453209[ppm]
Irr_Dec_Freq              = 399.73219668[MHz]
Irr_Dec_Merit_Factor      = 2.2
Irr_Decoupling            = TRUE
Irr_Noe                   = TRUE
Irr_Noise                 = WALTZ
Irr_Offset_Default        = 5[ppm]
Irr_Pwidth                = 0.115[ms]
Irr_Pwidth_Default        = 0.115[ms]
Irr_Pwidth_Default_Calc   = 0.115[ms]
Irr_Pwidth_Temp1          = 0.115[ms]
Irr_Wurst                 = FALSE
Decimation_Rate           = 0
Experiment_Path           = c:\Program Files\J
Initial_Wait              = 1[s]
Noe_Time                  = 2[s]
Noe_Time_Flag             = FALSE
Relaxation_Delay_Calc     = 0[s]
Relaxation_Delay_Temp     = 2[s]
Repetition_Time           = 2.23068672[s]
```

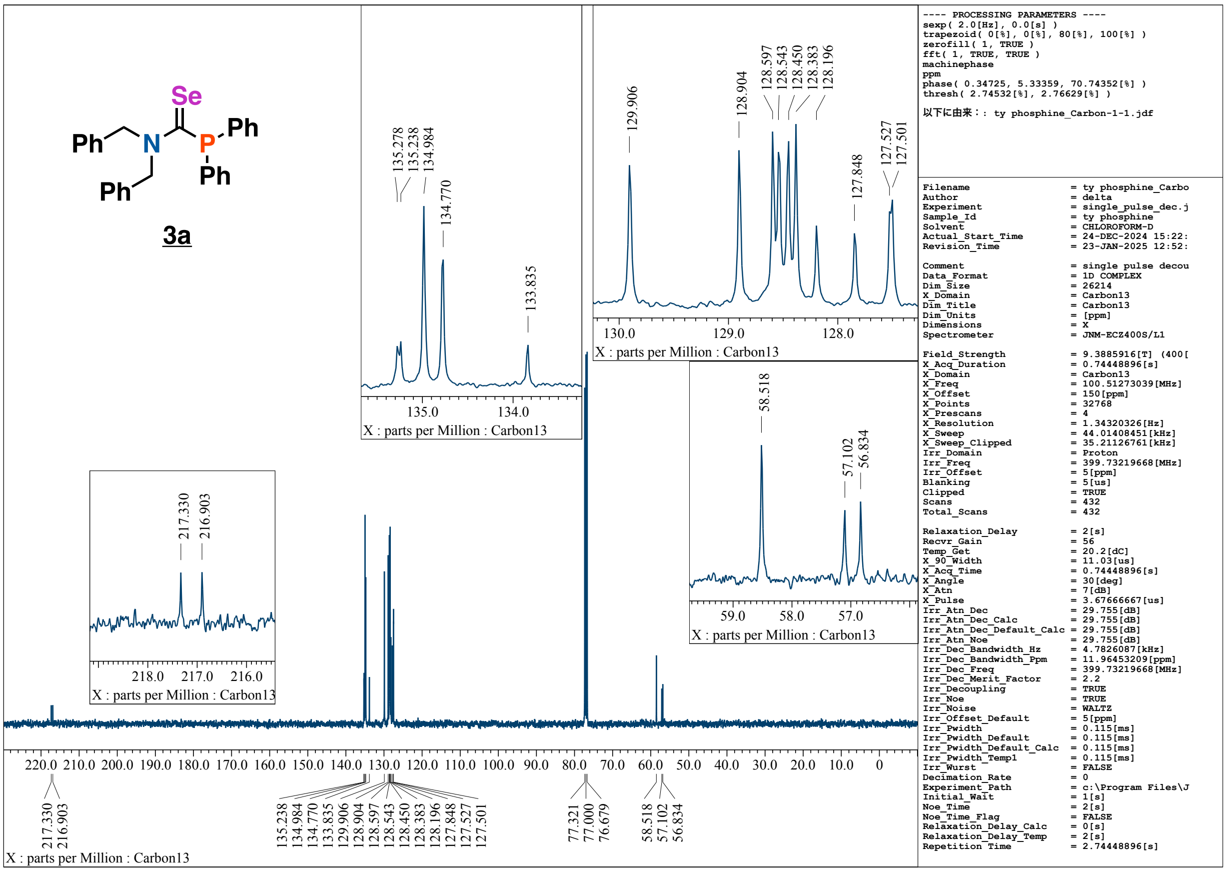
Figure S17. ^{77}Se{^{1}H} NMR spectrum (76 MHz, $CDCl_3$) of **4**.



```
---- PROCESSING PARAMETERS ----
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( 0.09722, -1.53374, 34.09126[%] )

以下に由来：: ty phosphine_Proton-1-1.jdf
```

```
Filename                  = ty phosphine_Proton-1
Author                    = delta
Experiment                = single_pulse.jxp
Sample_Id                 = ty phosphine
Solvent                   = CHLOROFORM-D
Actual_Start_Time         = 24-DEC-2024 15:20:04
Revision_Time             = 23-JAN-2025 12:40:00

Comment                   = single_pulse
Data_Format               = 1D COMPLEX
Dim_Size                  = 13107
X_Domain                  = Proton
Dim_Title                 = Proton
Dim_Units                 = [ppm]
Dimensions                = X
Spectrometer              = JNM-ECZ400S/L1

Field_Strength            = 9.3885916[T] (400[MHz
X_Acq_Duration            = 2.18628096[s]
X_Domain                  = Proton
X_Freq                    = 399.73219668[MHz]
X_Offset                  = 5[ppm]
X_Points                  = 16384
X_Prescans                = 1
X_Resolution              = 0.45739775[Hz]
X_Sweep                   = 7.4940048[kHz]
X_Sweep_Clipped           = 5.99520384[kHz]
Irr_Domain                = Proton
Irr_Freq                  = 399.73219668[MHz]
Irr_Offset                = 5[ppm]
Tri_Domain                = Proton
Tri_Freq                  = 399.73219668[MHz]
Tri_Offset                = 5[ppm]
Blanking                  = 2[us]
Clipped                   = FALSE
Scans                     = 8
Total_Scans               = 8

Relaxation_Delay          = 5[s]
Recvr_Gain                = 56
Temp_Get                  = 19.8[dC]
X_90_Width                = 6.28[us]
X_Acq_Time                = 2.18628096[s]
X_Angle                   = 45[deg]
X_Atn                     = 4.5[dB]
X_Pulse                   = 3.14[us]
Irr_Mode                  = Off
Tri_Mode                  = Off
Dante_Loop                = 500
Dante_Presat              = FALSE
Decimation_Rate           = 0
Experiment_Path           = c:\Program Files\JEOL
Initial_Wait              = 1[s]
Phase                     = {0, 90, 270, 180, 180
Presat_Time               = 5[s]
Presat_Time_Flag          = FALSE
Relaxation_Delay_Calc     = 0[s]
Relaxation_Delay_Temp     = 5[s]
Repetition_Time           = 7.18628096[s]
```

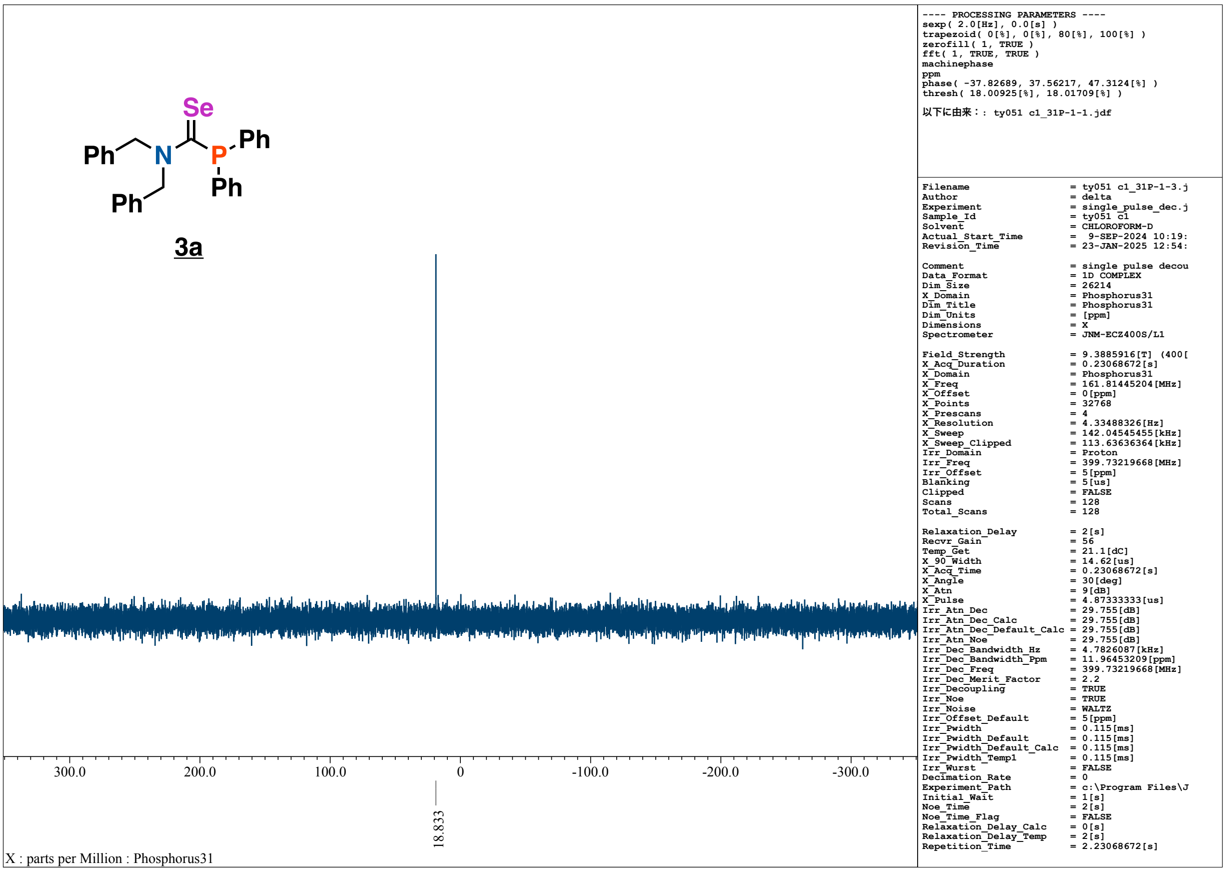
Figure S18. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **3a**.



```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( 0.34725, 5.33359, 70.74352[%] )
thresh( 2.74532[%], 2.76629[%] )

以下に由来：: ty phosphine_Carbon-1-1.jdf
```

```
Filename                    = ty phosphine_Carbo
Author                      = delta
Experiment                  = single_pulse_dec.j
Sample_Id                   = ty phosphine
Solvent                     = CHLOROFORM-D
Actual_Start_Time           = 24-DEC-2024 15:22:
Revision_Time               = 23-JAN-2025 12:52:

Comment                     = single pulse decou
Data_Format                 = 1D COMPLEX
Dim_Size                    = 26214
X_Domain                    = Carbon13
Dim_Title                   = Carbon13
Dim_Units                   = [ppm]
Dimensions                  = X
Spectrometer                = JNM-ECZ400S/L1

Field_Strength              = 9.3885916[T] (400[
X_Acq_Duration              = 0.74448896[s]
X_Domain                    = Carbon13
X_Freq                      = 100.51273039[MHz]
X_Offset                    = 150[ppm]
X_Points                    = 32768
X_Prescans                  = 4
X_Resolution                = 1.34320326[Hz]
X_Sweep                     = 44.01408451[kHz]
X_Sweep_Clipped             = 35.21126761[kHz]
Irr_Domain                  = Proton
Irr_Freq                    = 399.73219668[MHz]
Irr_Offset                  = 5[ppm]
Blanking                    = 5[us]
Clipped                     = TRUE
Scans                       = 432
Total_Scans                 = 432

Relaxation_Delay            = 2[s]
Recvr_Gain                  = 56
Temp_Get                    = 20.2[dC]
X_90_Width                  = 11.03[us]
X_Acq_Time                  = 0.74448896[s]
X_Angle                     = 30[deg]
X_Atn                       = 7[dB]
X_Pulse                     = 3.67666667[us]
Irr_Atn_Dec                 = 29.755[dB]
Irr_Atn_Dec_Calc            = 29.755[dB]
Irr_Atn_Dec_Default_Calc    = 29.755[dB]
Irr_Atn_Noe                 = 29.755[dB]
Irr_Dec_Bandwidth_Hz        = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm       = 11.96453209[ppm]
Irr_Dec_Freq                = 399.73219668[MHz]
Irr_Dec_Merit_Factor        = 2.2
Irr_Decoupling              = TRUE
Irr_Noe                     = TRUE
Irr_Noise                   = WALTZ
Irr_Offset_Default          = 5[ppm]
Irr_Pwidth                  = 0.115[ms]
Irr_Pwidth_Default          = 0.115[ms]
Irr_Pwidth_Default_Calc     = 0.115[ms]
Irr_Pwidth_Temp1            = 0.115[ms]
Irr_Wurst                   = FALSE
Decimation_Rate             = 0
Experiment_Path             = c:\Program Files\J
Initial_Wait                = 1[s]
Noe_Time                    = 2[s]
Noe_Time_Flag               = FALSE
Relaxation_Delay_Calc       = 0[s]
Relaxation_Delay_Temp       = 2[s]
Repetition Time             = 2.74448896[s]
```

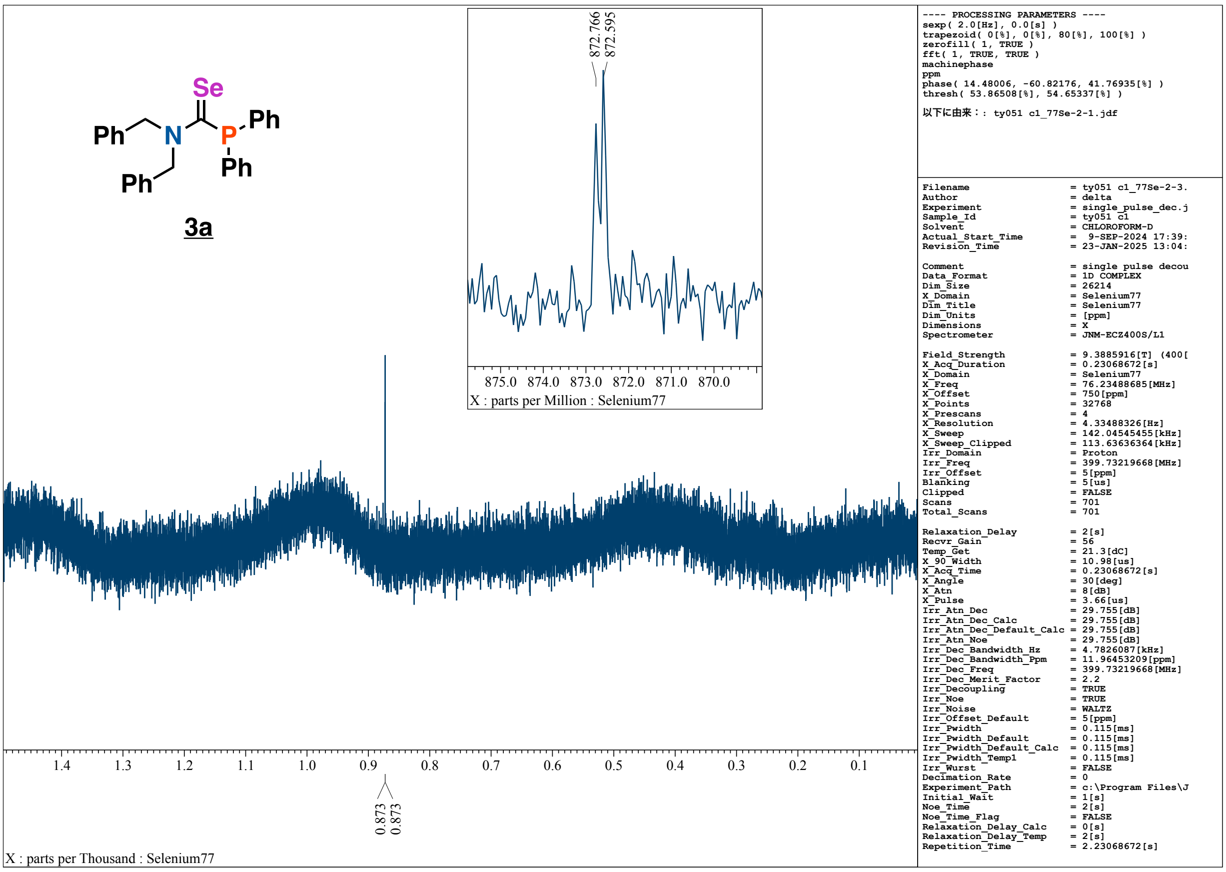
Figure S19. $^{13}C\{^{1}H\}$ NMR spectrum (100 MHz, $CDCl_3$) of **3a**.



```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( -37.82689, 37.56217, 47.3124[%] )
thresh( 18.00925[%], 18.01709[%] )

以下に由来：: ty051 c1_31P-1-1.jdf
```

```
Filename                    = ty051 c1_31P-1-3.j
Author                      = delta
Experiment                  = single_pulse_dec.j
Sample_Id                   = ty051 c1
Solvent                     = CHLOROFORM-D
Actual_Start_Time           =  9-SEP-2024 10:19:
Revision_Time               = 23-JAN-2025 12:54:

Comment                     = single pulse decou
Data_Format                 = 1D COMPLEX
Dim_Size                    = 26214
X_Domain                    = Phosphorus31
Dim_Title                   = Phosphorus31
Dim_Units                   = [ppm]
Dimensions                  = X
Spectrometer                = JNM-ECZ400S/L1

Field_Strength              = 9.3885916[T] (400[
X_Acq_Duration              = 0.23068672[s]
X_Domain                    = Phosphorus31
X_Freq                      = 161.81445204[MHz]
X_Offset                    = 0[ppm]
X_Points                    = 32768
X_Prescans                  = 4
X_Resolution                = 4.33488326[Hz]
X_Sweep                     = 142.04545455[kHz]
X_Sweep_Clipped             = 113.63636364[kHz]
Irr_Domain                  = Proton
Irr_Freq                    = 399.73219668[MHz]
Irr_Offset                  = 5[ppm]
Blanking                    = 5[us]
Clipped                     = FALSE
Scans                       = 128
Total_Scans                 = 128

Relaxation_Delay            = 2[s]
Recvr_Gain                  = 56
Temp_Get                    = 21.1[dC]
X_90_Width                  = 14.62[us]
X_Acq_Time                  = 0.23068672[s]
X_Angle                     = 30[deg]
X_Atn                       = 9[dB]
X_Pulse                     = 4.87333333[us]
Irr_Atn_Dec                 = 29.755[dB]
Irr_Atn_Dec_Calc            = 29.755[dB]
Irr_Atn_Dec_Default_Calc    = 29.755[dB]
Irr_Atn_Noe                 = 29.755[dB]
Irr_Dec_Bandwidth_Hz        = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm       = 11.96453209[ppm]
Irr_Dec_Freq                = 399.73219668[MHz]
Irr_Dec_Merit_Factor        = 2.2
Irr_Decoupling              = TRUE
Irr_Noe                     = TRUE
Irr_Noise                   = WALTZ
Irr_Offset_Default          = 5[ppm]
Irr_Pwidth                  = 0.115[ms]
Irr_Pwidth_Default          = 0.115[ms]
Irr_Pwidth_Default_Calc     = 0.115[ms]
Irr_Pwidth_Temp1            = 0.115[ms]
Irr_Wurst                   = FALSE
Decimation_Rate             = 0
Experiment_Path             = c:\Program Files\J
Initial_Wait                = 1[s]
Noe_Time                    = 2[s]
Noe_Time_Flag               = FALSE
Relaxation_Delay_Calc       = 0[s]
Relaxation_Delay_Temp       = 2[s]
Repetition_Time             = 2.23068672[s]
```

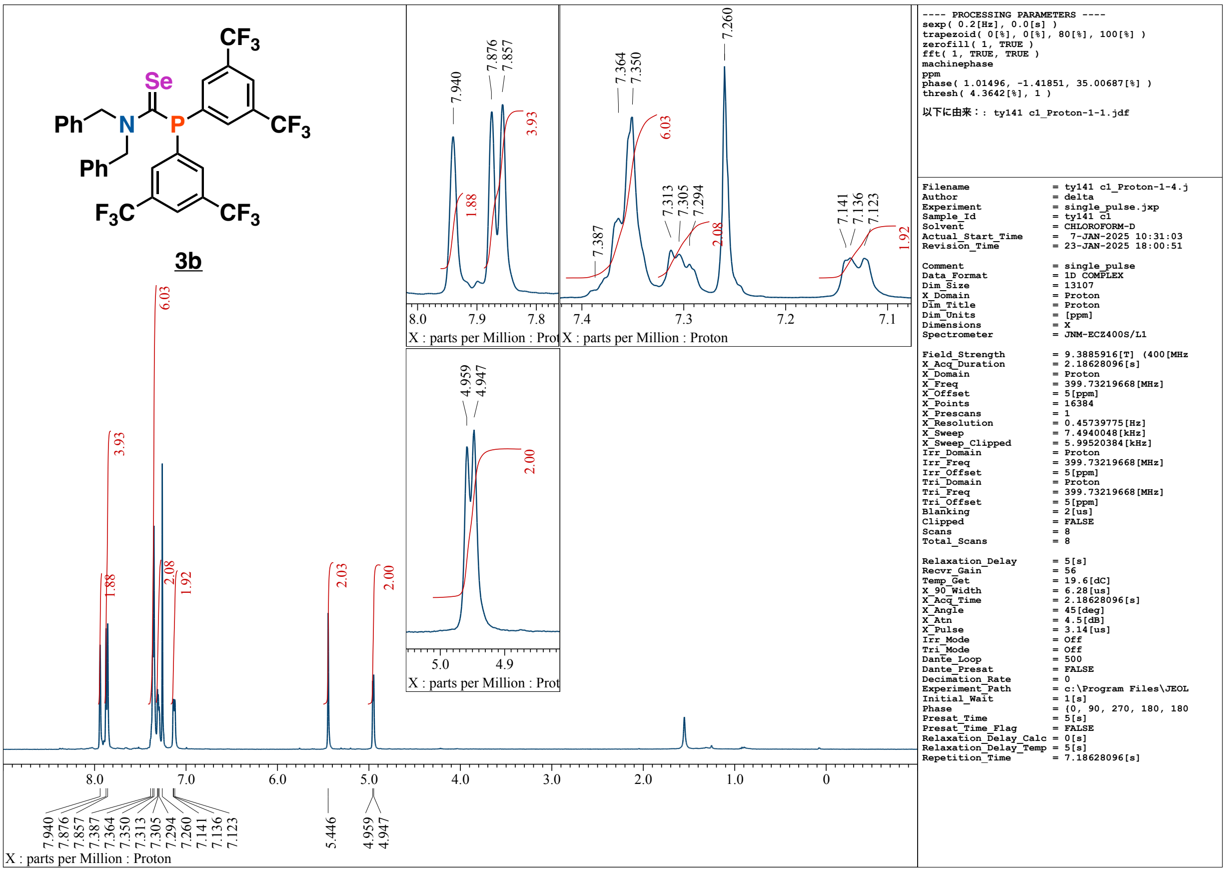
Figure S20. $^{31}P\{^1H\}$ NMR spectrum (162 MHz, $CDCl_3$) of **3a**.



```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( 14.48006, -60.82176, 41.76935[%] )
thresh( 53.86508[%], 54.65337[%] )

以下に由来：: ty051 c1_77Se-2-1.jdf
```

```
Filename                    = ty051 c1_77Se-2-3.
Author                      = delta
Experiment                  = single_pulse_dec.j
Sample_Id                   = ty051 c1
Solvent                     = CHLOROFORM-D
Actual_Start_Time           =  9-SEP-2024 17:39:
Revision_Time               = 23-JAN-2025 13:04:

Comment                     = single pulse decou
Data_Format                 = 1D COMPLEX
Dim_Size                    = 26214
X_Domain                    = Selenium77
Dim_Title                   = Selenium77
Dim_Units                   = [ppm]
Dimensions                  = X
Spectrometer                = JNM-ECZ400S/L1

Field_Strength              = 9.3885916[T] (400[
X_Acq_Duration              = 0.23068672[s]
X_Domain                    = Selenium77
X_Freq                      = 76.23488685[MHz]
X_Offset                    = 750[ppm]
X_Points                    = 32768
X_Prescans                  = 4
X_Resolution                = 4.33488326[Hz]
X_Sweep                     = 142.04545455[kHz]
X_Sweep_Clipped             = 113.63636364[kHz]
Irr_Domain                  = Proton
Irr_Freq                    = 399.73219668[MHz]
Irr_Offset                  = 5[ppm]
Blanking                    = 5[us]
Clipped                     = FALSE
Scans                       = 701
Total_Scans                 = 701

Relaxation_Delay            = 2[s]
Recvr_Gain                  = 56
Temp_Get                    = 21.3[dC]
X_90_Width                  = 10.98[us]
X_Acq_Time                  = 0.23068672[s]
X_Angle                     = 30[deg]
X_Atn                       = 8[dB]
X_Pulse                     = 3.66[us]
Irr_Atn_Dec                 = 29.755[dB]
Irr_Atn_Dec_Calc            = 29.755[dB]
Irr_Atn_Dec_Default_Calc    = 29.755[dB]
Irr_Atn_Noe                 = 29.755[dB]
Irr_Dec_Bandwidth_Hz        = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm       = 11.96453209[ppm]
Irr_Dec_Freq                = 399.73219668[MHz]
Irr_Dec_Merit_Factor        = 2.2
Irr_Decoupling              = TRUE
Irr_Noe                     = TRUE
Irr_Noise                   = WALTZ
Irr_Offset_Default          = 5[ppm]
Irr_Pwidth                  = 0.115[ms]
Irr_Pwidth_Default          = 0.115[ms]
Irr_Pwidth_Default_Calc     = 0.115[ms]
Irr_Pwidth_Temp1            = 0.115[ms]
Irr_Wurst                   = FALSE
Decimation_Rate             = 0
Experiment_Path             = c:\Program Files\J
Initial_Wait                = 1[s]
Noe_Time                    = 2[s]
Noe_Time_Flag               = FALSE
Relaxation_Delay_Calc       = 0[s]
Relaxation_Delay_Temp       = 2[s]
Repetition_Time             = 2.23068672[s]
```

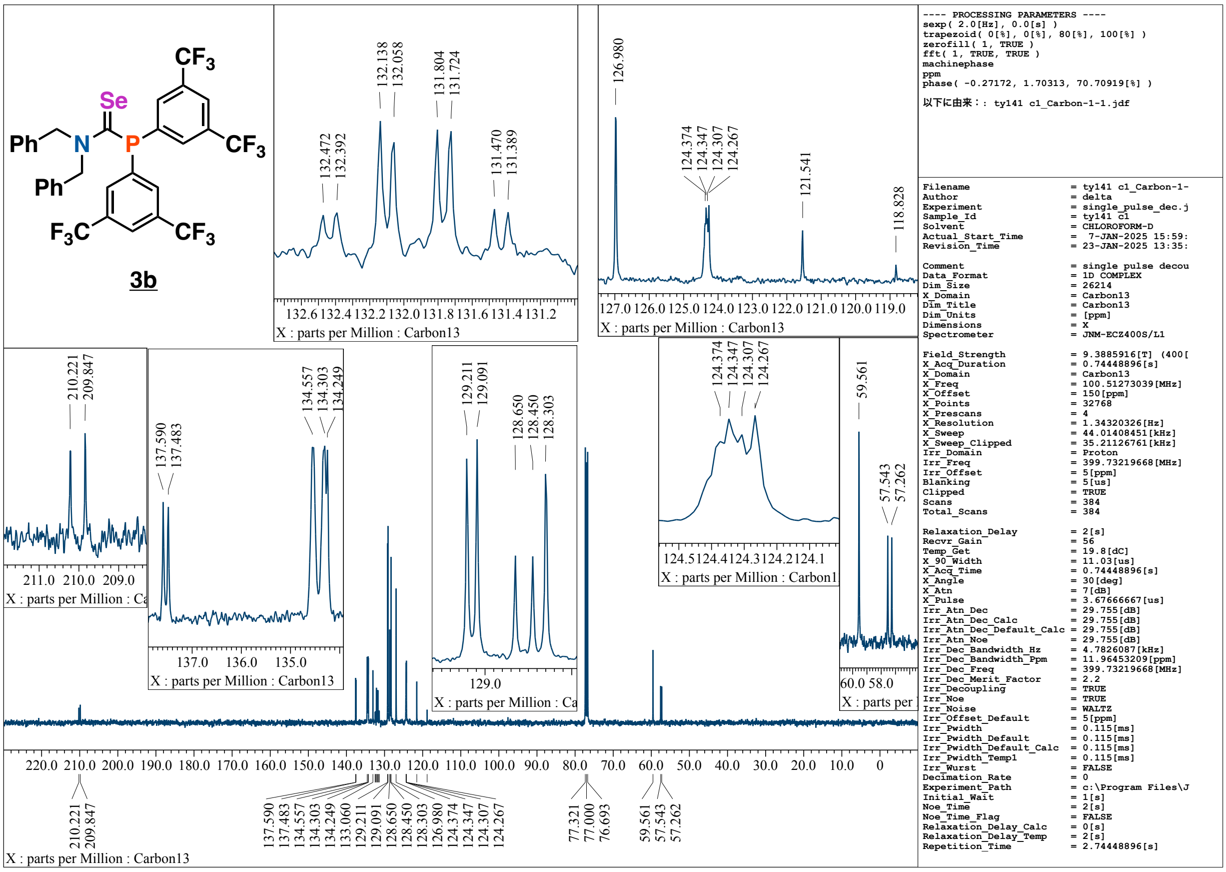
Figure S21. $^{77}Se\{^1H\}$ NMR spectrum (76 MHz, $CDCl_3$) of **3a**.



```
---- PROCESSING PARAMETERS ----
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( 1.01496, -1.41851, 35.00687[%] )
thresh( 4.3642[%], 1 )

以下に由来：: ty141 c1_Proton-1-1.jdf
```

```
Filename                = ty141 c1_Proton-1-4.j
Author                  = delta
Experiment              = single_pulse.jxp
Sample_Id               = ty141 c1
Solvent                 = CHLOROFORM-D
Actual_Start_Time       =  7-JAN-2025 10:31:03
Revision_Time           = 23-JAN-2025 18:00:51

Comment                 = single_pulse
Data_Format             = 1D COMPLEX
Dim_Size                = 13107
X_Domain                = Proton
Dim_Title               = Proton
Dim_Units               = [ppm]
Dimensions              = X
Spectrometer            = JNM-ECZ400S/L1

Field_Strength          = 9.3885916[T] (400[MHz
X_Acq_Duration          = 2.18628096[s]
X_Domain                = Proton
X_Freq                  = 399.73219668[MHz]
X_Offset                = 5[ppm]
X_Points                = 16384
X_Prescans              = 1
X_Resolution            = 0.45739775[Hz]
X_Sweep                 = 7.4940048[kHz]
X_Sweep_Clipped         = 5.99520384[kHz]
Irr_Domain              = Proton
Irr_Freq                = 399.73219668[MHz]
Irr_Offset              = 5[ppm]
Tri_Domain              = Proton
Tri_Freq                = 399.73219668[MHz]
Tri_Offset              = 5[ppm]
Blanking                = 2[us]
Clipped                 = FALSE
Scans                   = 8
Total_Scans             = 8

Relaxation_Delay        = 5[s]
Recvr_Gain              = 56
Temp_Get                = 19.6[dC]
X_90_Width              = 6.28[us]
X_Acq_Time              = 2.18628096[s]
X_Angle                 = 45[deg]
X_Atn                   = 4.5[dB]
X_Pulse                 = 3.14[us]
Irr_Mode                = Off
Tri_Mode                = Off
Dante_Loop              = 500
Dante_Presat            = FALSE
Decimation_Rate         = 0
Experiment_Path         = c:\Program Files\JEOL
Initial_Wait            = 1[s]
Phase                   = {0, 90, 270, 180, 180
Presat_Time             = 5[s]
Presat_Time_Flag        = FALSE
Relaxation_Delay_Calc   = 0[s]
Relaxation_Delay_Temp   = 5[s]
Repetition_Time         = 7.18628096[s]
```

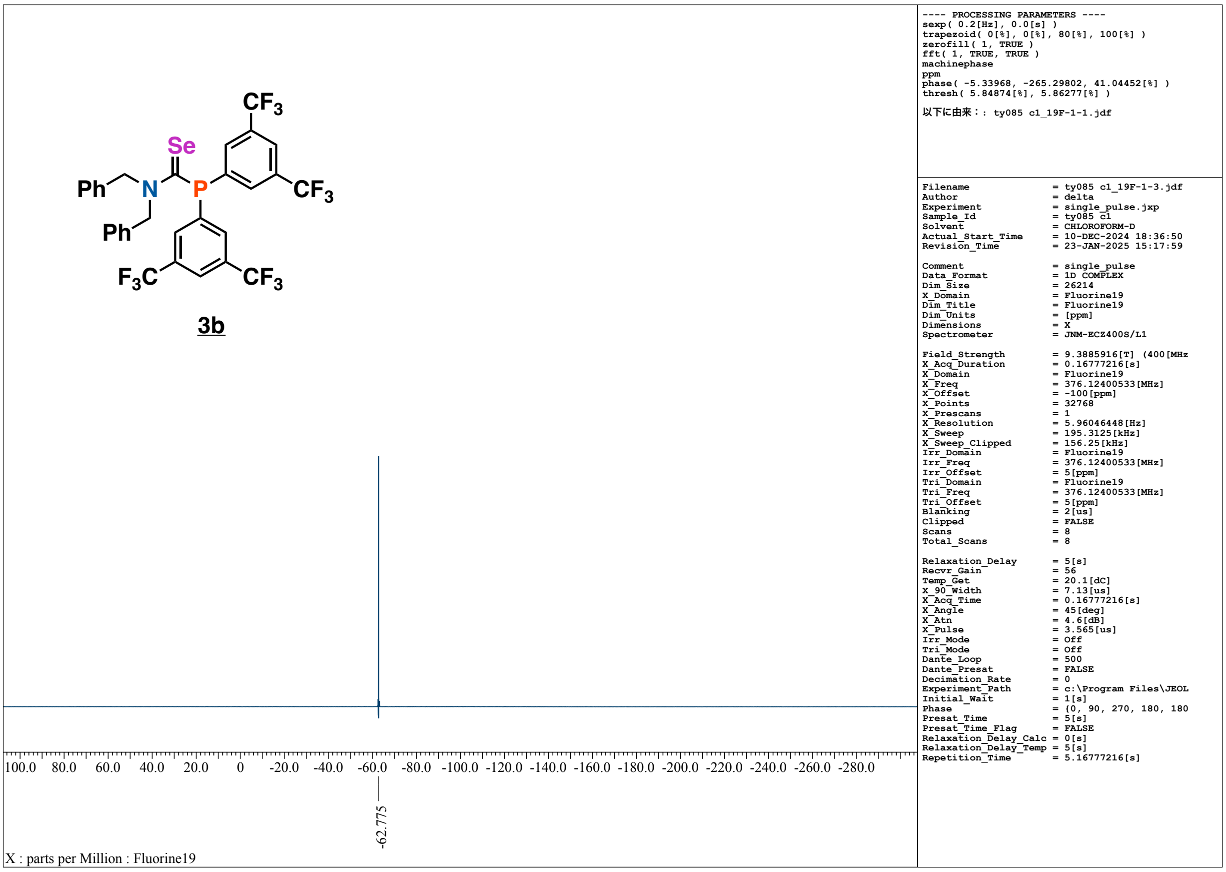
Figure S22. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **3b**.



```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( -0.27172, 1.70313, 70.70919[%] )

以下に由来：: ty141 c1_Carbon-1-1.jdf
```

```
Filename                  = ty141 c1_Carbon-1-
Author                    = delta
Experiment                = single_pulse_dec.j
Sample_Id                 = ty141 c1
Solvent                   = CHLOROFORM-D
Actual_Start_Time         =  7-JAN-2025 15:59:
Revision_Time             = 23-JAN-2025 13:35:

Comment                   = single pulse decou
Data_Format               = 1D COMPLEX
Dim_Size                  = 26214
X_Domain                  = Carbon13
Dim_Title                 = Carbon13
Dim_Units                 = [ppm]
Dimensions                = X
Spectrometer              = JNM-ECZ400S/L1

Field_Strength            = 9.3885916[T] (400[
X_Acq_Duration            = 0.74448896[s]
X_Domain                  = Carbon13
X_Freq                    = 100.51273039[MHz]
X_Offset                  = 150[ppm]
X_Points                  = 32768
X_Prescans                = 4
X_Resolution              = 1.34320326[Hz]
X_Sweep                   = 44.01408451[kHz]
X_Sweep_Clipped           = 35.21126761[kHz]
Irr_Domain                = Proton
Irr_Freq                  = 399.73219668[MHz]
Irr_Offset                = 5[ppm]
Blanking                  = 5[us]
Clipped                   = TRUE
Scans                     = 384
Total_Scans               = 384

Relaxation_Delay          = 2[s]
Recvr_Gain                = 56
Temp_Get                  = 19.8[dC]
X_90_Width                = 11.03[us]
X_Acq_Time                = 0.74448896[s]
X_Angle                   = 30[deg]
X_Atn                     = 7[dB]
X_Pulse                   = 3.67666667[us]
Irr_Atn_Dec               = 29.755[dB]
Irr_Atn_Dec_Calc          = 29.755[dB]
Irr_Atn_Dec_Default_Calc  = 29.755[dB]
Irr_Atn_Noe               = 29.755[dB]
Irr_Dec_Bandwidth_Hz      = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm     = 11.96453209[ppm]
Irr_Dec_Freq              = 399.73219668[MHz]
Irr_Dec_Merit_Factor      = 2.2
Irr_Decoupling            = TRUE
Irr_Noe                   = TRUE
Irr_Noise                 = WALTZ
Irr_Offset_Default        = 5[ppm]
Irr_Pwidth                = 0.115[ms]
Irr_Pwidth_Default        = 0.115[ms]
Irr_Pwidth_Default_Calc   = 0.115[ms]
Irr_Pwidth_Temp1          = 0.115[ms]
Irr_Wurst                 = FALSE
Decimation_Rate           = 0
Experiment_Path           = c:\Program Files\J
Initial_Wait              = 1[s]
Noe_Time                  = 2[s]
Noe_Time_Flag             = FALSE
Relaxation_Delay_Calc     = 0[s]
Relaxation_Delay_Temp     = 2[s]
Repetition_Time           = 2.74448896[s]
```

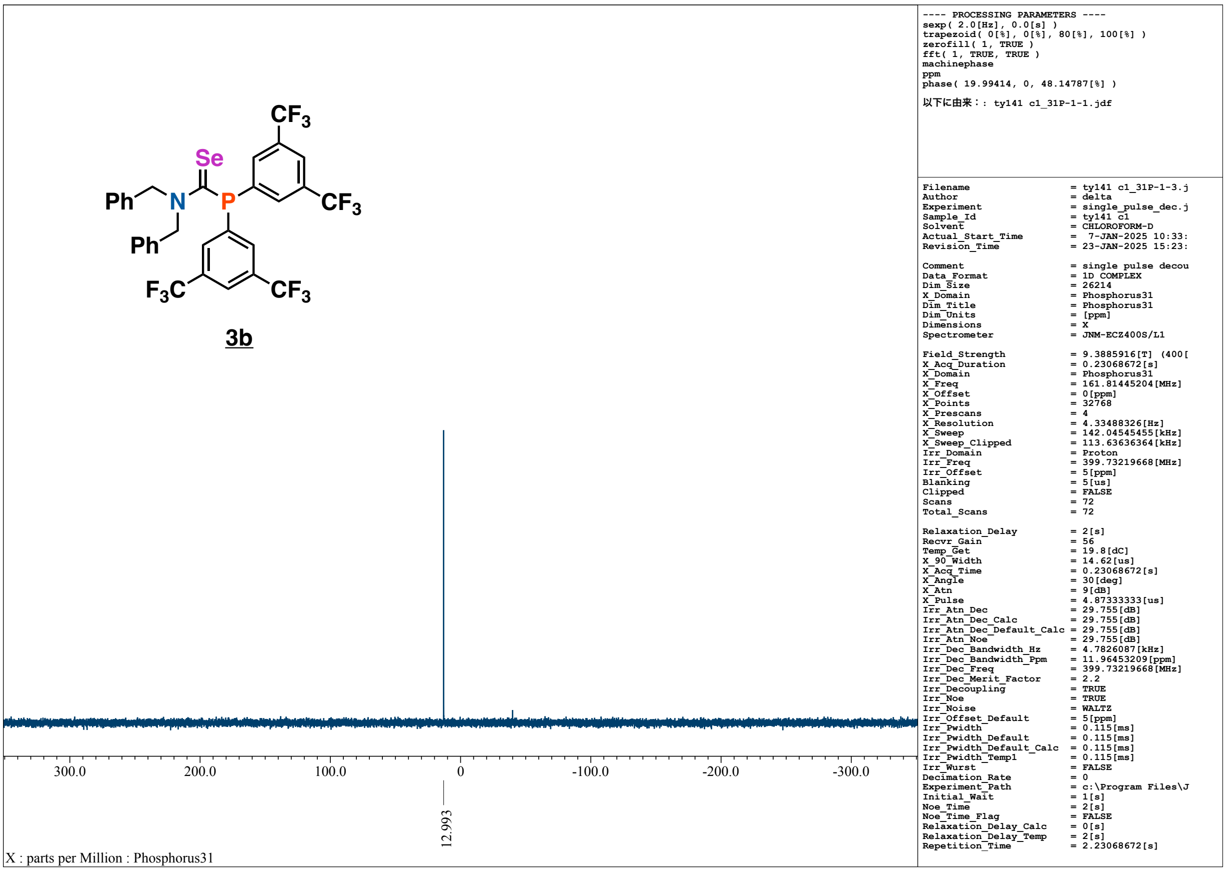
Figure S23. $^{13}C\{^{1}H\}$ NMR spectrum (100 MHz, $CDCl_3$) of **3b**.



```
---- PROCESSING PARAMETERS ----
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( -5.33968, -265.29802, 41.04452[%] )
thresh( 5.84874[%], 5.86277[%] )

以下に由来：: ty085 c1_19F-1-1.jdf
```

```
Filename               = ty085 c1_19F-1-3.jdf
Author                 = delta
Experiment             = single_pulse.jxp
Sample_Id              = ty085 c1
Solvent                = CHLOROFORM-D
Actual_Start_Time      = 10-DEC-2024 18:36:50
Revision_Time          = 23-JAN-2025 15:17:59

Comment                = single_pulse
Data_Format            = 1D COMPLEX
Dim_Size               = 26214
X_Domain               = Fluorine19
Dim_Title              = Fluorine19
Dim_Units              = [ppm]
Dimensions             = X
Spectrometer           = JNM-ECZ400S/L1

Field_Strength         = 9.3885916[T] (400[MHz
X_Acq_Duration         = 0.16777216[s]
X_Domain               = Fluorine19
X_Freq                 = 376.12400533[MHz]
X_Offset               = -100[ppm]
X_Points               = 32768
X_Prescans             = 1
X_Resolution           = 5.96046448[Hz]
X_Sweep                = 195.3125[kHz]
X_Sweep_Clipped        = 156.25[kHz]
Irr_Domain             = Fluorine19
Irr_Freq               = 376.12400533[MHz]
Irr_Offset             = 5[ppm]
Tri_Domain             = Fluorine19
Tri_Freq               = 376.12400533[MHz]
Tri_Offset             = 5[ppm]
Blanking               = 2[us]
Clipped                = FALSE
Scans                  = 8
Total_Scans            = 8

Relaxation_Delay       = 5[s]
Recvr_Gain             = 56
Temp_Get               = 20.1[dC]
X_90_Width             = 7.13[us]
X_Acq_Time             = 0.16777216[s]
X_Angle                = 45[deg]
X_Atn                  = 4.6[dB]
X_Pulse                = 3.565[us]
Irr_Mode               = Off
Tri_Mode               = Off
Dante_Loop             = 500
Dante_Presat           = FALSE
Decimation_Rate        = 0
Experiment_Path        = c:\Program Files\JEOL
Initial_Wait           = 1[s]
Phase                  = {0, 90, 270, 180, 180
Presat_Time            = 5[s]
Presat_Time_Flag       = FALSE
Relaxation_Delay_Calc  = 0[s]
Relaxation_Delay_Temp  = 5[s]
Repetition_Time        = 5.16777216[s]
```

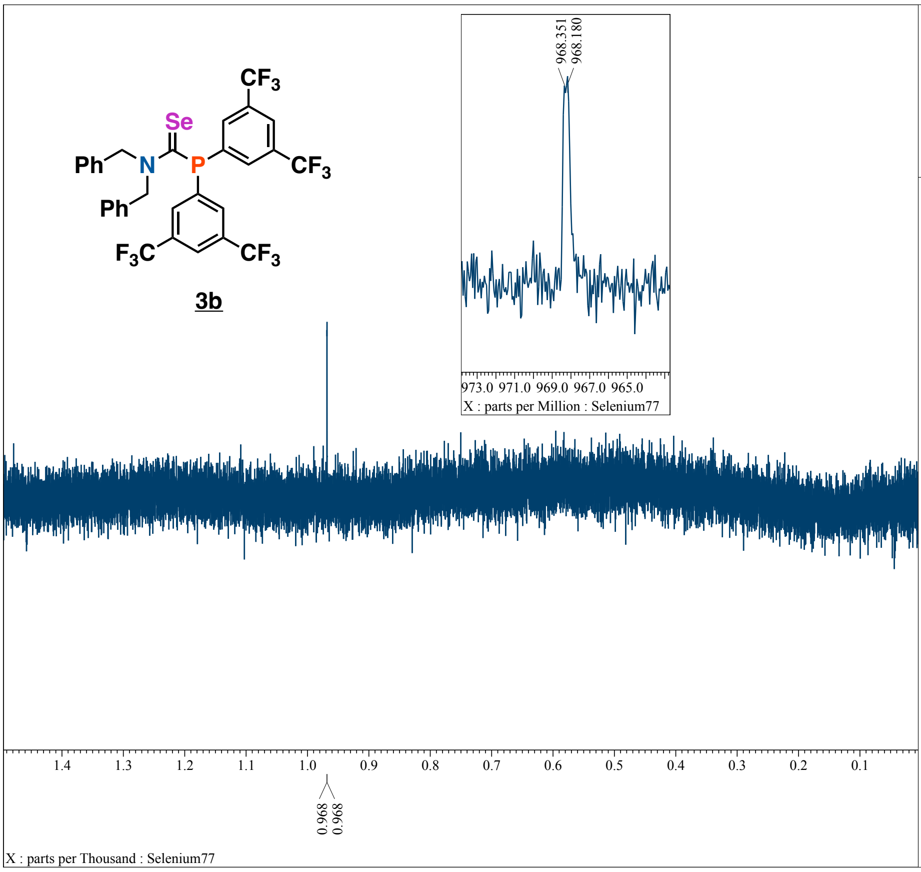
Figure S24. $^{19}F\{^{1}H\}$ NMR spectrum (376 MHz, $CDCl_3$) of **3b**.



```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( 19.99414, 0, 48.14787[%] )

以下に由来：: ty141 c1_31P-1-1.jdf
```

```
Filename                    = ty141 c1_31P-1-3.j
Author                      = delta
Experiment                  = single_pulse_dec.j
Sample_Id                   = ty141 c1
Solvent                     = CHLOROFORM-D
Actual_Start_Time           =  7-JAN-2025 10:33:
Revision_Time               = 23-JAN-2025 15:23:

Comment                     = single pulse decou
Data_Format                 = 1D COMPLEX
Dim_Size                    = 26214
X_Domain                    = Phosphorus31
Dim_Title                   = Phosphorus31
Dim_Units                   = [ppm]
Dimensions                  = X
Spectrometer                = JNM-ECZ400S/L1

Field_Strength              = 9.3885916[T] (400[
X_Acq_Duration              = 0.23068672[s]
X_Domain                    = Phosphorus31
X_Freq                      = 161.81445204[MHz]
X_Offset                    = 0[ppm]
X_Points                    = 32768
X_Prescans                  = 4
X_Resolution                = 4.33488326[Hz]
X_Sweep                     = 142.04545455[kHz]
X_Sweep_Clipped             = 113.63636364[kHz]
Irr_Domain                  = Proton
Irr_Freq                    = 399.73219668[MHz]
Irr_Offset                  = 5[ppm]
Blanking                    = 5[us]
Clipped                     = FALSE
Scans                       = 72
Total_Scans                 = 72

Relaxation_Delay            = 2[s]
Recvr_Gain                  = 56
Temp_Get                    = 19.8[dC]
X_90_Width                  = 14.62[us]
X_Acq_Time                  = 0.23068672[s]
X_Angle                     = 30[deg]
X_Atn                       = 9[dB]
X_Pulse                     = 4.87333333[us]
Irr_Atn_Dec                 = 29.755[dB]
Irr_Atn_Dec_Calc            = 29.755[dB]
Irr_Atn_Dec_Default_Calc    = 29.755[dB]
Irr_Atn_Noe                 = 29.755[dB]
Irr_Dec_Bandwidth_Hz        = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm       = 11.96453209[ppm]
Irr_Dec_Freq                = 399.73219668[MHz]
Irr_Dec_Merit_Factor        = 2.2
Irr_Decoupling              = TRUE
Irr_Noe                     = TRUE
Irr_Noise                   = WALTZ
Irr_Offset_Default          = 5[ppm]
Irr_Pwidth                  = 0.115[ms]
Irr_Pwidth_Default          = 0.115[ms]
Irr_Pwidth_Default_Calc     = 0.115[ms]
Irr_Pwidth_Templ            = 0.115[ms]
Irr_Wurst                   = FALSE
Decimation_Rate             = 0
Experiment_Path             = c:\Program Files\J
Initial_Wait                = 1[s]
Noe_Time                    = 2[s]
Noe_Time_Flag               = FALSE
Relaxation_Delay_Calc       = 0[s]
Relaxation_Delay_Temp       = 2[s]
Repetition_Time             = 2.23068672[s]
```

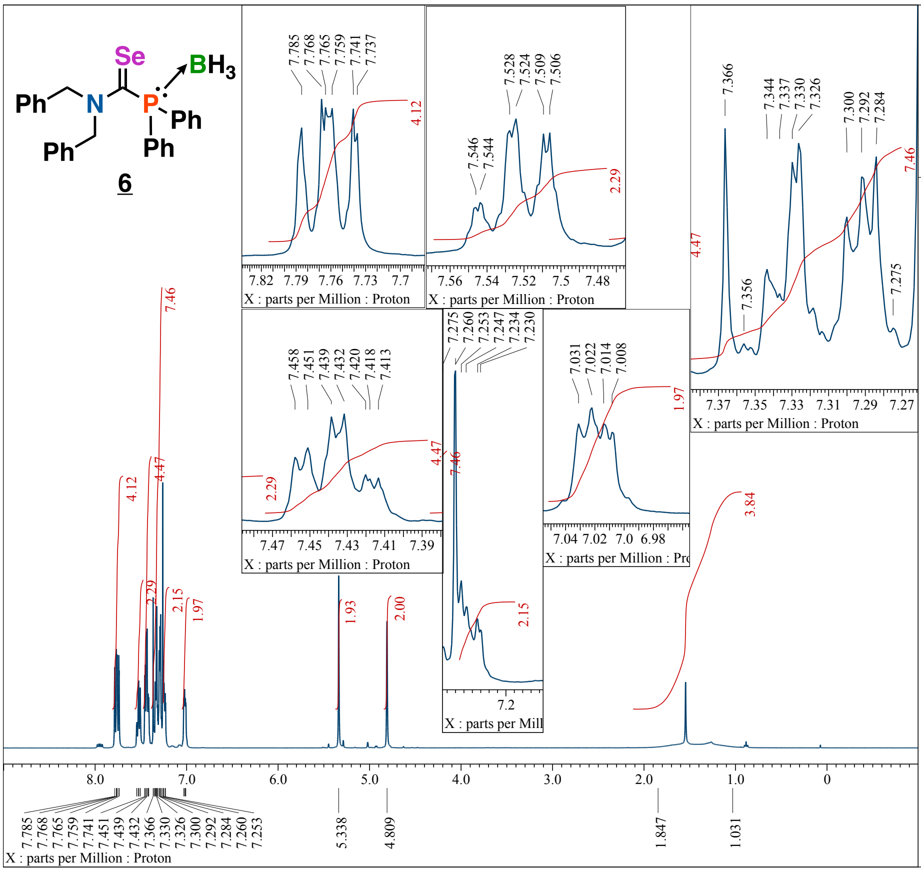
Figure S25. $^{31}P\{^{1}H\}$ NMR spectrum (126 MHz, $CDCl_3$) of **3b**.



```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( 122.98327, -542.3454, 68.01206[%] )
thresh( 53.26448[%], 54.66441[%] )

以下に由来：: ty141 c1_77Se-3-1.jdf
```

```
Filename                     = ty141 c1_77Se-3-3.
Author                       = delta
Experiment                   = single_pulse_dec.j
Sample_Id                    = ty141 c1
Solvent                      = CHLOROFORM-D
Actual_Start_Time            =  7-JAN-2025 17:01:
Revision_Time                = 24-JAN-2025 16:13:

Comment                      = single pulse decou
Data_Format                  = 1D COMPLEX
Dim_Size                     = 26214
X_Domain                     = Selenium77
Dim_Title                    = Selenium77
Dim_Units                    = [ppm]
Dimensions                   = X
Spectrometer                 = JNM-ECZ400S/L1

Field_Strength               = 9.3885916[T] (400[
X_Acq_Duration               = 0.23068672[s]
X_Domain                     = Selenium77
X_Freq                       = 76.23488685[MHz]
X_Offset                     = 750[ppm]
X_Points                     = 32768
X_Prescans                   = 4
X_Resolution                 = 4.33488326[Hz]
X_Sweep                      = 142.04545455[kHz]
X_Sweep_Clipped              = 113.63636364[kHz]
Irr_Domain                   = Proton
Irr_Freq                     = 399.73219668[MHz]
Irr_Offset                   = 5[ppm]
Blanking                     = 5[us]
Clipped                      = TRUE
Scans                        = 616
Total_Scans                  = 616

Relaxation_Delay             = 2[s]
Recvr_Gain                   = 56
Temp_Get                     = 20.3[dC]
X_90_Width                   = 10.98[us]
X_Acq_Time                   = 0.23068672[s]
X_Angle                      = 30[deg]
X_Atn                        = 8[dB]
X_Pulse                      = 3.66[us]
Irr_Atn_Dec                  = 29.755[dB]
Irr_Atn_Dec_Calc             = 29.755[dB]
Irr_Atn_Dec_Default_Calc = 29.755[dB]
Irr_Atn_Noe                  = 29.755[dB]
Irr_Dec_Bandwidth_Hz         = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm        = 11.96453209[ppm]
Irr_Dec_Freq                 = 399.73219668[MHz]
Irr_Dec_Merit_Factor         = 2.2
Irr_Decoupling               = TRUE
Irr_Noe                      = TRUE
Irr_Noise                    = WALTZ
Irr_Offset_Default           = 5[ppm]
Irr_Pwidth                   = 0.115[ms]
Irr_Pwidth_Default           = 0.115[ms]
Irr_Pwidth_Default_Calc      = 0.115[ms]
Irr_Pwidth_Temp1             = 0.115[ms]
Irr_Wurst                    = FALSE
Decimation_Rate              = 0
Experiment_Path              = c:\Program Files\J
Initial_Wait                 = 1[s]
Noe_Time                     = 2[s]
Noe_Time_Flag                = FALSE
Relaxation_Delay_Calc        = 0[s]
Relaxation_Delay_Temp        = 2[s]
Repetition_Time              = 2.23068672[s]
```

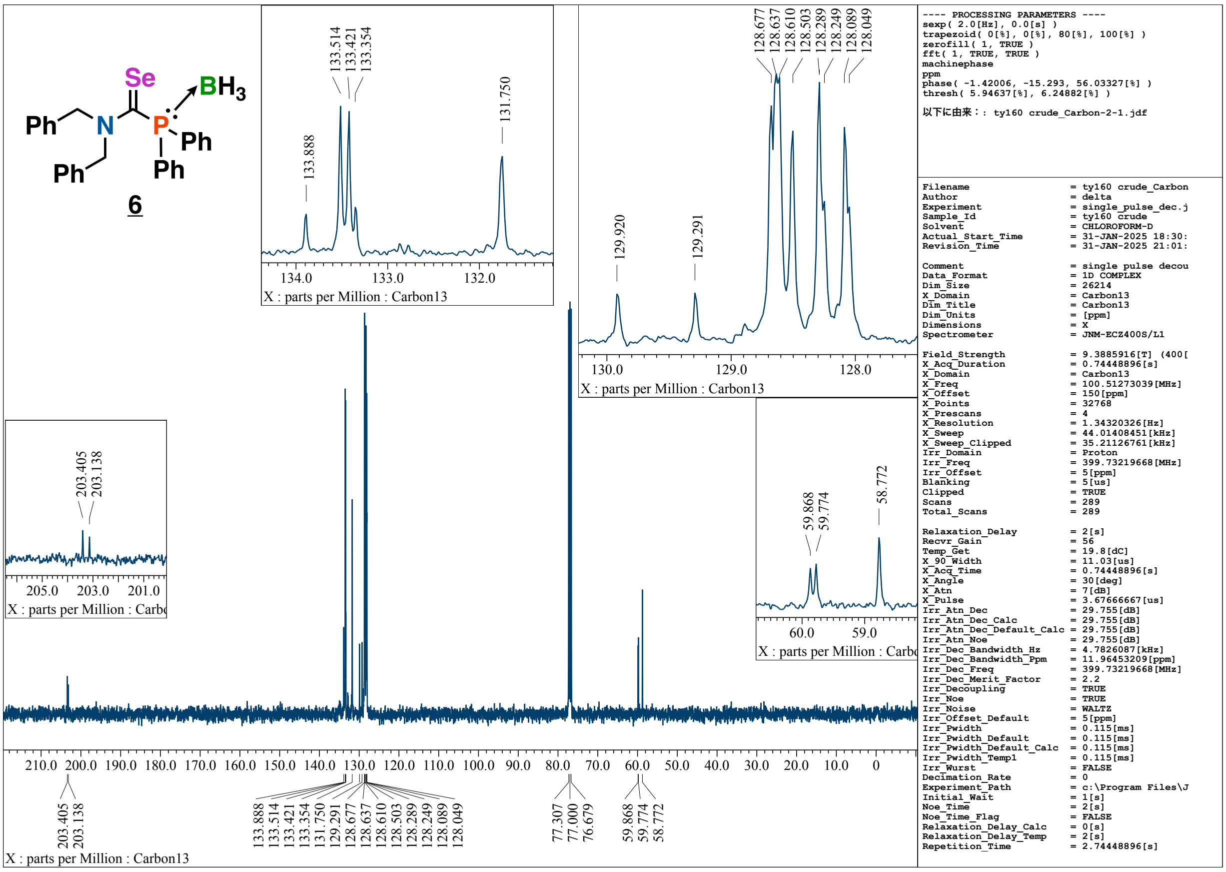
Figure S26. ^{77}Se{^{1}H} NMR spectrum (76 MHz, CDCl$_3$) of **3b**.



```
---- PROCESSING PARAMETERS ----
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( 1.15818, 0.28824, 34.78559[%] )
phase( 0, 0, 50[%] )

以下に由来：: ty160 crude_Proton-2-1.jdf
```

```
Filename                 = ty160 crude_Proton-2-
Author                   = delta
Experiment               = single_pulse.jxp
Sample_Id                = ty160 crude
Solvent                  = CHLOROFORM-D
Actual_Start_Time        = 29-JAN-2025 21:01:52
Revision_Time            = 30-JAN-2025 12:03:32

Comment                  = single_pulse
Data_Format              = 1D COMPLEX
Dim_Size                 = 13107
X_Domain                 = Proton
Dim_Title                = Proton
Dim_Units                = [ppm]
Dimensions               = X
Spectrometer             = JNM-ECZ400S/L1

Field_Strength           = 9.3885916[T] (400[MHz
X_Acq_Duration           = 2.18628096[s]
X_Domain                 = Proton
X_Freq                   = 399.73219668[MHz]
X_Offset                 = 5[ppm]
X_Points                 = 16384
X_Prescans               = 1
X_Resolution             = 0.45739775[Hz]
X_Sweep                  = 7.4940048[kHz]
X_Sweep_Clipped          = 5.99520384[kHz]
Irr_Domain               = Proton
Irr_Freq                 = 399.73219668[MHz]
Irr_Offset               = 5[ppm]
Tri_Domain               = Proton
Tri_Freq                 = 399.73219668[MHz]
Tri_Offset               = 5[ppm]
Blanking                 = 2[us]
Clipped                  = FALSE
Scans                    = 8
Total_Scans              = 8

Relaxation_Delay         = 5[s]
Recvr_Gain               = 56
Temp_Get                 = 20.1[dC]
X_90_Width               = 6.28[us]
X_Acq_Time               = 2.18628096[s]
X_Angle                  = 45[deg]
X_Atn                    = 4.5[dB]
X_Pulse                  = 3.14[us]
Irr_Mode                 = Off
Tri_Mode                 = Off
Dante_Loop               = 500
Dante_Presat             = FALSE
Decimation_Rate          = 0
Experiment_Path          = c:\Program Files\JEOL
Initial_Wait             = 1[s]
Phase                    = {0, 90, 270, 180, 180
Presat_Time              = 5[s]
Presat_Time_Flag         = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 5[s]
Repetition_Time          = 7.18628096[s]
```

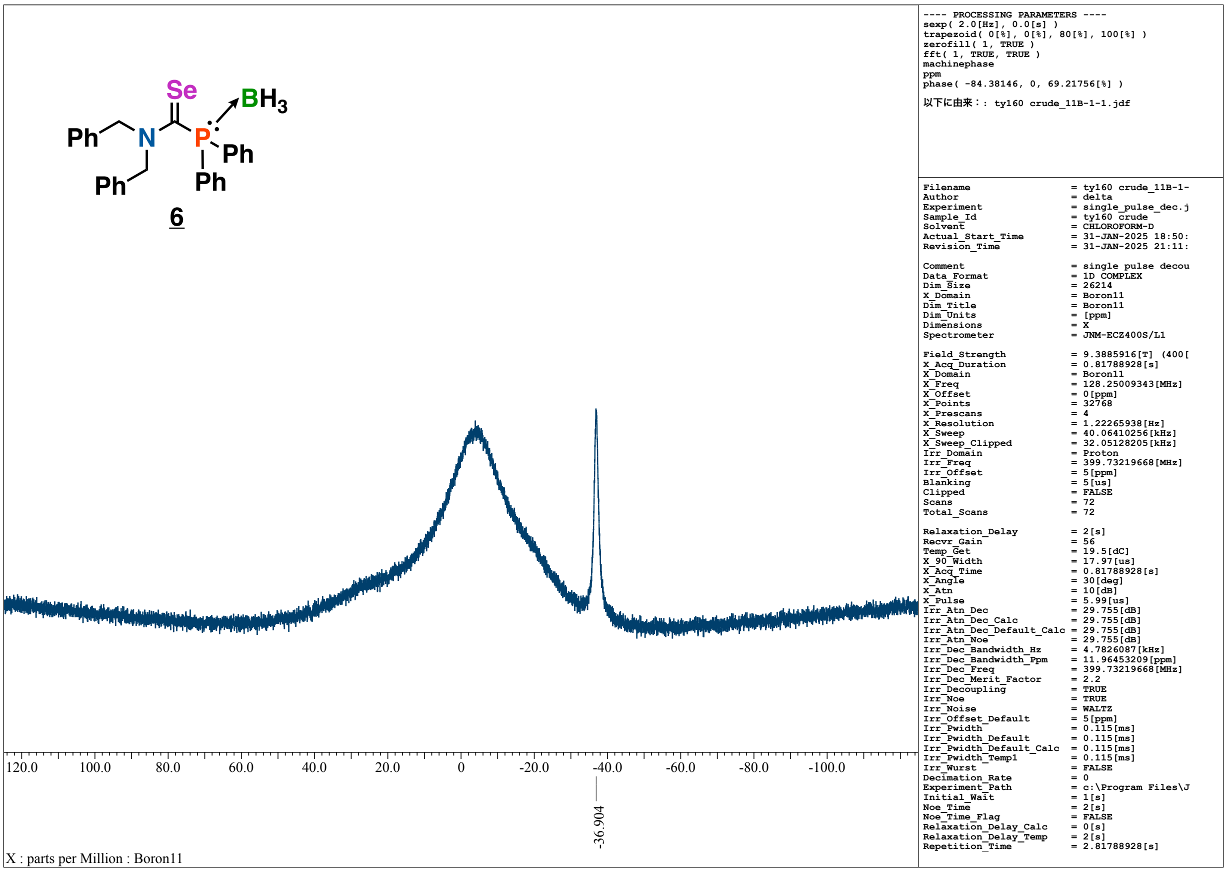
Figure S27. ^{1}H NMR spectrum (400 MHz, CDCl$_3$) of **6**.



```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( -1.42006, -15.293, 56.03327[%] )
thresh( 5.94637[%], 6.24882[%] )

以下に由来：: ty160 crude_Carbon-2-1.jdf
```

```
Filename                   = ty160 crude_Carbon
Author                     = delta
Experiment                 = single_pulse_dec.j
Sample_Id                  = ty160 crude
Solvent                    = CHLOROFORM-D
Actual_Start_Time          = 31-JAN-2025 18:30:
Revision_Time              = 31-JAN-2025 21:01:

Comment                    = single pulse decou
Data_Format                = 1D COMPLEX
Dim_Size                   = 26214
X_Domain                   = Carbon13
Dim_Title                  = Carbon13
Dim_Units                  = [ppm]
Dimensions                 = X
Spectrometer               = JNM-ECZ400S/L1

Field_Strength             = 9.3885916[T] (400[
X_Acq_Duration             = 0.74448896[s]
X_Domain                   = Carbon13
X_Freq                     = 100.51273039[MHz]
X_Offset                   = 150[ppm]
X_Points                   = 32768
X_Prescans                 = 4
X_Resolution               = 1.34320326[Hz]
X_Sweep                    = 44.01408451[kHz]
X_Sweep_Clipped            = 35.21126761[kHz]
Irr_Domain                 = Proton
Irr_Freq                   = 399.73219668[MHz]
Irr_Offset                 = 5[ppm]
Blanking                   = 5[us]
Clipped                    = TRUE
Scans                      = 289
Total_Scans                = 289

Relaxation_Delay           = 2[s]
Recvr_Gain                 = 56
Temp_Get                   = 19.8[dC]
X_90_Width                 = 11.03[us]
X_Acq_Time                 = 0.74448896[s]
X_Angle                    = 30[deg]
X_Atn                      = 7[dB]
X_Pulse                    = 3.67666667[us]
Irr_Atn_Dec                = 29.755[dB]
Irr_Atn_Dec_Calc           = 29.755[dB]
Irr_Atn_Dec_Default_Calc = 29.755[dB]
Irr_Atn_Noe                = 29.755[dB]
Irr_Dec_Bandwidth_Hz       = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm      = 11.96453209[ppm]
Irr_Dec_Freq               = 399.73219668[MHz]
Irr_Dec_Merit_Factor       = 2.2
Irr_Decoupling             = TRUE
Irr_Noe                    = TRUE
Irr_Noise                  = WALTZ
Irr_Offset_Default         = 5[ppm]
Irr_Pwidth                 = 0.115[ms]
Irr_Pwidth_Default         = 0.115[ms]
Irr_Pwidth_Default_Calc    = 0.115[ms]
Irr_Pwidth_Temp1           = 0.115[ms]
Irr_Wurst                  = FALSE
Decimation_Rate            = 0
Experiment_Path            = c:\Program Files\J
Initial_Wait               = 1[s]
Noe_Time                   = 2[s]
Noe_Time_Flag              = FALSE
Relaxation_Delay_Calc      = 0[s]
Relaxation_Delay_Temp      = 2[s]
Repetition_Time            = 2.74448896[s]
```

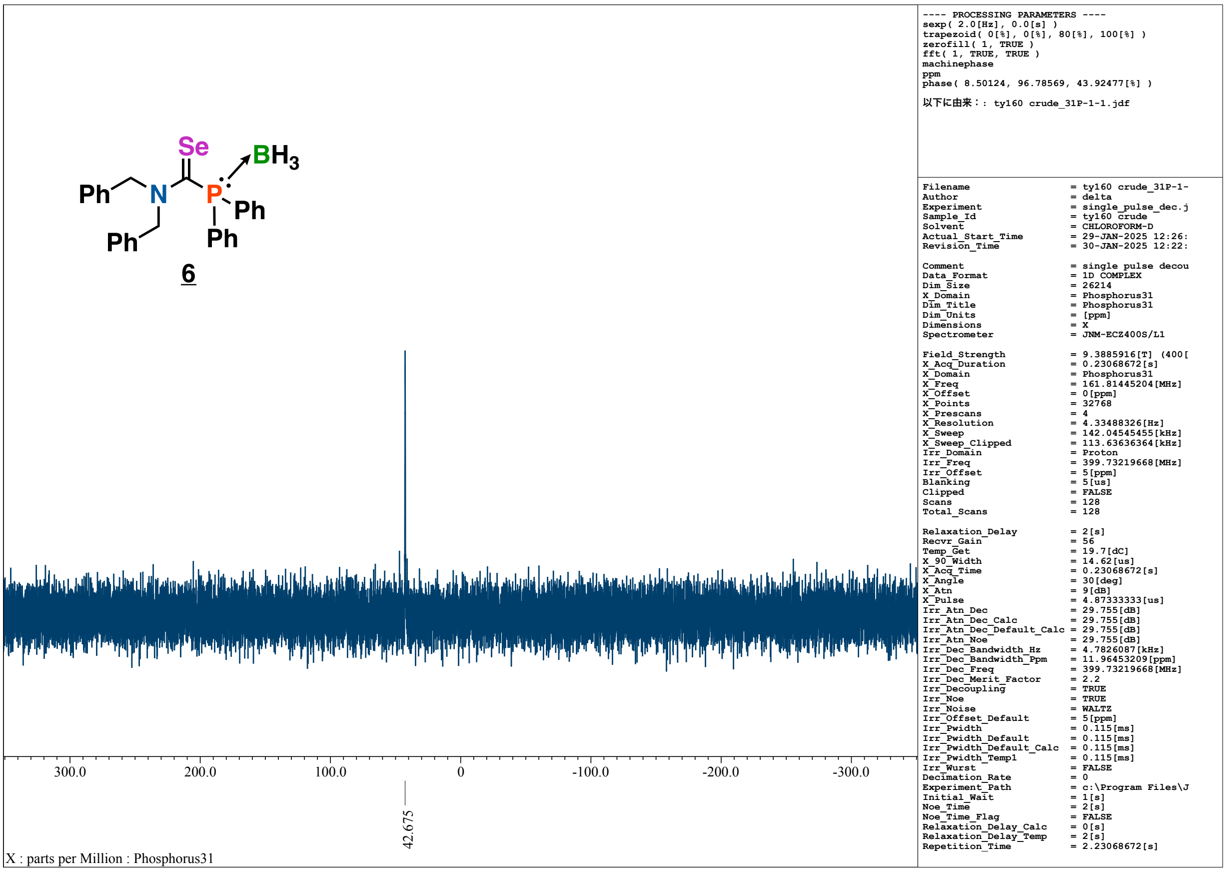
Figure S28. $^{13}C\{^1H\}$ NMR spectrum (100 MHz, $CDCl_3$) of **6**.



```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( -84.38146, 0, 69.21756[%] )

以下に由来：: ty160 crude_11B-1-1.jdf
```

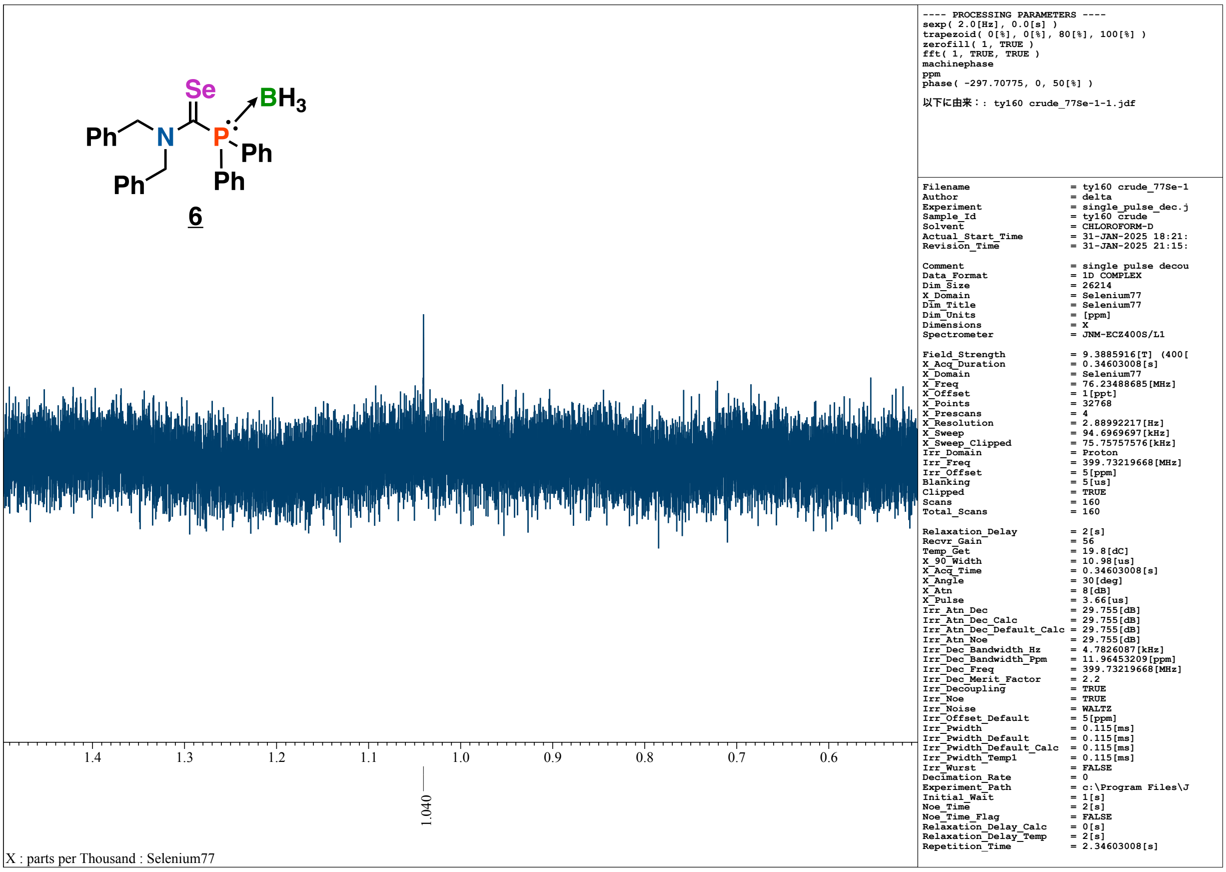
```
Filename                   = ty160 crude_11B-1-
Author                     = delta
Experiment                 = single_pulse_dec.j
Sample_Id                  = ty160 crude
Solvent                    = CHLOROFORM-D
Actual_Start_Time          = 31-JAN-2025 18:50:
Revision_Time              = 31-JAN-2025 21:11:

Comment                    = single pulse decou
Data_Format                = 1D COMPLEX
Dim_Size                   = 26214
X_Domain                   = Boron11
Dim_Title                  = Boron11
Dim_Units                  = [ppm]
Dimensions                 = X
Spectrometer               = JNM-ECZ400S/L1

Field_Strength             = 9.3885916[T] (400[
X_Acq_Duration             = 0.81788928[s]
X_Domain                   = Boron11
X_Freq                     = 128.25009343[MHz]
X_Offset                   = 0[ppm]
X_Points                   = 32768
X_Prescans                 = 4
X_Resolution               = 1.22265938[Hz]
X_Sweep                    = 40.06410256[kHz]
X_Sweep_Clipped            = 32.05128205[kHz]
Irr_Domain                 = Proton
Irr_Freq                   = 399.73219668[MHz]
Irr_Offset                 = 5[ppm]
Blanking                   = 5[us]
Clipped                    = FALSE
Scans                      = 72
Total_Scans                = 72

Relaxation_Delay           = 2[s]
Recvr_Gain                 = 56
Temp_Get                   = 19.5[dC]
X_90_Width                 = 17.97[us]
X_Acq_Time                 = 0.81788928[s]
X_Angle                    = 30[deg]
X_Atn                      = 10[dB]
X_Pulse                    = 5.99[us]
Irr_Atn_Dec                = 29.755[dB]
Irr_Atn_Dec_Calc           = 29.755[dB]
Irr_Atn_Dec_Default_Calc = 29.755[dB]
Irr_Atn_Noe                = 29.755[dB]
Irr_Dec_Bandwidth_Hz       = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm      = 11.96453209[ppm]
Irr_Dec_Freq               = 399.73219668[MHz]
Irr_Dec_Merit_Factor       = 2.2
Irr_Decoupling             = TRUE
Irr_Noe                    = TRUE
Irr_Noise                  = WALTZ
Irr_Offset_Default         = 5[ppm]
Irr_Pwidth                 = 0.115[ms]
Irr_Pwidth_Default         = 0.115[ms]
Irr_Pwidth_Default_Calc    = 0.115[ms]
Irr_Pwidth_Temp1           = 0.115[ms]
Irr_Wurst                  = FALSE
Decimation_Rate            = 0
Experiment_Path            = c:\Program Files\J
Initial_Wait               = 1[s]
Noe_Time                   = 2[s]
Noe_Time_Flag              = FALSE
Relaxation_Delay_Calc      = 0[s]
Relaxation_Delay_Temp      = 2[s]
Repetition_Time            = 2.81788928[s]
```

Figure S29. $^{11}B\{^1H\}$ NMR spectrum (128 MHz, $CDCl_3$) of **6**.



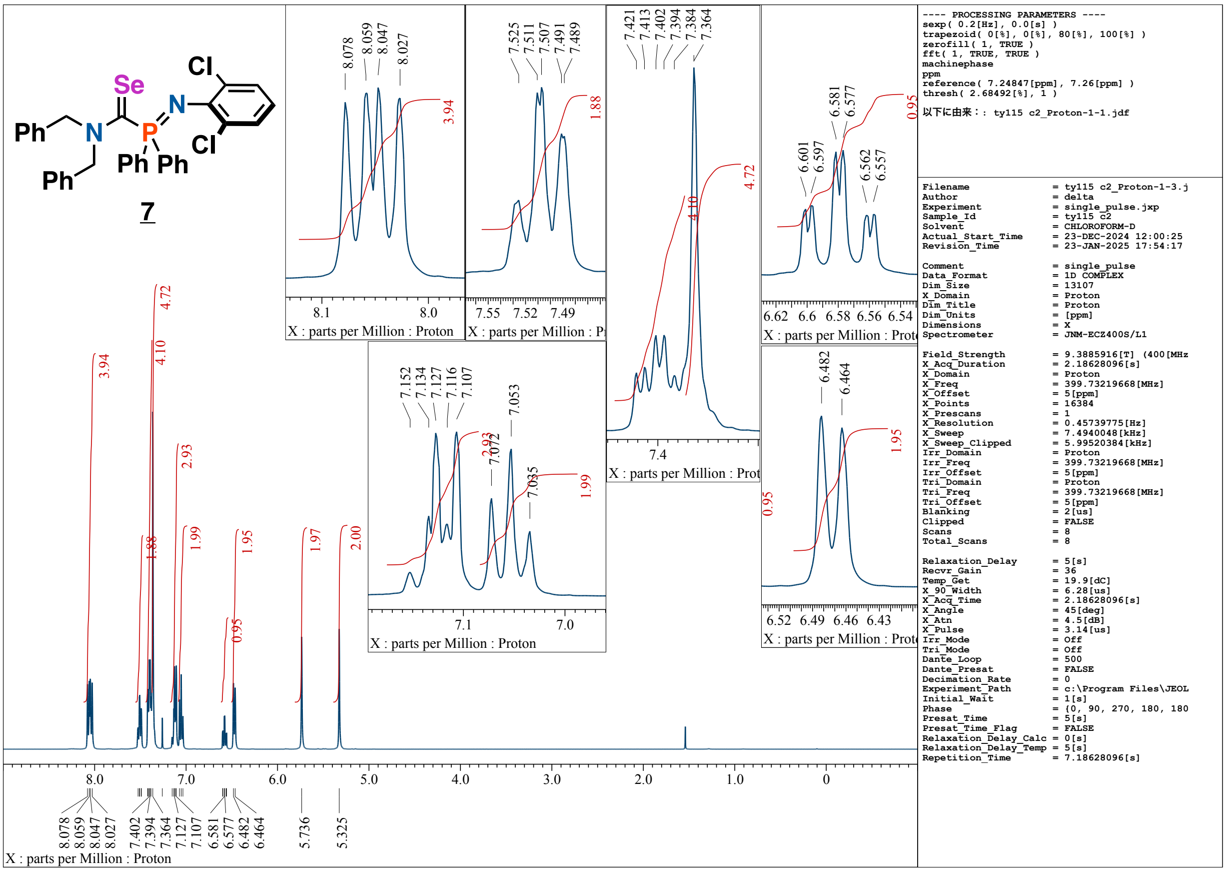
```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( 8.50124, 96.78569, 43.92477[%] )

以下に由来：: ty160 crude_31P-1-1.jdf

Filename                    = ty160 crude_31P-1-
Author                      = delta
Experiment                  = single_pulse_dec.j
Sample_Id                   = ty160 crude
Solvent                     = CHLOROFORM-D
Actual_Start_Time           = 29-JAN-2025 12:26:
Revision_Time               = 30-JAN-2025 12:22:

Comment                     = single pulse decou
Data_Format                 = 1D COMPLEX
Dim_Size                    = 26214
X_Domain                    = Phosphorus31
Dim_Title                   = Phosphorus31
Dim_Units                   = [ppm]
Dimensions                  = X
Spectrometer                = JNM-ECZ400S/L1

Field_Strength              = 9.3885916[T] (400[
X_Acq_Duration              = 0.23068672[s]
X_Domain                    = Phosphorus31
X_Freq                      = 161.81445204[MHz]
X_Offset                    = 0[ppm]
X_Points                    = 32768
X_Prescans                  = 4
X_Resolution                = 4.33488326[Hz]
X_Sweep                     = 142.04545455[kHz]
X_Sweep_Clipped             = 113.63636364[kHz]
Irr_Domain                  = Proton
Irr_Freq                    = 399.73219668[MHz]
Irr_Offset                  = 5[ppm]
Blanking                    = 5[us]
Clipped                     = FALSE
Scans                       = 128
Total_Scans                 = 128

Relaxation_Delay            = 2[s]
Recvr_Gain                  = 56
Temp_Get                    = 19.7[dC]
X_90_Width                  = 14.62[us]
X_Acq_Time                  = 0.23068672[s]
X_Angle                     = 30[deg]
X_Atn                       = 9[dB]
X_Pulse                     = 4.87333333[us]
Irr_Atn_Dec                 = 29.755[dB]
Irr_Atn_Dec_Calc            = 29.755[dB]
Irr_Atn_Dec_Default_Calc    = 29.755[dB]
Irr_Atn_Noe                 = 29.755[dB]
Irr_Dec_Bandwidth_Hz        = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm       = 11.96453209[ppm]
Irr_Dec_Freq                = 399.73219668[MHz]
Irr_Dec_Merit_Factor        = 2.2
Irr_Decoupling              = TRUE
Irr_Noe                     = TRUE
Irr_Noise                   = WALTZ
Irr_Offset_Default          = 5[ppm]
Irr_Pwidth                  = 0.115[ms]
Irr_Pwidth_Default          = 0.115[ms]
Irr_Pwidth_Default_Calc     = 0.115[ms]
Irr_Pwidth_Temp1            = 0.115[ms]
Irr_Wurst                   = FALSE
Decimation_Rate             = 0
Experiment_Path             = c:\Program Files\J
Initial_Wait                = 1[s]
Noe_Time                    = 2[s]
Noe_Time_Flag               = FALSE
Relaxation_Delay_Calc       = 0[s]
Relaxation_Delay_Temp       = 2[s]
Repetition_Time             = 2.23068672[s]
```

Figure S30. $^{31}P\{^1H\}$ NMR spectrum (126 MHz, $CDCl_3$) of **6**.



```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( -297.70775, 0, 50[%] )

以下に由来：: ty160 crude_77Se-1-1.jdf

Filename                    = ty160 crude_77Se-1
Author                      = delta
Experiment                  = single_pulse_dec.j
Sample_Id                   = ty160 crude
Solvent                     = CHLOROFORM-D
Actual_Start_Time           = 31-JAN-2025 18:21:
Revision_Time               = 31-JAN-2025 21:15:

Comment                     = single pulse decou
Data_Format                 = 1D COMPLEX
Dim_Size                    = 26214
X_Domain                    = Selenium77
Dim_Title                   = Selenium77
Dim_Units                   = [ppm]
Dimensions                  = X
Spectrometer                = JNM-ECZ400S/L1

Field_Strength              = 9.3885916[T] (400[
X_Acq_Duration              = 0.34603008[s]
X_Domain                    = Selenium77
X_Freq                      = 76.23488685[MHz]
X_Offset                    = 1[ppt]
X_Points                    = 32768
X_Prescans                  = 4
X_Resolution                = 2.88992217[Hz]
X_Sweep                     = 94.6969697[kHz]
X_Sweep_Clipped             = 75.75757576[kHz]
Irr_Domain                  = Proton
Irr_Freq                    = 399.73219668[MHz]
Irr_Offset                  = 5[ppm]
Blanking                    = 5[us]
Clipped                     = TRUE
Scans                       = 160
Total_Scans                 = 160

Relaxation_Delay            = 2[s]
Recvr_Gain                  = 56
Temp_Get                    = 19.8[dC]
X_90_Width                  = 10.98[us]
X_Acq_Time                  = 0.34603008[s]
X_Angle                     = 30[deg]
X_Atn                       = 8[dB]
X_Pulse                     = 3.66[us]
Irr_Atn_Dec                 = 29.755[dB]
Irr_Atn_Dec_Calc            = 29.755[dB]
Irr_Atn_Dec_Default_Calc    = 29.755[dB]
Irr_Atn_Noe                 = 29.755[dB]
Irr_Dec_Bandwidth_Hz        = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm       = 11.96453209[ppm]
Irr_Dec_Freq                = 399.73219668[MHz]
Irr_Dec_Merit_Factor        = 2.2
Irr_Decoupling              = TRUE
Irr_Noe                     = TRUE
Irr_Noise                   = WALTZ
Irr_Offset_Default          = 5[ppm]
Irr_Pwidth                  = 0.115[ms]
Irr_Pwidth_Default          = 0.115[ms]
Irr_Pwidth_Default_Calc     = 0.115[ms]
Irr_Pwidth_Temp1            = 0.115[ms]
Irr_Wurst                   = FALSE
Decimation_Rate             = 0
Experiment_Path             = c:\Program Files\J
Initial_Wait                = 1[s]
Noe_Time                    = 2[s]
Noe_Time_Flag               = FALSE
Relaxation_Delay_Calc       = 0[s]
Relaxation_Delay_Temp       = 2[s]
Repetition_Time             = 2.34603008[s]
```

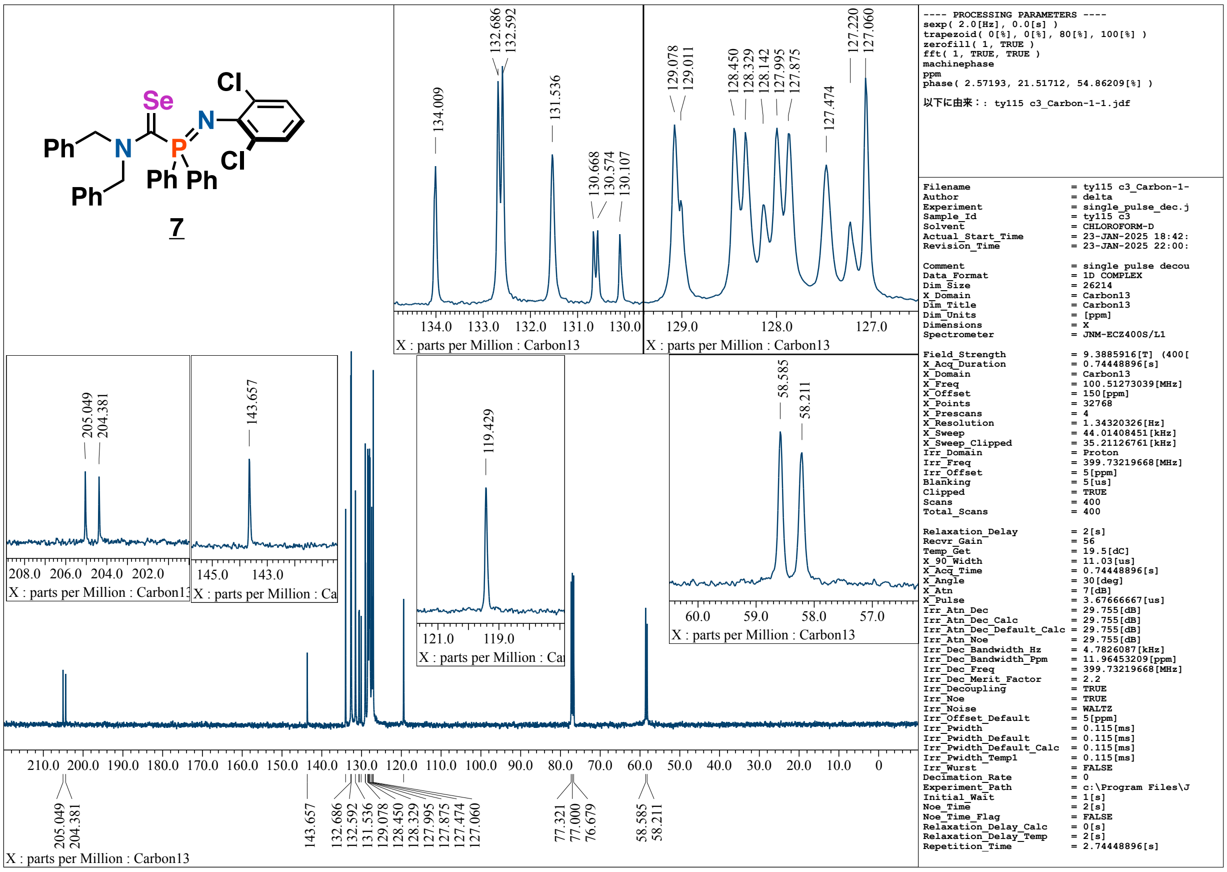
Figure S31. $^{77}Se\{^1H\}$ NMR spectrum (76 MHz, $CDCl_3$) of **6**.



```
---- PROCESSING PARAMETERS ----
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( 7.24847[ppm], 7.26[ppm] )
thresh( 2.68492[%], 1 )

以下に由来：: ty115 c2_Proton-1-1.jdf
```

```
Filename                 = ty115 c2_Proton-1-3.j
Author                   = delta
Experiment               = single_pulse.jxp
Sample_Id                = ty115 c2
Solvent                  = CHLOROFORM-D
Actual_Start_Time        = 23-DEC-2024 12:00:25
Revision_Time            = 23-JAN-2025 17:54:17

Comment                  = single_pulse
Data_Format              = 1D COMPLEX
Dim_Size                 = 13107
X_Domain                 = Proton
Dim_Title                = Proton
Dim_Units                = [ppm]
Dimensions               = X
Spectrometer             = JNM-ECZ400S/L1

Field_Strength           = 9.3885916[T] (400[MHz
X_Acq_Duration           = 2.18628096[s]
X_Domain                 = Proton
X_Freq                   = 399.73219668[MHz]
X_Offset                 = 5[ppm]
X_Points                 = 16384
X_Prescans               = 1
X_Resolution             = 0.45739775[Hz]
X_Sweep                  = 7.4940048[kHz]
X_Sweep_Clipped          = 5.99520384[kHz]
Irr_Domain               = Proton
Irr_Freq                 = 399.73219668[MHz]
Irr_Offset               = 5[ppm]
Tri_Domain               = Proton
Tri_Freq                 = 399.73219668[MHz]
Tri_Offset               = 5[ppm]
Blanking                 = 2[us]
Clipped                  = FALSE
Scans                    = 8
Total_Scans              = 8

Relaxation_Delay         = 5[s]
Recvr_Gain               = 36
Temp_Get                 = 19.9[dC]
X_90_Width               = 6.28[us]
X_Acq_Time               = 2.18628096[s]
X_Angle                  = 45[deg]
X_Atn                    = 4.5[dB]
X_Pulse                  = 3.14[us]
Irr_Mode                 = Off
Tri_Mode                 = Off
Dante_Loop               = 500
Dante_Presat             = FALSE
Decimation_Rate          = 0
Experiment_Path          = c:\Program Files\JEOL
Initial_Wait             = 1[s]
Phase                    = {0, 90, 270, 180, 180
Presat_Time              = 5[s]
Presat_Time_Flag         = FALSE
Relaxation_Delay_Calc    = 0[s]
Relaxation_Delay_Temp    = 5[s]
Repetition_Time          = 7.18628096[s]
```

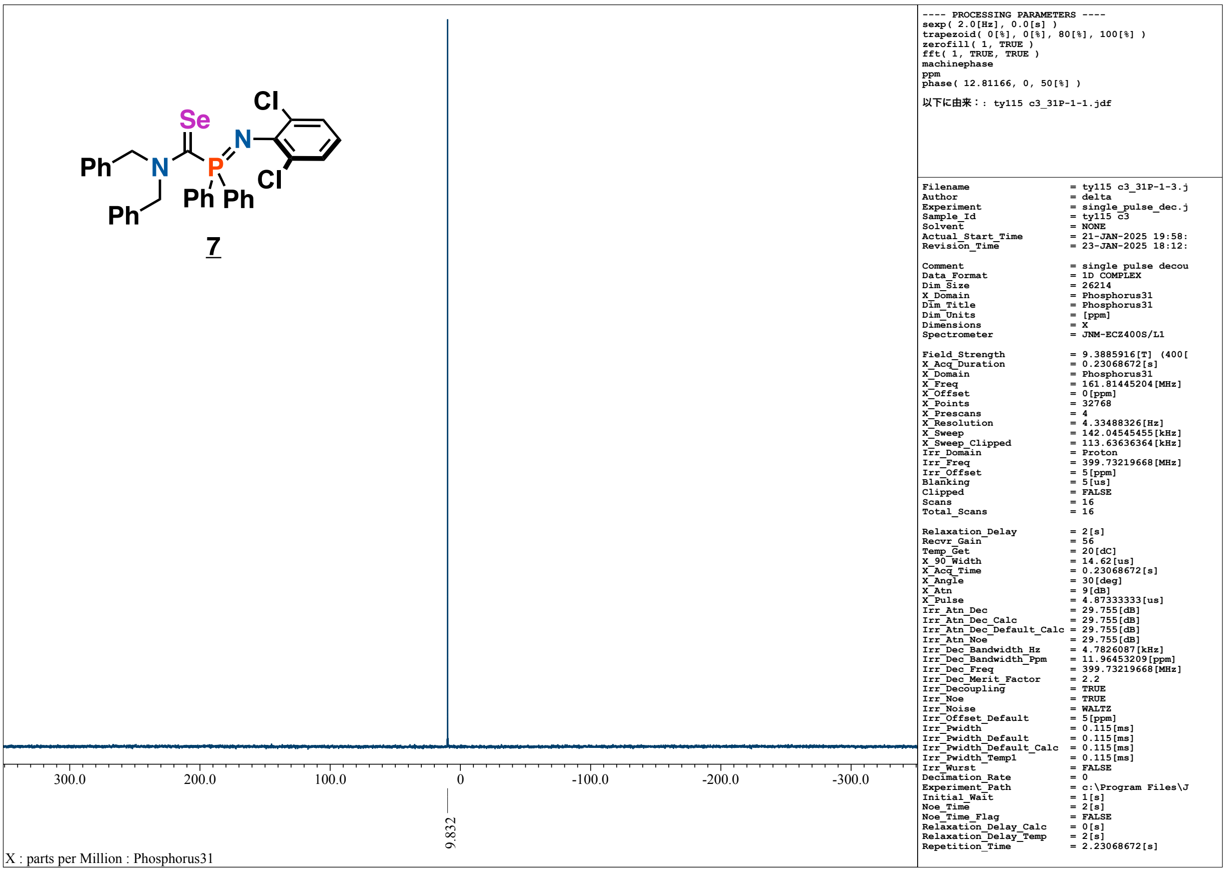
Figure S32. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **7**.



```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( 2.57193, 21.51712, 54.86209[%] )

以下に由来：: ty115 c3_Carbon-1-1.jdf
```

```
Filename                     = ty115 c3_Carbon-1-
Author                       = delta
Experiment                   = single_pulse_dec.j
Sample_Id                    = ty115 c3
Solvent                      = CHLOROFORM-D
Actual_Start_Time            = 23-JAN-2025 18:42:
Revision_Time                = 23-JAN-2025 22:00:

Comment                      = single pulse decou
Data_Format                  = 1D COMPLEX
Dim_Size                     = 26214
X_Domain                     = Carbon13
Dim_Title                    = Carbon13
Dim_Units                    = [ppm]
Dimensions                   = X
Spectrometer                 = JNM-ECZ400S/L1

Field_Strength               = 9.3885916[T] (400[
X_Acq_Duration               = 0.74448896[s]
X_Domain                     = Carbon13
X_Freq                       = 100.51273039[MHz]
X_Offset                     = 150[ppm]
X_Points                     = 32768
X_Prescans                   = 4
X_Resolution                 = 1.34320326[Hz]
X_Sweep                      = 44.01408451[kHz]
X_Sweep_Clipped              = 35.21126761[kHz]
Irr_Domain                   = Proton
Irr_Freq                     = 399.73219668[MHz]
Irr_Offset                   = 5[ppm]
Blanking                     = 5[us]
Clipped                      = TRUE
Scans                        = 400
Total_Scans                  = 400

Relaxation_Delay             = 2[s]
Recvr_Gain                   = 56
Temp_Get                     = 19.5[dC]
X_90_Width                   = 11.03[us]
X_Acq_Time                   = 0.74448896[s]
X_Angle                      = 30[deg]
X_Atn                        = 7[dB]
X_Pulse                      = 3.67666667[us]
Irr_Atn_Dec                  = 29.755[dB]
Irr_Atn_Dec_Calc             = 29.755[dB]
Irr_Atn_Dec_Default_Calc     = 29.755[dB]
Irr_Atn_Noe                  = 29.755[dB]
Irr_Dec_Bandwidth_Hz         = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm        = 11.96453209[ppm]
Irr_Dec_Freq                 = 399.73219668[MHz]
Irr_Dec_Merit_Factor         = 2.2
Irr_Decoupling               = TRUE
Irr_Noe                      = TRUE
Irr_Noise                    = WALTZ
Irr_Offset_Default           = 5[ppm]
Irr_Pwidth                   = 0.115[ms]
Irr_Pwidth_Default           = 0.115[ms]
Irr_Pwidth_Default_Calc      = 0.115[ms]
Irr_Pwidth_Temp1             = 0.115[ms]
Irr_Wurst                    = FALSE
Decimation_Rate              = 0
Experiment_Path              = c:\Program Files\J
Initial_Wait                 = 1[s]
Noe_Time                     = 2[s]
Noe_Time_Flag                = FALSE
Relaxation_Delay_Calc        = 0[s]
Relaxation_Delay_Temp        = 2[s]
Repetition_Time              = 2.74448896[s]
```

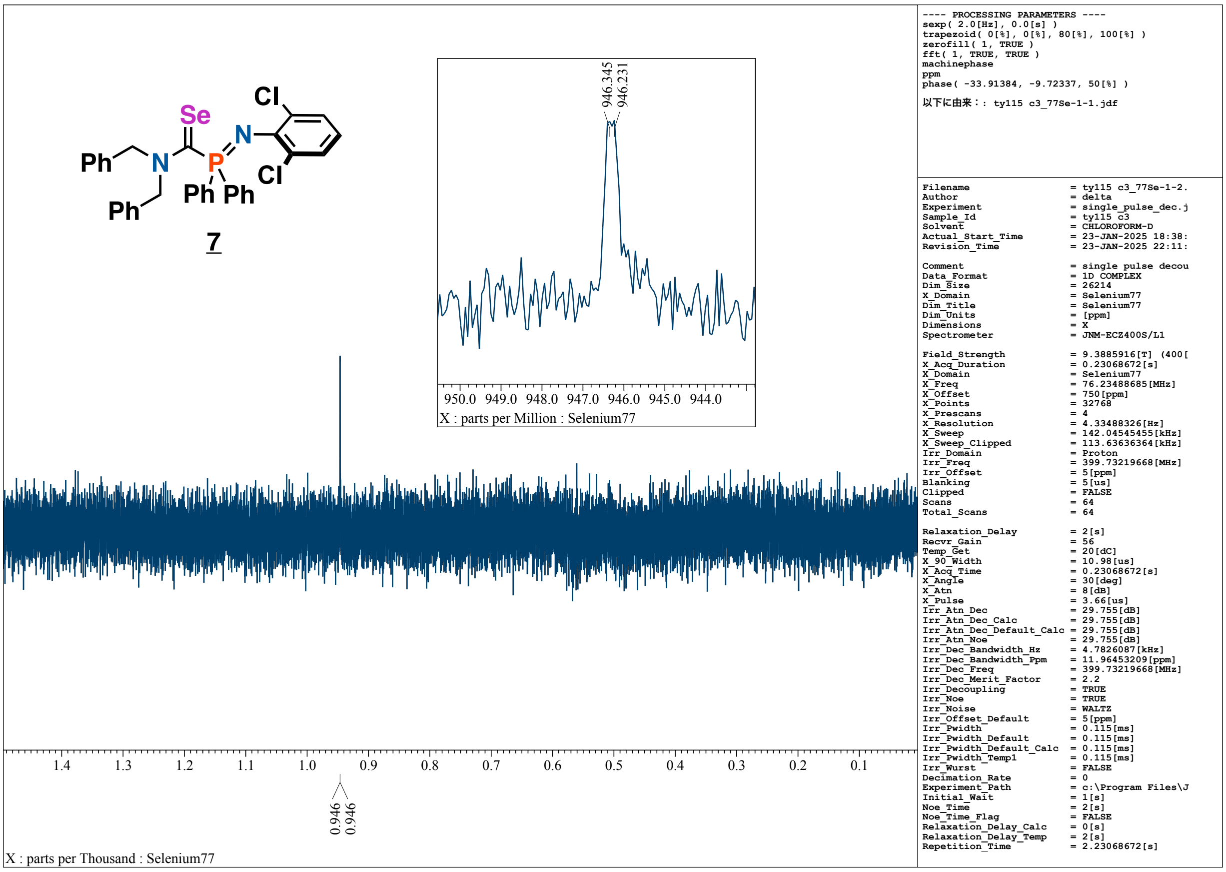
Figure S33. $^{13}C\{^{1}H\}$ NMR spectrum (100 MHz, $CDCl_3$) of **7**.



```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( 12.81166, 0, 50[%] )

以下に由来：: ty115 c3_31P-1-1.jdf
```

```
Filename                   = ty115 c3_31P-1-3.j
Author                     = delta
Experiment                 = single_pulse_dec.j
Sample_Id                  = ty115 c3
Solvent                    = NONE
Actual_Start_Time          = 21-JAN-2025 19:58:
Revision_Time              = 23-JAN-2025 18:12:

Comment                    = single pulse decou
Data_Format                = 1D COMPLEX
Dim_Size                   = 26214
X_Domain                   = Phosphorus31
Dim_Title                  = Phosphorus31
Dim_Units                  = [ppm]
Dimensions                 = X
Spectrometer               = JNM-ECZ400S/L1

Field_Strength             = 9.3885916[T] (400[
X_Acq_Duration             = 0.23068672[s]
X_Domain                   = Phosphorus31
X_Freq                     = 161.81445204[MHz]
X_Offset                   = 0[ppm]
X_Points                   = 32768
X_Prescans                 = 4
X_Resolution               = 4.33488326[Hz]
X_Sweep                    = 142.04545455[kHz]
X_Sweep_Clipped            = 113.63636364[kHz]
Irr_Domain                 = Proton
Irr_Freq                   = 399.73219668[MHz]
Irr_Offset                 = 5[ppm]
Blanking                   = 5[us]
Clipped                    = FALSE
Scans                      = 16
Total_Scans                = 16

Relaxation_Delay           = 2[s]
Recvr_Gain                 = 56
Temp_Get                   = 20[dC]
X_90_Width                 = 14.62[us]
X_Acq_Time                 = 0.23068672[s]
X_Angle                    = 30[deg]
X_Atn                      = 9[dB]
X_Pulse                    = 4.87333333[us]
Irr_Atn_Dec                = 29.755[dB]
Irr_Atn_Dec_Calc           = 29.755[dB]
Irr_Atn_Dec_Default_Calc = 29.755[dB]
Irr_Atn_Noe                = 29.755[dB]
Irr_Dec_Bandwidth_Hz       = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm      = 11.96453209[ppm]
Irr_Dec_Freq               = 399.73219668[MHz]
Irr_Dec_Merit_Factor       = 2.2
Irr_Decoupling             = TRUE
Irr_Noe                    = TRUE
Irr_Noise                  = WALTZ
Irr_Offset_Default         = 5[ppm]
Irr_Pwidth                 = 0.115[ms]
Irr_Pwidth_Default         = 0.115[ms]
Irr_Pwidth_Default_Calc    = 0.115[ms]
Irr_Pwidth_Temp1           = 0.115[ms]
Irr_Wurst                  = FALSE
Decimation_Rate            = 0
Experiment_Path            = c:\Program Files\J
Initial_Wait               = 1[s]
Noe_Time                   = 2[s]
Noe_Time_Flag              = FALSE
Relaxation_Delay_Calc      = 0[s]
Relaxation_Delay_Temp      = 2[s]
Repetition_Time            = 2.23068672[s]
```

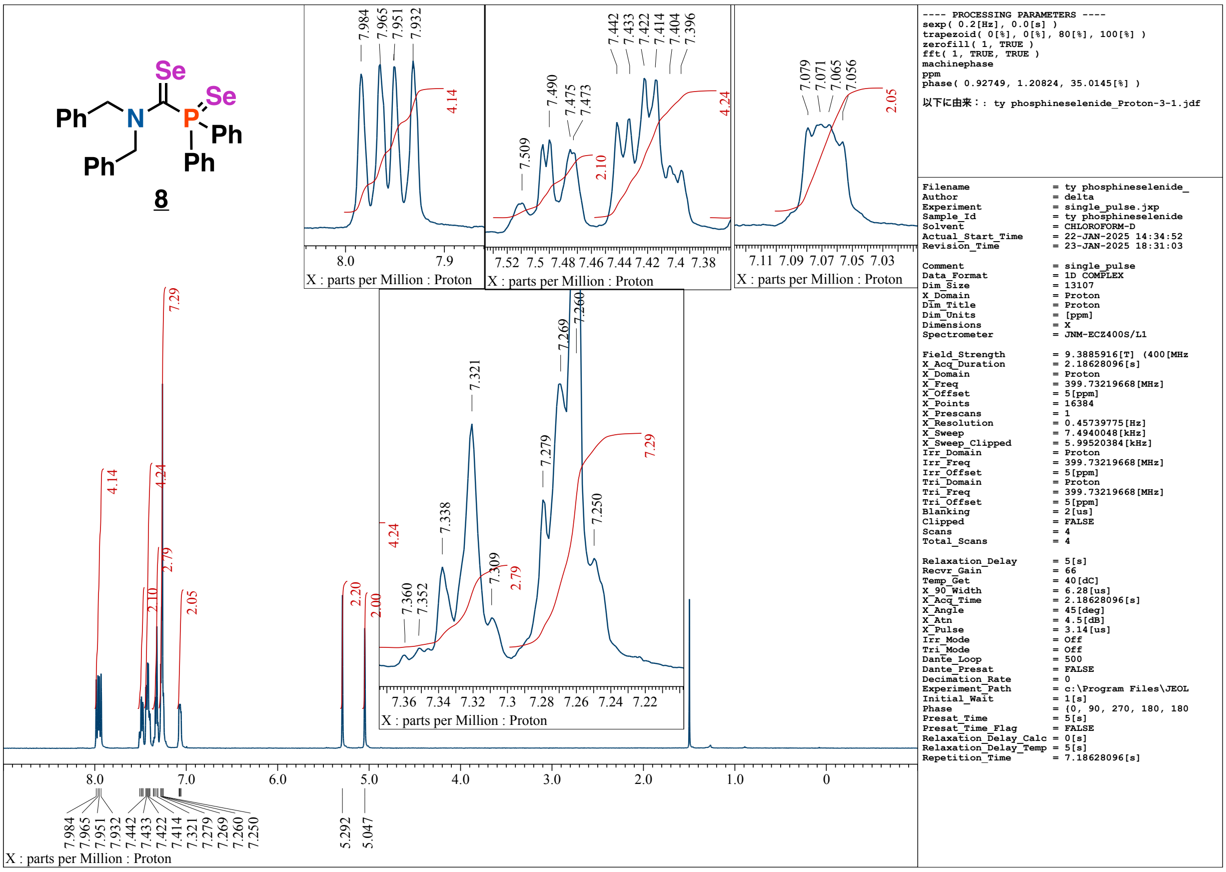
Figure S34. $^{31}P\{^{1}H\}$ NMR spectrum (126 MHz, $CDCl_3$) of **7**.



```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( -33.91384, -9.72337, 50[%] )

以下に由来：: ty115 c3_77Se-1-1.jdf
```

```
Filename                   = ty115 c3_77Se-1-2.
Author                     = delta
Experiment                 = single_pulse_dec.j
Sample_Id                  = ty115 c3
Solvent                    = CHLOROFORM-D
Actual_Start_Time          = 23-JAN-2025 18:38:
Revision_Time              = 23-JAN-2025 22:11:

Comment                    = single pulse decou
Data_Format                = 1D COMPLEX
Dim_Size                   = 26214
X_Domain                   = Selenium77
Dim_Title                  = Selenium77
Dim_Units                  = [ppm]
Dimensions                 = X
Spectrometer               = JNM-ECZ400S/L1

Field_Strength             = 9.3885916[T] (400[
X_Acq_Duration             = 0.23068672[s]
X_Domain                   = Selenium77
X_Freq                     = 76.23488685[MHz]
X_Offset                   = 750[ppm]
X_Points                   = 32768
X_Prescans                 = 4
X_Resolution               = 4.33488326[Hz]
X_Sweep                    = 142.04545455[kHz]
X_Sweep_Clipped            = 113.63636364[kHz]
Irr_Domain                 = Proton
Irr_Freq                   = 399.73219668[MHz]
Irr_Offset                 = 5[ppm]
Blanking                   = 5[us]
Clipped                    = FALSE
Scans                      = 64
Total_Scans                = 64

Relaxation_Delay           = 2[s]
Recvr_Gain                 = 56
Temp_Get                   = 20[dC]
X_90_Width                 = 10.98[us]
X_Acq_Time                 = 0.23068672[s]
X_Angle                    = 30[deg]
X_Atn                      = 8[dB]
X_Pulse                    = 3.66[us]
Irr_Atn_Dec                = 29.755[dB]
Irr_Atn_Dec_Calc           = 29.755[dB]
Irr_Atn_Dec_Default_Calc = 29.755[dB]
Irr_Atn_Noe                = 29.755[dB]
Irr_Dec_Bandwidth_Hz       = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm      = 11.96453209[ppm]
Irr_Dec_Freq               = 399.73219668[MHz]
Irr_Dec_Merit_Factor       = 2.2
Irr_Decoupling             = TRUE
Irr_Noe                    = TRUE
Irr_Noise                  = WALTZ
Irr_Offset_Default         = 5[ppm]
Irr_Pwidth                 = 0.115[ms]
Irr_Pwidth_Default         = 0.115[ms]
Irr_Pwidth_Default_Calc    = 0.115[ms]
Irr_Pwidth_Temp1           = 0.115[ms]
Irr_Wurst                  = FALSE
Decimation_Rate            = 0
Experiment_Path            = c:\Program Files\J
Initial_Wait               = 1[s]
Noe_Time                   = 2[s]
Noe_Time_Flag              = FALSE
Relaxation_Delay_Calc      = 0[s]
Relaxation_Delay_Temp      = 2[s]
Repetition_Time            = 2.23068672[s]
```

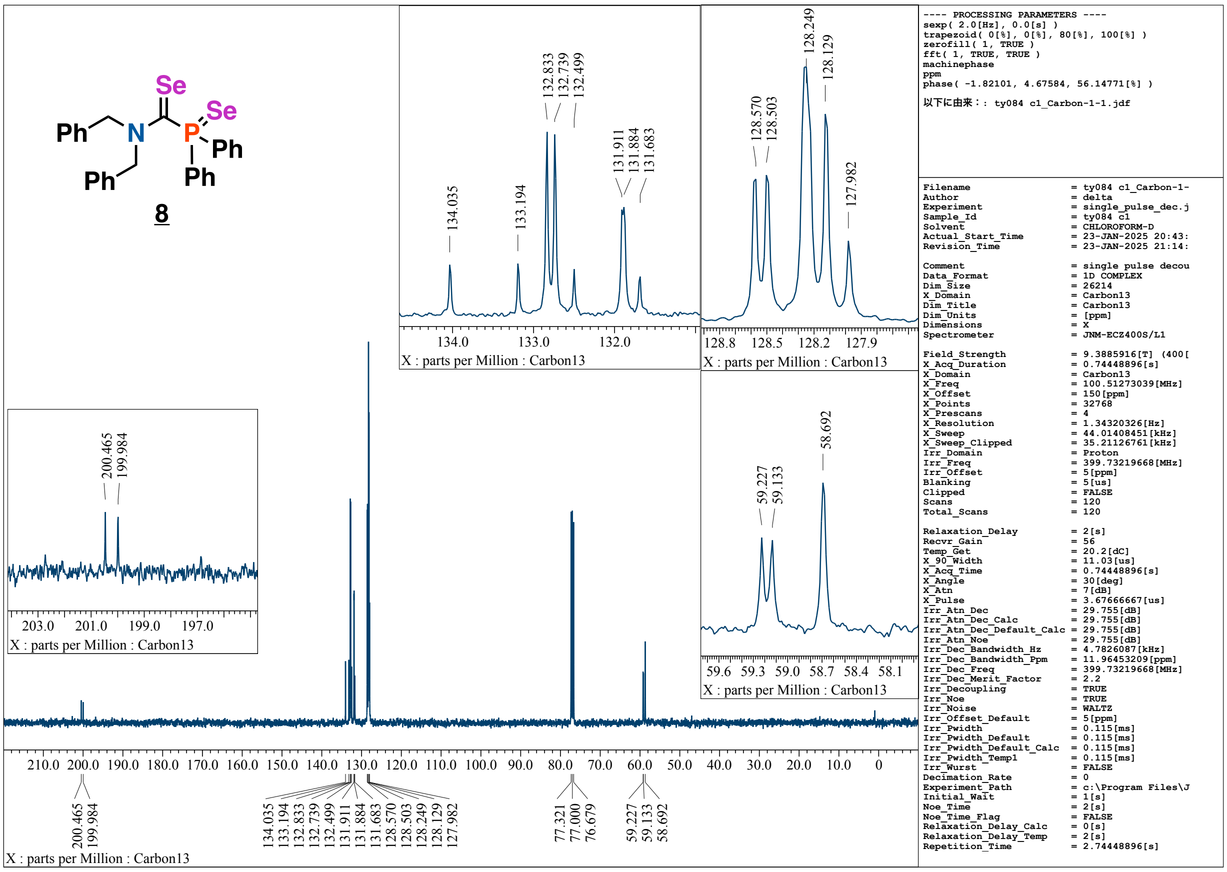
Figure S35. $^{77}Se\{^{1}H\}$ NMR spectrum (76 MHz, $CDCl_3$) of **7**.



```
---- PROCESSING PARAMETERS ----
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( 0.92749, 1.20824, 35.0145[%] )

以下に由来：: ty phosphineselenide_Proton-3-1.jdf
```

```
Filename                 = ty phosphineselenide_
Author                   = delta
Experiment               = single_pulse.jxp
Sample_Id                = ty phosphineselenide
Solvent                  = CHLOROFORM-D
Actual_Start_Time        = 22-JAN-2025 14:34:52
Revision_Time            = 23-JAN-2025 18:31:03

Comment                  = single_pulse
Data_Format              = 1D COMPLEX
Dim_Size                 = 13107
X_Domain                 = Proton
Dim_Title                = Proton
Dim_Units                = [ppm]
Dimensions               = X
Spectrometer             = JNM-ECZ400S/L1

Field_Strength           = 9.3885916[T] (400[MHz
X_Acq_Duration           = 2.18628096[s]
X_Domain                 = Proton
X_Freq                   = 399.73219668[MHz]
X_Offset                 = 5[ppm]
X_Points                 = 16384
X_Prescans               = 1
X_Resolution             = 0.45739775[Hz]
X_Sweep                  = 7.4940048[kHz]
X_Sweep_Clipped          = 5.99520384[kHz]
Irr_Domain               = Proton
Irr_Freq                 = 399.73219668[MHz]
Irr_Offset               = 5[ppm]
Tri_Domain               = Proton
Tri_Freq                 = 399.73219668[MHz]
Tri_Offset               = 5[ppm]
Blanking                 = 2[us]
Clipped                  = FALSE
Scans                    = 4
Total_Scans              = 4

Relaxation_Delay         = 5[s]
Recvr_Gain               = 66
Temp_Get                 = 40[dC]
X_90_Width               = 6.28[us]
X_Acq_Time               = 2.18628096[s]
X_Angle                  = 45[deg]
X_Atn                    = 4.5[dB]
X_Pulse                  = 3.14[us]
Irr_Mode                 = Off
Tri_Mode                 = Off
Dante_Loop               = 500
Dante_Presat             = FALSE
Decimation_Rate          = 0
Experiment_Path          = c:\Program Files\JEOL
Initial_Wait             = 1[s]
Phase                    = {0, 90, 270, 180, 180
Presat_Time              = 5[s]
Presat_Time_Flag         = FALSE
Relaxation_Delay_Calc    = 0[s]
Relaxation_Delay_Temp    = 5[s]
Repetition_Time          = 7.18628096[s]
```

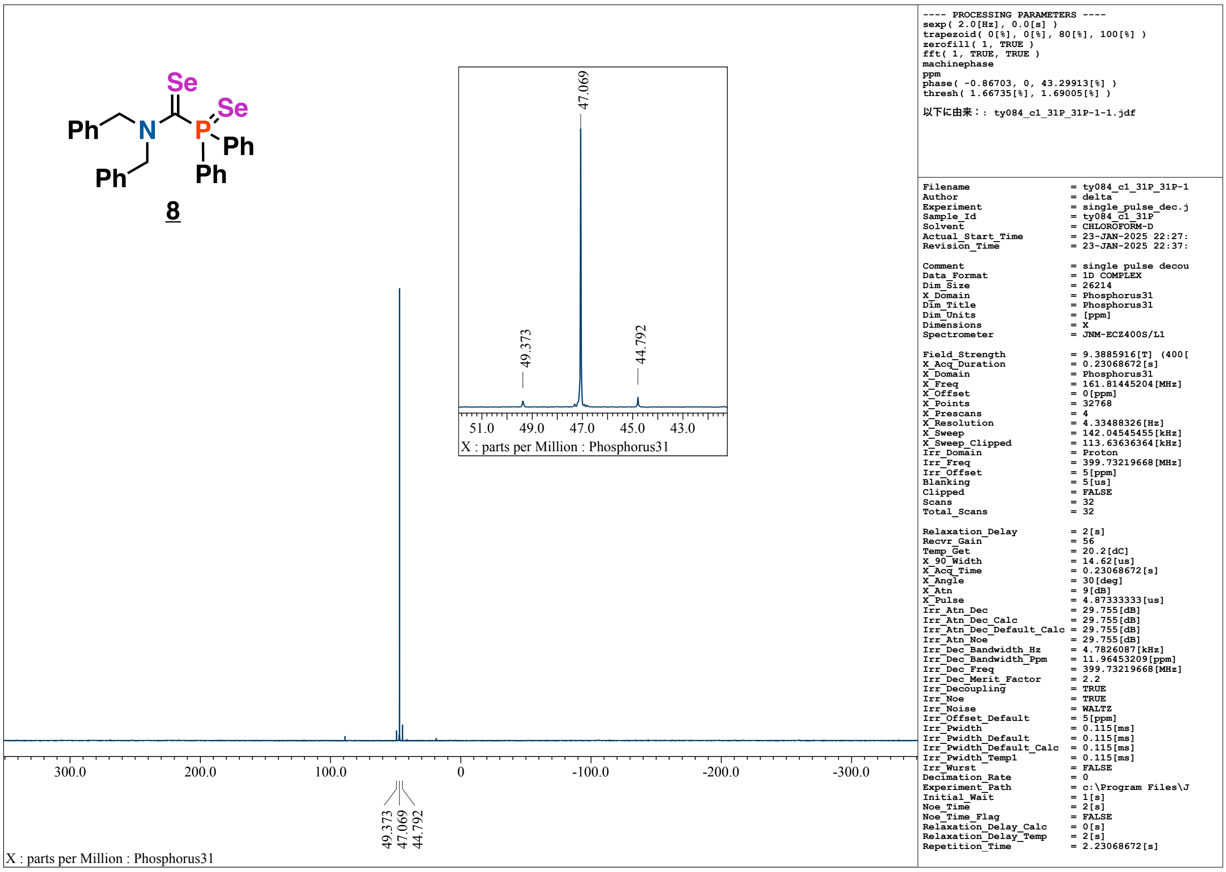
Figure S36. ^{1}H NMR spectrum (400 MHz, $CDCl_3$, 40 °C) of **8**.



```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( -1.82101, 4.67584, 56.14771[%] )

以下に由来：: ty084 c1_Carbon-1-1.jdf
```

```
Filename                 = ty084 c1_Carbon-1-
Author                   = delta
Experiment               = single_pulse_dec.j
Sample_Id                = ty084 c1
Solvent                  = CHLOROFORM-D
Actual_Start_Time        = 23-JAN-2025 20:43:
Revision_Time            = 23-JAN-2025 21:14:

Comment                  = single pulse decou
Data_Format              = 1D COMPLEX
Dim_Size                 = 26214
X_Domain                 = Carbon13
Dim_Title                = Carbon13
Dim_Units                = [ppm]
Dimensions               = X
Spectrometer             = JNM-ECZ400S/L1

Field_Strength           = 9.3885916[T] (400[
X_Acq_Duration           = 0.74448896[s]
X_Domain                 = Carbon13
X_Freq                   = 100.51273039[MHz]
X_Offset                 = 150[ppm]
X_Points                 = 32768
X_Prescans               = 4
X_Resolution             = 1.34320326[Hz]
X_Sweep                  = 44.01408451[kHz]
X_Sweep_Clipped          = 35.21126761[kHz]
Irr_Domain               = Proton
Irr_Freq                 = 399.73219668[MHz]
Irr_Offset               = 5[ppm]
Blanking                 = 5[us]
Clipped                  = FALSE
Scans                    = 120
Total_Scans              = 120

Relaxation_Delay         = 2[s]
Recvr_Gain               = 56
Temp_Get                 = 20.2[dC]
X_90_Width               = 11.03[us]
X_Acq_Time               = 0.74448896[s]
X_Angle                  = 30[deg]
X_Atn                    = 7[dB]
X_Pulse                  = 3.67666667[us]
Irr_Atn_Dec              = 29.755[dB]
Irr_Atn_Dec_Calc         = 29.755[dB]
Irr_Atn_Dec_Default_Calc = 29.755[dB]
Irr_Atn_Noe              = 29.755[dB]
Irr_Dec_Bandwidth_Hz     = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm    = 11.96453209[ppm]
Irr_Dec_Freq             = 399.73219668[MHz]
Irr_Dec_Merit_Factor     = 2.2
Irr_Decoupling           = TRUE
Irr_Noe                  = TRUE
Irr_Noise                = WALTZ
Irr_Offset_Default       = 5[ppm]
Irr_Pwidth               = 0.115[ms]
Irr_Pwidth_Default       = 0.115[ms]
Irr_Pwidth_Default_Calc  = 0.115[ms]
Irr_Pwidth_Temp1         = 0.115[ms]
Irr_Wurst                = FALSE
Decimation_Rate          = 0
Experiment_Path          = c:\Program Files\J
Initial_Wait             = 1[s]
Noe_Time                 = 2[s]
Noe_Time_Flag            = FALSE
Relaxation_Delay_Calc    = 0[s]
Relaxation_Delay_Temp    = 2[s]
Repetition_Time          = 2.74448896[s]
```

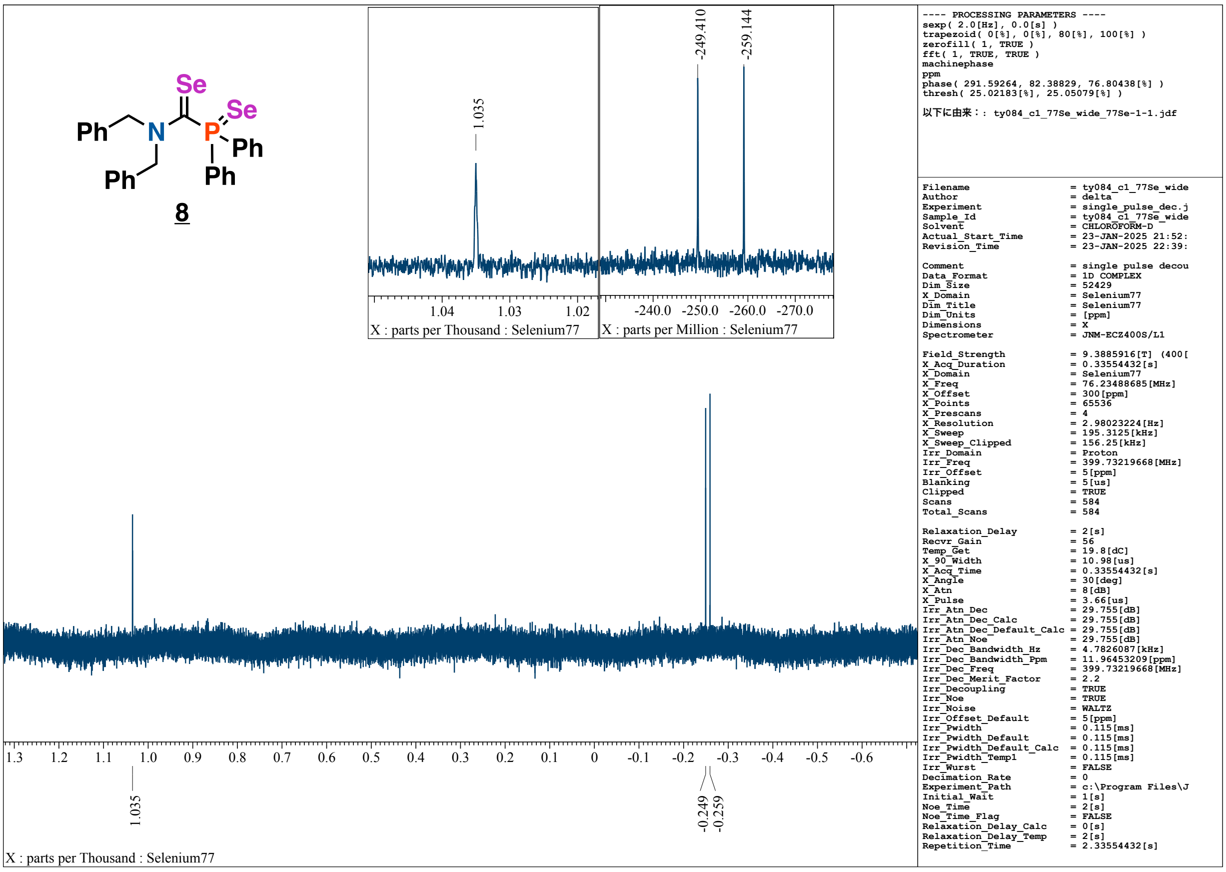
Figure S37. $^{13}C\{^{1}H\}$ NMR spectrum (100 MHz, $CDCl_3$) of **8**.



```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( -0.86703, 0, 43.29913[%] )
thresh( 1.66735[%], 1.69005[%] )

以下に由来：: ty084_c1_31P_31P-1-1.jdf
```

```
Filename                    = ty084_c1_31P_31P-1
Author                      = delta
Experiment                  = single_pulse_dec.j
Sample_Id                   = ty084_c1_31P
Solvent                     = CHLOROFORM-D
Actual_Start_Time           = 23-JAN-2025 22:27:
Revision_Time               = 23-JAN-2025 22:37:

Comment                     = single pulse decou
Data_Format                 = 1D COMPLEX
Dim_Size                    = 26214
X_Domain                    = Phosphorus31
Dim_Title                   = Phosphorus31
Dim_Units                   = [ppm]
Dimensions                  = X
Spectrometer                = JNM-ECZ400S/L1

Field_Strength              = 9.3885916[T] (400[
X_Acq_Duration              = 0.23068672[s]
X_Domain                    = Phosphorus31
X_Freq                      = 161.81445204[MHz]
X_Offset                    = 0[ppm]
X_Points                    = 32768
X_Prescans                  = 4
X_Resolution                = 4.33488326[Hz]
X_Sweep                     = 142.04545455[kHz]
X_Sweep_Clipped             = 113.63636364[kHz]
Irr_Domain                  = Proton
Irr_Freq                    = 399.73219668[MHz]
Irr_Offset                  = 5[ppm]
Blanking                    = 5[us]
Clipped                     = FALSE
Scans                       = 32
Total_Scans                 = 32

Relaxation_Delay            = 2[s]
Recvr_Gain                  = 56
Temp_Get                    = 20.2[dC]
X_90_Width                  = 14.62[us]
X_Acq_Time                  = 0.23068672[s]
X_Angle                     = 30[deg]
X_Atn                       = 9[dB]
X_Pulse                     = 4.87333333[us]
Irr_Atn_Dec                 = 29.755[dB]
Irr_Atn_Dec_Calc            = 29.755[dB]
Irr_Atn_Dec_Default_Calc    = 29.755[dB]
Irr_Atn_Noe                 = 29.755[dB]
Irr_Dec_Bandwidth_Hz        = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm       = 11.96453209[ppm]
Irr_Dec_Freq                = 399.73219668[MHz]
Irr_Dec_Merit_Factor        = 2.2
Irr_Decoupling              = TRUE
Irr_Noe                     = TRUE
Irr_Noise                   = WALTZ
Irr_Offset_Default          = 5[ppm]
Irr_Pwidth                  = 0.115[ms]
Irr_Pwidth_Default          = 0.115[ms]
Irr_Pwidth_Default_Calc     = 0.115[ms]
Irr_Pwidth_Temp1            = 0.115[ms]
Irr_Wurst                   = FALSE
Decimation_Rate             = 0
Experiment_Path             = c:\Program Files\J
Initial_Wait                = 1[s]
Noe_Time                    = 2[s]
Noe_Time_Flag               = FALSE
Relaxation_Delay_Calc       = 0[s]
Relaxation_Delay_Temp       = 2[s]
Repetition_Time             = 2.23068672[s]
```

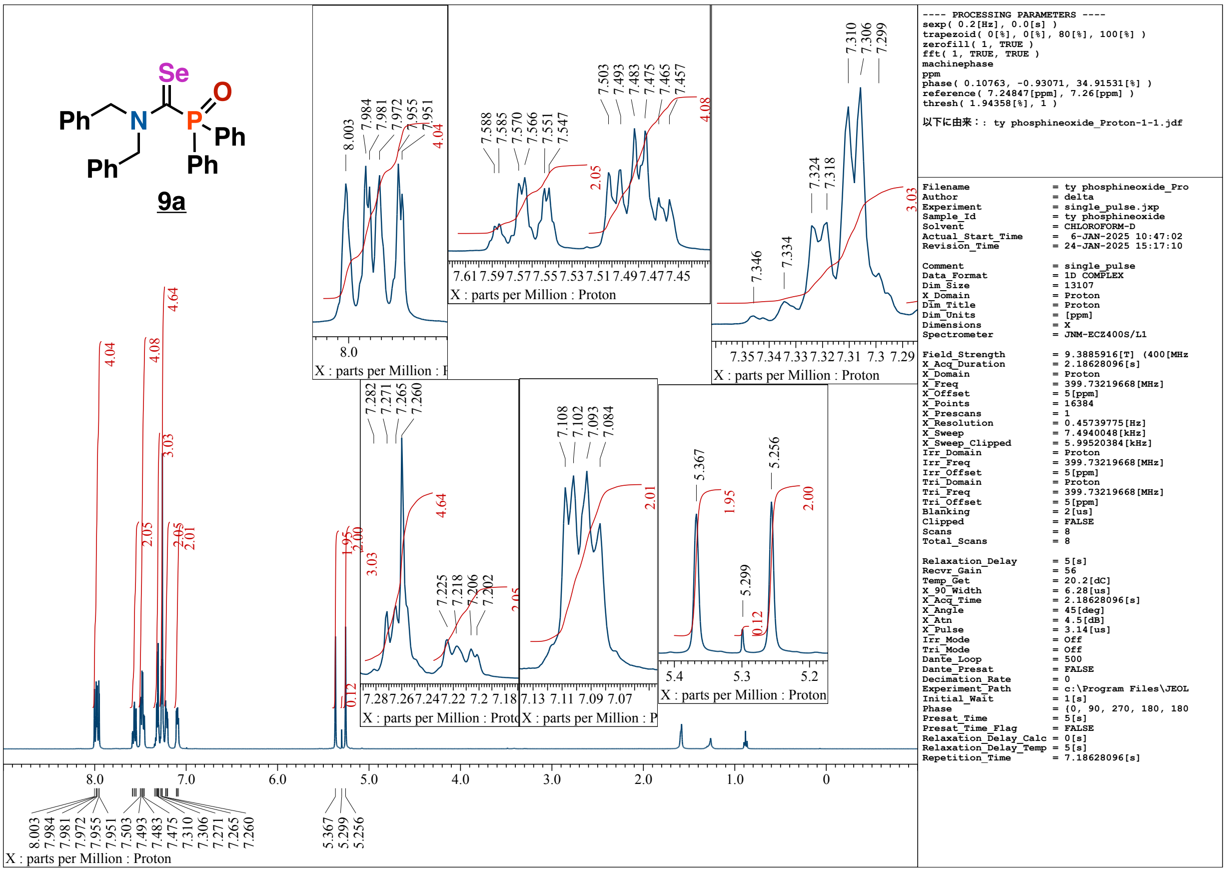
Figure S38. $^{31}P\{^1H\}$ NMR spectrum (162 MHz, $CDCl_3$) of **8**.



```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( 291.59264, 82.38829, 76.80438[%] )
thresh( 25.02183[%], 25.05079[%] )

以下に由来：: ty084_c1_77Se_wide_77Se-1-1.jdf
```

```
Filename                    = ty084_c1_77Se_wide
Author                      = delta
Experiment                  = single_pulse_dec.j
Sample_Id                   = ty084_c1_77Se_wide
Solvent                     = CHLOROFORM-D
Actual_Start_Time           = 23-JAN-2025 21:52:
Revision_Time               = 23-JAN-2025 22:39:

Comment                     = single pulse decou
Data_Format                 = 1D COMPLEX
Dim_Size                    = 52429
X_Domain                    = Selenium77
Dim_Title                   = Selenium77
Dim_Units                   = [ppm]
Dimensions                  = X
Spectrometer                = JNM-ECZ400S/L1

Field_Strength              = 9.3885916[T] (400[
X_Acq_Duration              = 0.33554432[s]
X_Domain                    = Selenium77
X_Freq                      = 76.23488685[MHz]
X_Offset                    = 300[ppm]
X_Points                    = 65536
X_Prescans                  = 4
X_Resolution                = 2.98023224[Hz]
X_Sweep                     = 195.3125[kHz]
X_Sweep_Clipped             = 156.25[kHz]
Irr_Domain                  = Proton
Irr_Freq                    = 399.73219668[MHz]
Irr_Offset                  = 5[ppm]
Blanking                    = 5[us]
Clipped                     = TRUE
Scans                       = 584
Total_Scans                 = 584

Relaxation_Delay            = 2[s]
Recvr_Gain                  = 56
Temp_Get                    = 19.8[dC]
X_90_Width                  = 10.98[us]
X_Acq_Time                  = 0.33554432[s]
X_Angle                     = 30[deg]
X_Atn                       = 8[dB]
X_Pulse                     = 3.66[us]
Irr_Atn_Dec                 = 29.755[dB]
Irr_Atn_Dec_Calc            = 29.755[dB]
Irr_Atn_Dec_Default_Calc    = 29.755[dB]
Irr_Atn_Noe                 = 29.755[dB]
Irr_Dec_Bandwidth_Hz        = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm       = 11.96453209[ppm]
Irr_Dec_Freq                = 399.73219668[MHz]
Irr_Dec_Merit_Factor        = 2.2
Irr_Decoupling              = TRUE
Irr_Noe                     = TRUE
Irr_Noise                   = WALTZ
Irr_Offset_Default          = 5[ppm]
Irr_Pwidth                  = 0.115[ms]
Irr_Pwidth_Default          = 0.115[ms]
Irr_Pwidth_Default_Calc     = 0.115[ms]
Irr_Pwidth_Temp1            = 0.115[ms]
Irr_Wurst                   = FALSE
Decimation_Rate             = 0
Experiment_Path             = c:\Program Files\J
Initial_Wait                = 1[s]
Noe_Time                    = 2[s]
Noe_Time_Flag               = FALSE
Relaxation_Delay_Calc       = 0[s]
Relaxation_Delay_Temp       = 2[s]
Repetition_Time             = 2.33554432[s]
```

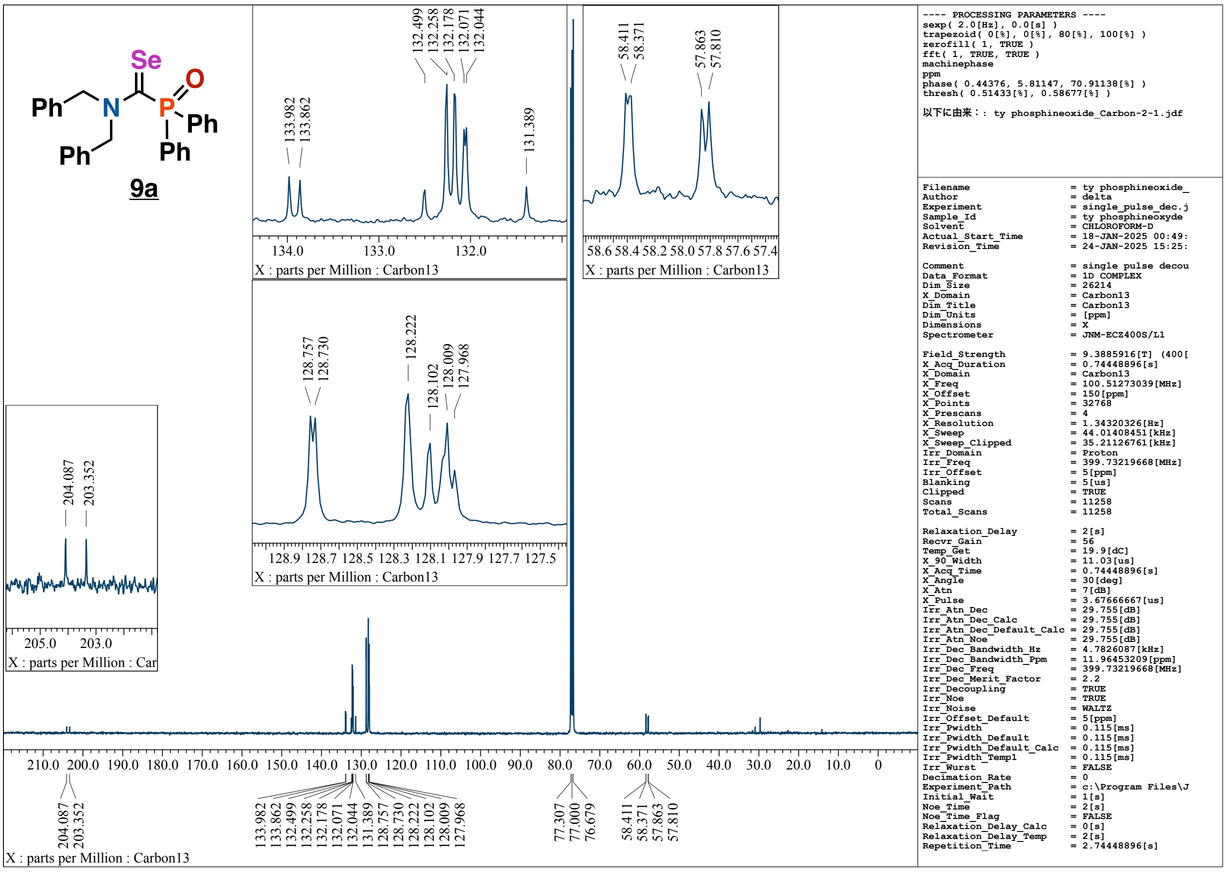
Figure S39. $^{77}Se\{^1H\}$ NMR spectrum (76 MHz, $CDCl_3$) of **8**.



```
---- PROCESSING PARAMETERS ----
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( 0.10763, -0.93071, 34.91531[%] )
reference( 7.24847[ppm], 7.26[ppm] )
thresh( 1.94358[%], 1 )

以下に由来：: ty phosphineoxide_Proton-1-1.jdf
```

```
Filename                = ty phosphineoxide_Pro
Author                  = delta
Experiment              = single_pulse.jxp
Sample_Id               = ty phosphineoxide
Solvent                 = CHLOROFORM-D
Actual_Start_Time       =  6-JAN-2025 10:47:02
Revision_Time           = 24-JAN-2025 15:17:10

Comment                 = single_pulse
Data_Format             = 1D COMPLEX
Dim_Size                = 13107
X_Domain                = Proton
Dim_Title               = Proton
Dim_Units               = [ppm]
Dimensions              = X
Spectrometer            = JNM-ECZ400S/L1

Field_Strength          = 9.3885916[T] (400[MHz
X_Acq_Duration          = 2.18628096[s]
X_Domain                = Proton
X_Freq                  = 399.73219668[MHz]
X_Offset                = 5[ppm]
X_Points                = 16384
X_Prescans              = 1
X_Resolution            = 0.45739775[Hz]
X_Sweep                 = 7.4940048[kHz]
X_Sweep_Clipped         = 5.99520384[kHz]
Irr_Domain              = Proton
Irr_Freq                = 399.73219668[MHz]
Irr_Offset              = 5[ppm]
Tri_Domain              = Proton
Tri_Freq                = 399.73219668[MHz]
Tri_Offset              = 5[ppm]
Blanking                = 2[us]
Clipped                 = FALSE
Scans                   = 8
Total_Scans             = 8

Relaxation_Delay        = 5[s]
Recvr_Gain              = 56
Temp_Get                = 20.2[dC]
X_90_Width              = 6.28[us]
X_Acq_Time              = 2.18628096[s]
X_Angle                 = 45[deg]
X_Atn                   = 4.5[dB]
X_Pulse                 = 3.14[us]
Irr_Mode                = Off
Tri_Mode                = Off
Dante_Loop              = 500
Dante_Presat            = FALSE
Decimation_Rate         = 0
Experiment_Path         = c:\Program Files\JEOL
Initial_Wait            = 1[s]
Phase                   = {0, 90, 270, 180, 180
Presat_Time             = 5[s]
Presat_Time_Flag        = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 5[s]
Repetition_Time         = 7.18628096[s]
```

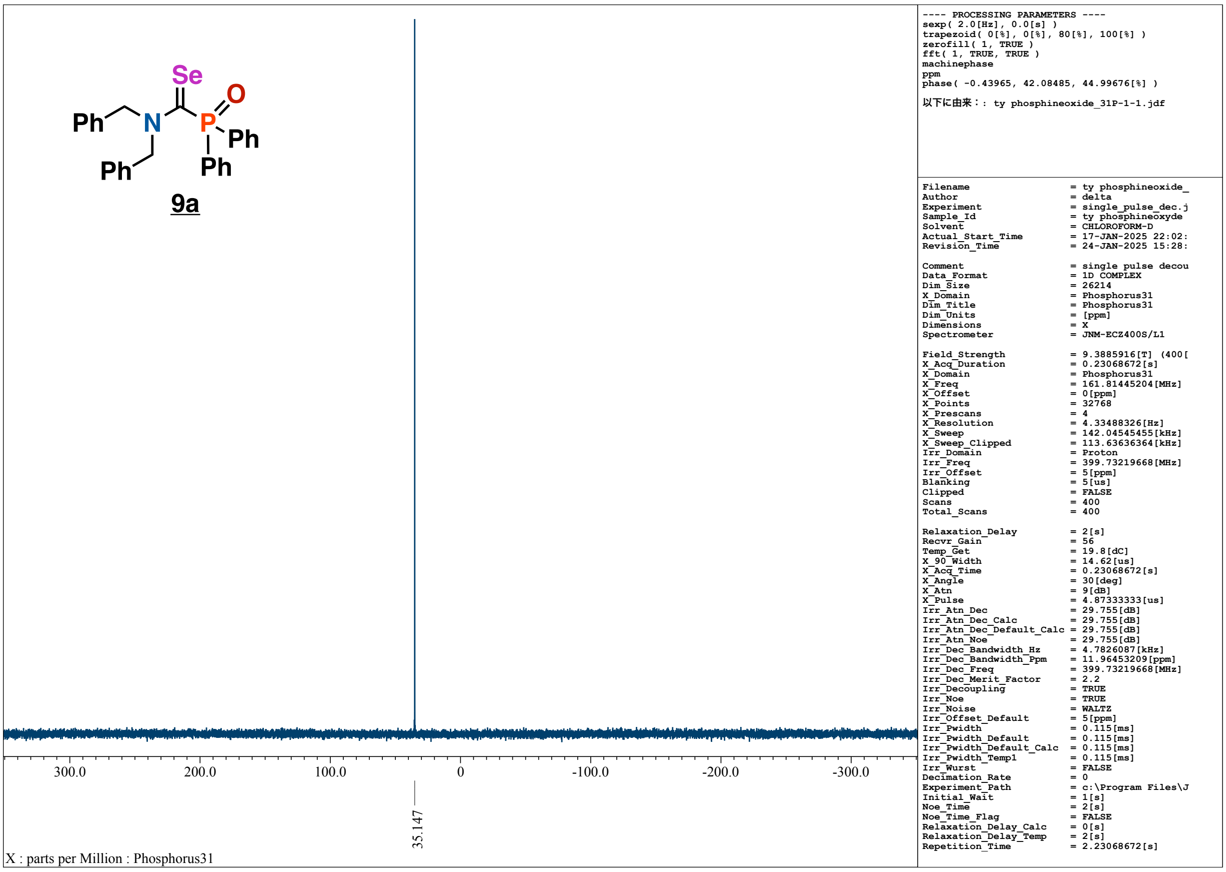
Figure S40. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **9a**.



```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( 0.44376, 5.81147, 70.91138[%] )
thresh( 0.51433[%], 0.58677[%] )

以下に由来：: ty phosphineoxide_Carbon-2-1.jdf
```

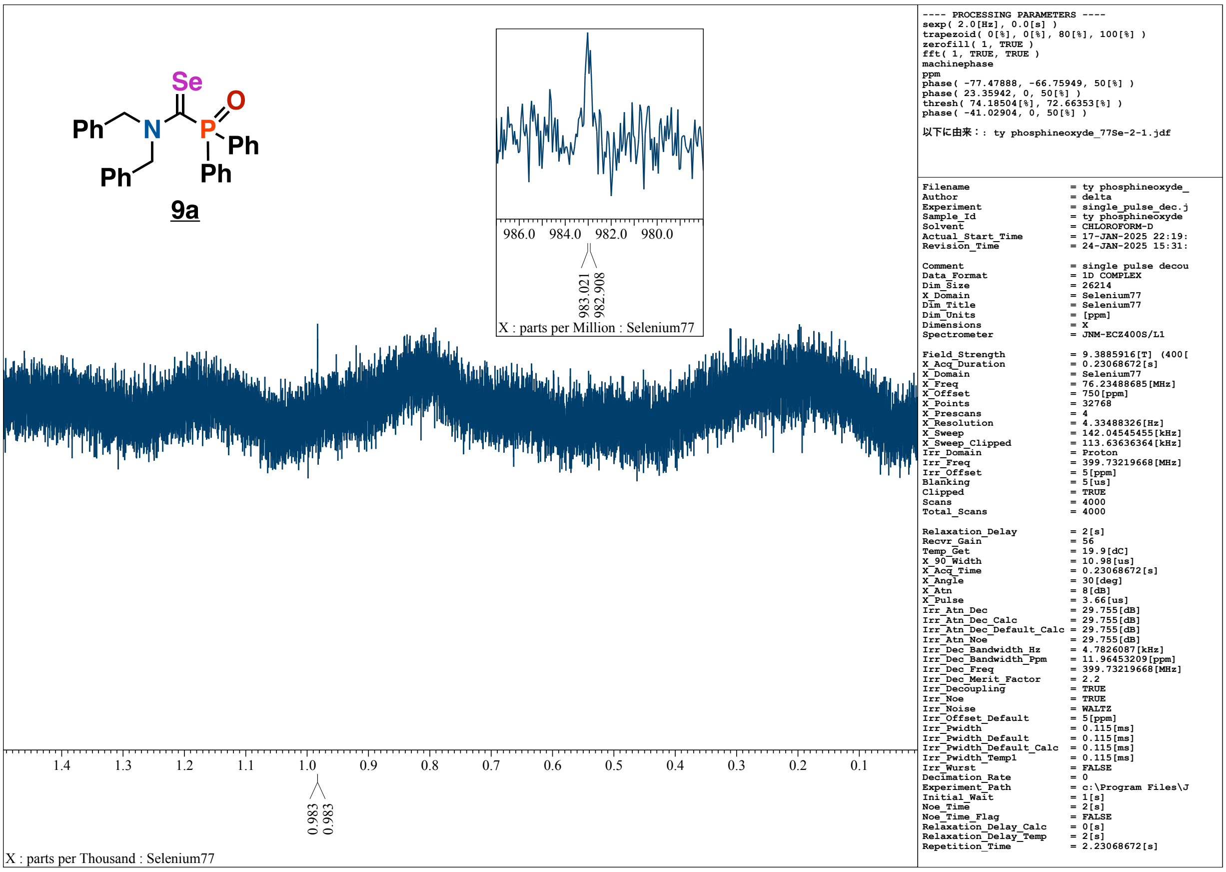
```
Filename                    = ty phosphineoxide_
Author                      = delta
Experiment                  = single_pulse_dec.j
Sample_Id                   = ty phosphineoxyde
Solvent                     = CHLOROFORM-D
Actual_Start_Time           = 18-JAN-2025 00:49:
Revision_Time               = 24-JAN-2025 15:25:

Comment                     = single pulse decou
Data_Format                 = 1D COMPLEX
Dim_Size                    = 26214
X_Domain                    = Carbon13
Dim_Title                   = Carbon13
Dim_Units                   = [ppm]
Dimensions                  = X
Spectrometer                = JNM-ECZ400S/L1

Field_Strength              = 9.3885916[T] (400[
X_Acq_Duration              = 0.74448896[s]
X_Domain                    = Carbon13
X_Freq                      = 100.51273039[MHz]
X_Offset                    = 150[ppm]
X_Points                    = 32768
X_Prescans                  = 4
X_Resolution                = 1.34320326[Hz]
X_Sweep                     = 44.01408451[kHz]
X_Sweep_Clipped             = 35.21126761[kHz]
Irr_Domain                  = Proton
Irr_Freq                    = 399.73219668[MHz]
Irr_Offset                  = 5[ppm]
Blanking                    = 5[us]
Clipped                     = TRUE
Scans                       = 11258
Total_Scans                 = 11258

Relaxation_Delay            = 2[s]
Recvr_Gain                  = 56
Temp_Get                    = 19.9[dC]
X_90_Width                  = 11.03[us]
X_Acq_Time                  = 0.74448896[s]
X_Angle                     = 30[deg]
X_Atn                       = 7[dB]
X_Pulse                     = 3.67666667[us]
Irr_Atn_Dec                 = 29.755[dB]
Irr_Atn_Dec_Calc            = 29.755[dB]
Irr_Atn_Dec_Default_Calc = 29.755[dB]
Irr_Atn_Noe                 = 29.755[dB]
Irr_Dec_Bandwidth_Hz        = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm       = 11.96453209[ppm]
Irr_Dec_Freq                = 399.73219668[MHz]
Irr_Dec_Merit_Factor        = 2.2
Irr_Decoupling              = TRUE
Irr_Noe                     = TRUE
Irr_Noise                   = WALTZ
Irr_Offset_Default          = 5[ppm]
Irr_Pwidth                  = 0.115[ms]
Irr_Pwidth_Default          = 0.115[ms]
Irr_Pwidth_Default_Calc     = 0.115[ms]
Irr_Pwidth_Temp1            = 0.115[ms]
Irr_Wurst                   = FALSE
Decimation_Rate             = 0
Experiment_Path             = c:\Program Files\J
Initial_Wait                = 1[s]
Noe_Time                    = 2[s]
Noe_Time_Flag               = FALSE
Relaxation_Delay_Calc       = 0[s]
Relaxation_Delay_Temp       = 2[s]
Repetition_Time             = 2.74448896[s]
```

Figure S41. $^{13}C\{^1H\}$ NMR spectrum (100 MHz, $CDCl_3$) of **10**.



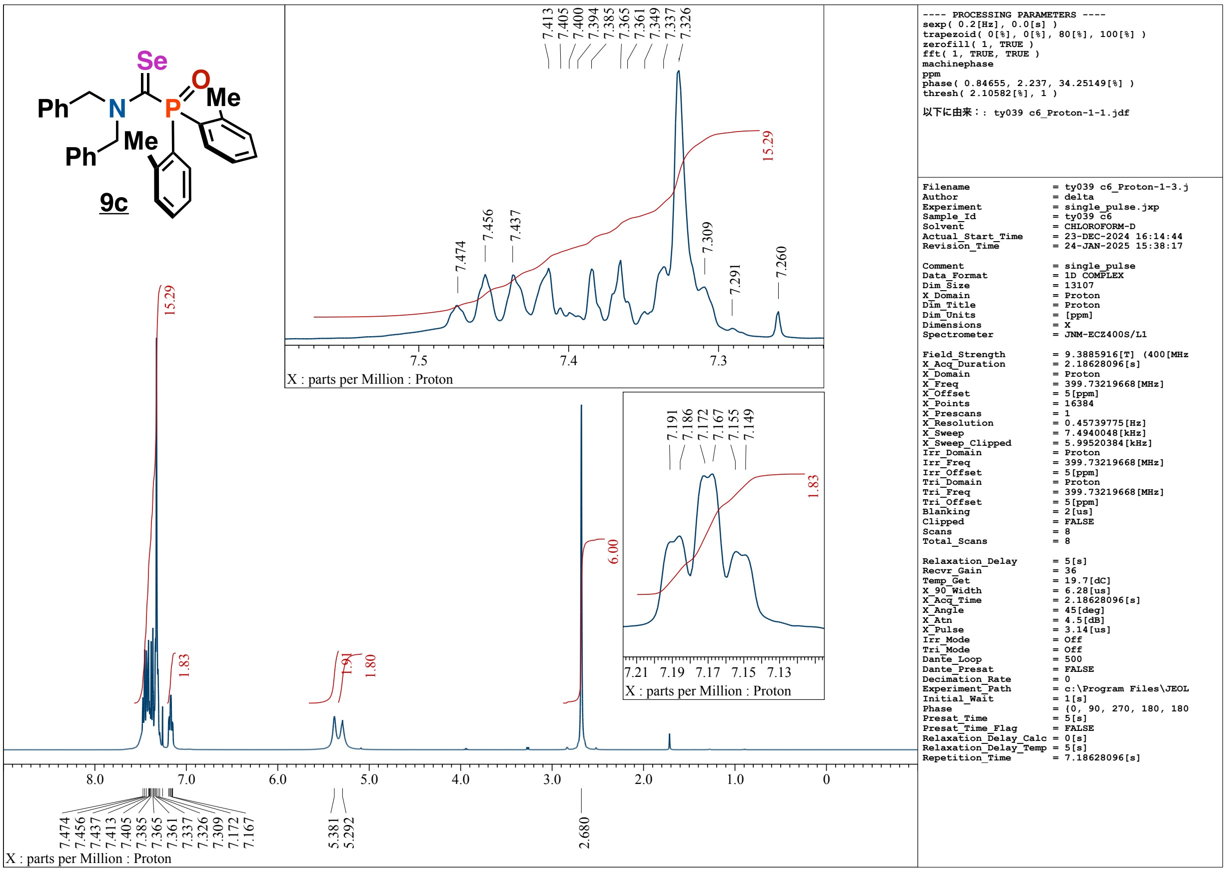
```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( -0.43965, 42.08485, 44.99676[%] )

以下に由来：: ty phosphineoxide_31P-1-1.jdf

Filename                    = ty phosphineoxide_
Author                      = delta
Experiment                  = single_pulse_dec.j
Sample_Id                   = ty phosphineoxyde
Solvent                     = CHLOROFORM-D
Actual_Start_Time           = 17-JAN-2025 22:02:
Revision_Time               = 24-JAN-2025 15:28:

Comment                     = single pulse decou
Data_Format                 = 1D COMPLEX
Dim_Size                    = 26214
X_Domain                    = Phosphorus31
Dim_Title                   = Phosphorus31
Dim_Units                   = [ppm]
Dimensions                  = X
Spectrometer                = JNM-ECZ400S/L1

Field_Strength              = 9.3885916[T] (400[
X_Acq_Duration              = 0.23068672[s]
X_Domain                    = Phosphorus31
X_Freq                      = 161.81445204[MHz]
X_Offset                    = 0[ppm]
X_Points                    = 32768
X_Prescans                  = 4
X_Resolution                = 4.33488326[Hz]
X_Sweep                     = 142.04545455[kHz]
X_Sweep_Clipped             = 113.63636364[kHz]
Irr_Domain                  = Proton
Irr_Freq                    = 399.73219668[MHz]
Irr_Offset                  = 5[ppm]
Blanking                    = 5[us]
Clipped                     = FALSE
Scans                       = 400
Total_Scans                 = 400

Relaxation_Delay            = 2[s]
Recvr_Gain                  = 56
Temp_Get                    = 19.8[dC]
X_90_Width                  = 14.62[us]
X_Acq_Time                  = 0.23068672[s]
X_Angle                     = 30[deg]
X_Atn                       = 9[dB]
X_Pulse                     = 4.87333333[us]
Irr_Atn_Dec                 = 29.755[dB]
Irr_Atn_Dec_Calc            = 29.755[dB]
Irr_Atn_Dec_Default_Calc    = 29.755[dB]
Irr_Atn_Noe                 = 29.755[dB]
Irr_Dec_Bandwidth_Hz        = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm       = 11.96453209[ppm]
Irr_Dec_Freq                = 399.73219668[MHz]
Irr_Dec_Merit_Factor        = 2.2
Irr_Decoupling              = TRUE
Irr_Noe                     = TRUE
Irr_Noise                   = WALTZ
Irr_Offset_Default          = 5[ppm]
Irr_Pwidth                  = 0.115[ms]
Irr_Pwidth_Default          = 0.115[ms]
Irr_Pwidth_Default_Calc     = 0.115[ms]
Irr_Pwidth_Temp1            = 0.115[ms]
Irr_Wurst                   = FALSE
Decimation_Rate             = 0
Experiment_Path             = c:\Program Files\J
Initial_Wait                = 1[s]
Noe_Time                    = 2[s]
Noe_Time_Flag               = FALSE
Relaxation_Delay_Calc       = 0[s]
Relaxation_Delay_Temp       = 2[s]
Repetition_Time             = 2.23068672[s]
```

Figure S42. $^{31}P\{^1H\}$ NMR spectrum (126 MHz, $CDCl_3$) of **9a**.



```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( -77.47888, -66.75949, 50[%] )
phase( 23.35942, 0, 50[%] )
thresh( 74.18504[%], 72.66353[%] )
phase( -41.02904, 0, 50[%] )

以下に由来：: ty phosphineoxyde_77Se-2-1.jdf

Filename                    = ty phosphineoxyde_
Author                      = delta
Experiment                  = single_pulse_dec.j
Sample_Id                   = ty phosphineoxyde
Solvent                     = CHLOROFORM-D
Actual_Start_Time           = 17-JAN-2025 22:19:
Revision_Time               = 24-JAN-2025 15:31:

Comment                     = single pulse decou
Data_Format                 = 1D COMPLEX
Dim_Size                    = 26214
X_Domain                    = Selenium77
Dim_Title                   = Selenium77
Dim_Units                   = [ppm]
Dimensions                  = X
Spectrometer                = JNM-ECZ400S/L1

Field_Strength              = 9.3885916[T] (400[
X_Acq_Duration              = 0.23068672[s]
X_Domain                    = Selenium77
X_Freq                      = 76.23488685[MHz]
X_Offset                    = 750[ppm]
X_Points                    = 32768
X_Prescans                  = 4
X_Resolution                = 4.33488326[Hz]
X_Sweep                     = 142.04545455[kHz]
X_Sweep_Clipped             = 113.63636364[kHz]
Irr_Domain                  = Proton
Irr_Freq                    = 399.73219668[MHz]
Irr_Offset                  = 5[ppm]
Blanking                    = 5[us]
Clipped                     = TRUE
Scans                       = 4000
Total_Scans                 = 4000

Relaxation_Delay            = 2[s]
Recvr_Gain                  = 56
Temp_Get                    = 19.9[dC]
X_90_Width                  = 10.98[us]
X_Acq_Time                  = 0.23068672[s]
X_Angle                     = 30[deg]
X_Atn                       = 8[dB]
X_Pulse                     = 3.66[us]
Irr_Atn_Dec                 = 29.755[dB]
Irr_Atn_Dec_Calc            = 29.755[dB]
Irr_Atn_Dec_Default_Calc    = 29.755[dB]
Irr_Atn_Noe                 = 29.755[dB]
Irr_Dec_Bandwidth_Hz        = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm       = 11.96453209[ppm]
Irr_Dec_Freq                = 399.73219668[MHz]
Irr_Dec_Merit_Factor        = 2.2
Irr_Decoupling              = TRUE
Irr_Noe                     = TRUE
Irr_Noise                   = WALTZ
Irr_Offset_Default          = 5[ppm]
Irr_Pwidth                  = 0.115[ms]
Irr_Pwidth_Default          = 0.115[ms]
Irr_Pwidth_Default_Calc     = 0.115[ms]
Irr_Pwidth_Temp1            = 0.115[ms]
Irr_Wurst                   = FALSE
Decimation_Rate             = 0
Experiment_Path             = c:\Program Files\J
Initial_Wait                = 1[s]
Noe_Time                    = 2[s]
Noe_Time_Flag               = FALSE
Relaxation_Delay_Calc       = 0[s]
Relaxation_Delay_Temp       = 2[s]
Repetition_Time             = 2.23068672[s]
```

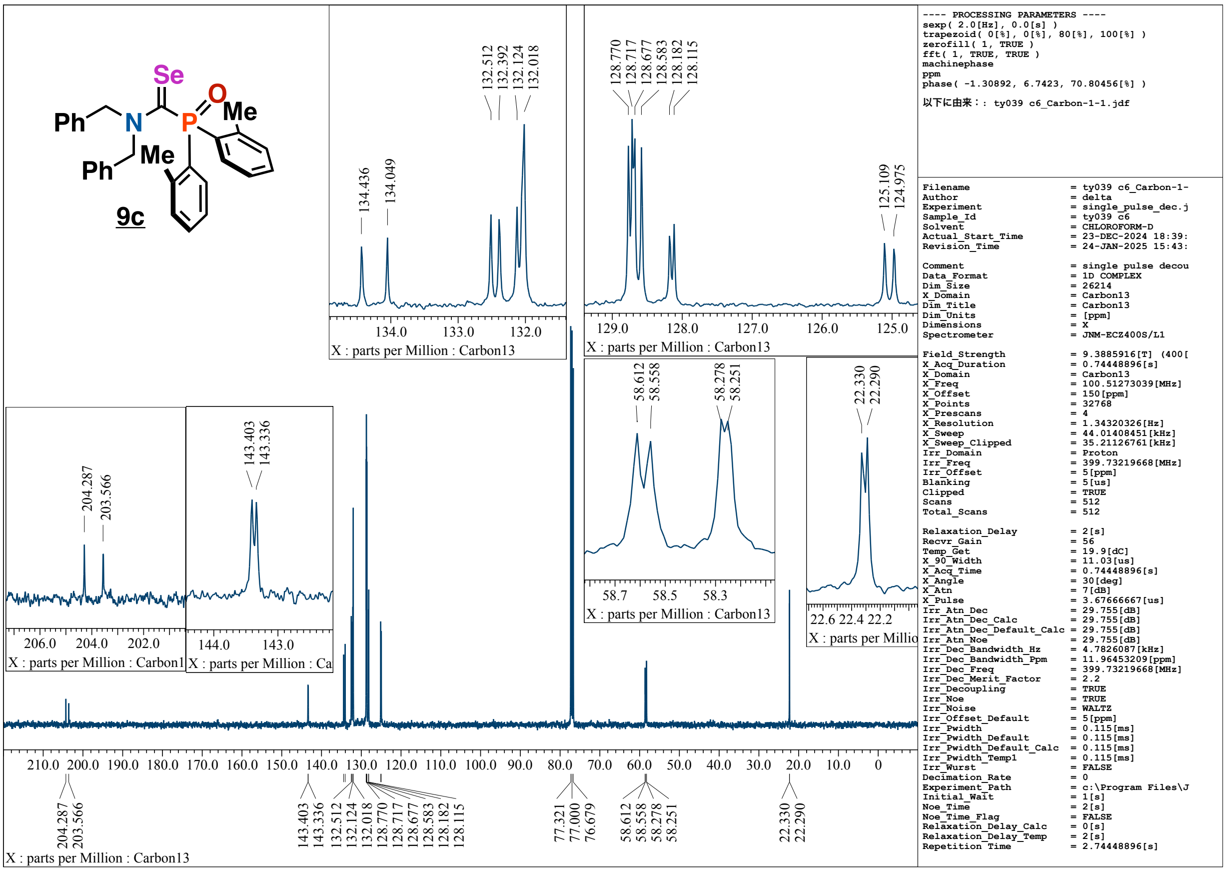
Figure S43. $^{77}Se\{^1H\}$ NMR spectrum (76 MHz, $CDCl_3$) of **9a**.



```
---- PROCESSING PARAMETERS ----
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( 0.84655, 2.237, 34.25149[%] )
thresh( 2.10582[%], 1 )

以下に由来：: ty039 c6_Proton-1-1.jdf
```

```
Filename                = ty039 c6_Proton-1-3.j
Author                  = delta
Experiment              = single_pulse.jxp
Sample_Id               = ty039 c6
Solvent                 = CHLOROFORM-D
Actual_Start_Time       = 23-DEC-2024 16:14:44
Revision_Time           = 24-JAN-2025 15:38:17

Comment                 = single_pulse
Data_Format             = 1D COMPLEX
Dim_Size                = 13107
X_Domain                = Proton
Dim_Title               = Proton
Dim_Units               = [ppm]
Dimensions              = X
Spectrometer            = JNM-ECZ400S/L1

Field_Strength          = 9.3885916[T] (400[MHz
X_Acq_Duration          = 2.18628096[s]
X_Domain                = Proton
X_Freq                  = 399.73219668[MHz]
X_Offset                = 5[ppm]
X_Points                = 16384
X_Prescans              = 1
X_Resolution            = 0.45739775[Hz]
X_Sweep                 = 7.4940048[kHz]
X_Sweep_Clipped         = 5.99520384[kHz]
Irr_Domain              = Proton
Irr_Freq                = 399.73219668[MHz]
Irr_Offset              = 5[ppm]
Tri_Domain              = Proton
Tri_Freq                = 399.73219668[MHz]
Tri_Offset              = 5[ppm]
Blanking                = 2[us]
Clipped                 = FALSE
Scans                   = 8
Total_Scans             = 8

Relaxation_Delay        = 5[s]
Recvr_Gain              = 36
Temp_Get                = 19.7[dC]
X_90_Width              = 6.28[us]
X_Acq_Time              = 2.18628096[s]
X_Angle                 = 45[deg]
X_Atn                   = 4.5[dB]
X_Pulse                 = 3.14[us]
Irr_Mode                = Off
Tri_Mode                = Off
Dante_Loop              = 500
Dante_Presat            = FALSE
Decimation_Rate         = 0
Experiment_Path         = c:\Program Files\JEOL
Initial_Wait            = 1[s]
Phase                   = {0, 90, 270, 180, 180
Presat_Time             = 5[s]
Presat_Time_Flag        = FALSE
Relaxation_Delay_Calc   = 0[s]
Relaxation_Delay_Temp   = 5[s]
Repetition_Time         = 7.18628096[s]
```

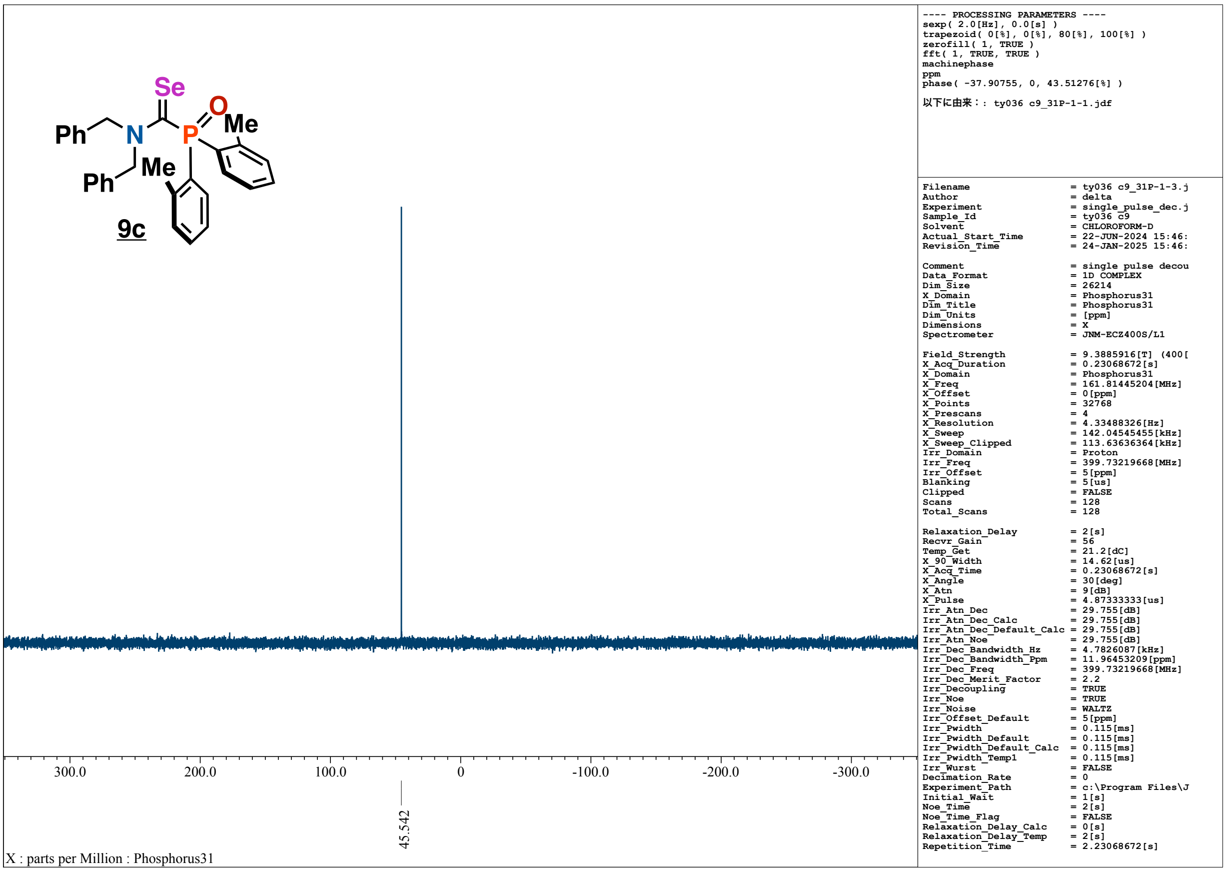
Figure S44. ^{1}H NMR spectrum (400 MHz, CDCl$_3$) of **9c**.



```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( -1.30892, 6.7423, 70.80456[%] )

以下に由来：: ty039 c6_Carbon-1-1.jdf
```

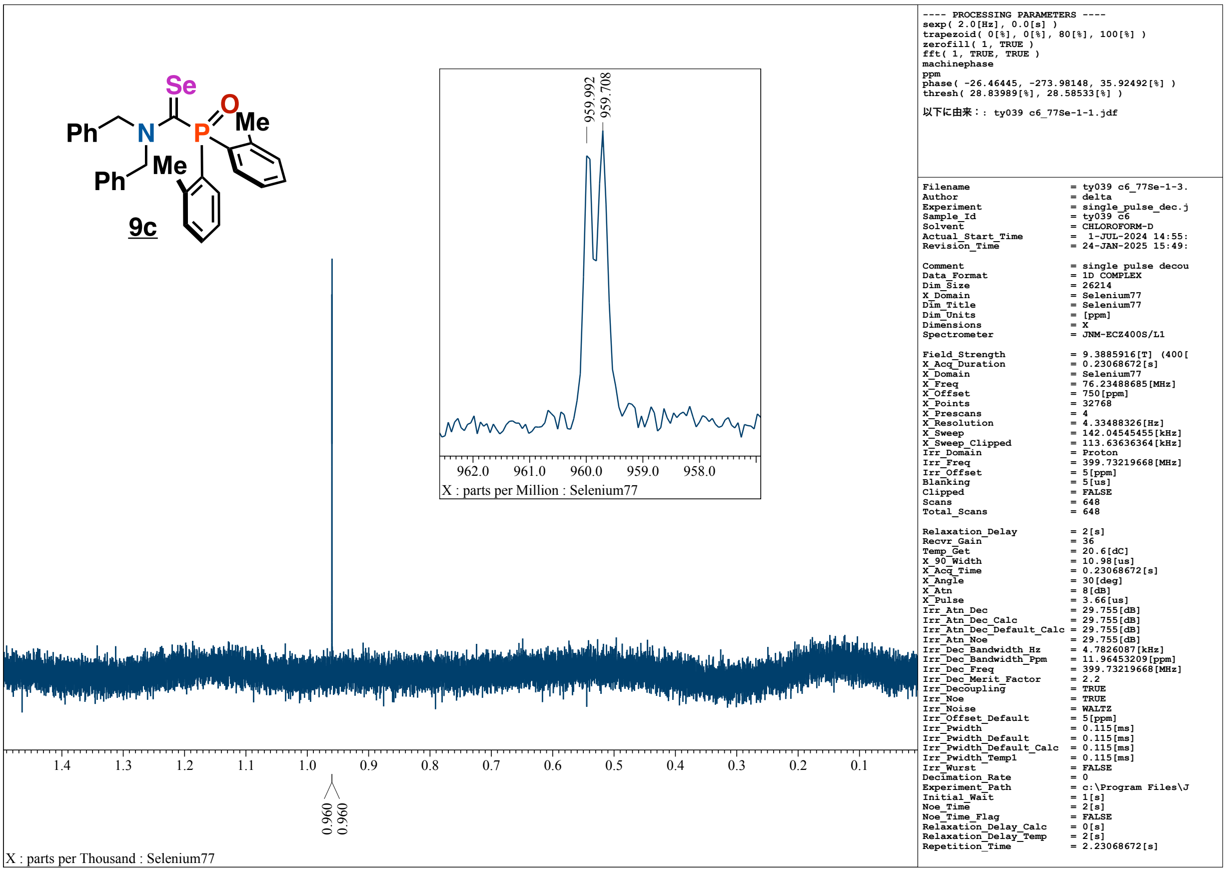
```
Filename                     = ty039 c6_Carbon-1-
Author                       = delta
Experiment                   = single_pulse_dec.j
Sample_Id                    = ty039 c6
Solvent                      = CHLOROFORM-D
Actual_Start_Time            = 23-DEC-2024 18:39:
Revision_Time                = 24-JAN-2025 15:43:

Comment                      = single pulse decou
Data_Format                  = 1D COMPLEX
Dim_Size                     = 26214
X_Domain                     = Carbon13
Dim_Title                    = Carbon13
Dim_Units                    = [ppm]
Dimensions                   = X
Spectrometer                 = JNM-ECZ400S/L1

Field_Strength               = 9.3885916[T] (400[
X_Acq_Duration               = 0.74448896[s]
X_Domain                     = Carbon13
X_Freq                       = 100.51273039[MHz]
X_Offset                     = 150[ppm]
X_Points                     = 32768
X_Prescans                   = 4
X_Resolution                 = 1.34320326[Hz]
X_Sweep                      = 44.01408451[kHz]
X_Sweep_Clipped              = 35.21126761[kHz]
Irr_Domain                   = Proton
Irr_Freq                     = 399.73219668[MHz]
Irr_Offset                   = 5[ppm]
Blanking                     = 5[us]
Clipped                      = TRUE
Scans                        = 512
Total_Scans                  = 512

Relaxation_Delay             = 2[s]
Recvr_Gain                   = 56
Temp_Get                     = 19.9[dC]
X_90_Width                   = 11.03[us]
X_Acq_Time                   = 0.74448896[s]
X_Angle                      = 30[deg]
X_Atn                        = 7[dB]
X_Pulse                      = 3.67666667[us]
Irr_Atn_Dec                  = 29.755[dB]
Irr_Atn_Dec_Calc             = 29.755[dB]
Irr_Atn_Dec_Default_Calc     = 29.755[dB]
Irr_Atn_Noe                  = 29.755[dB]
Irr_Dec_Bandwidth_Hz         = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm        = 11.96453209[ppm]
Irr_Dec_Freq                 = 399.73219668[MHz]
Irr_Dec_Merit_Factor         = 2.2
Irr_Decoupling               = TRUE
Irr_Noe                      = TRUE
Irr_Noise                    = WALTZ
Irr_Offset_Default           = 5[ppm]
Irr_Pwidth                   = 0.115[ms]
Irr_Pwidth_Default           = 0.115[ms]
Irr_Pwidth_Default_Calc      = 0.115[ms]
Irr_Pwidth_Temp1             = 0.115[ms]
Irr_Wurst                    = FALSE
Decimation_Rate              = 0
Experiment_Path              = c:\Program Files\J
Initial_Wait                 = 1[s]
Noe_Time                     = 2[s]
Noe_Time_Flag                = FALSE
Relaxation_Delay_Calc        = 0[s]
Relaxation_Delay_Temp        = 2[s]
Repetition_Time              = 2.74448896[s]
```

Figure S45. ^{13}C{^{1}H} NMR spectrum (100 MHz, CDCl$_3$) of **9c**.



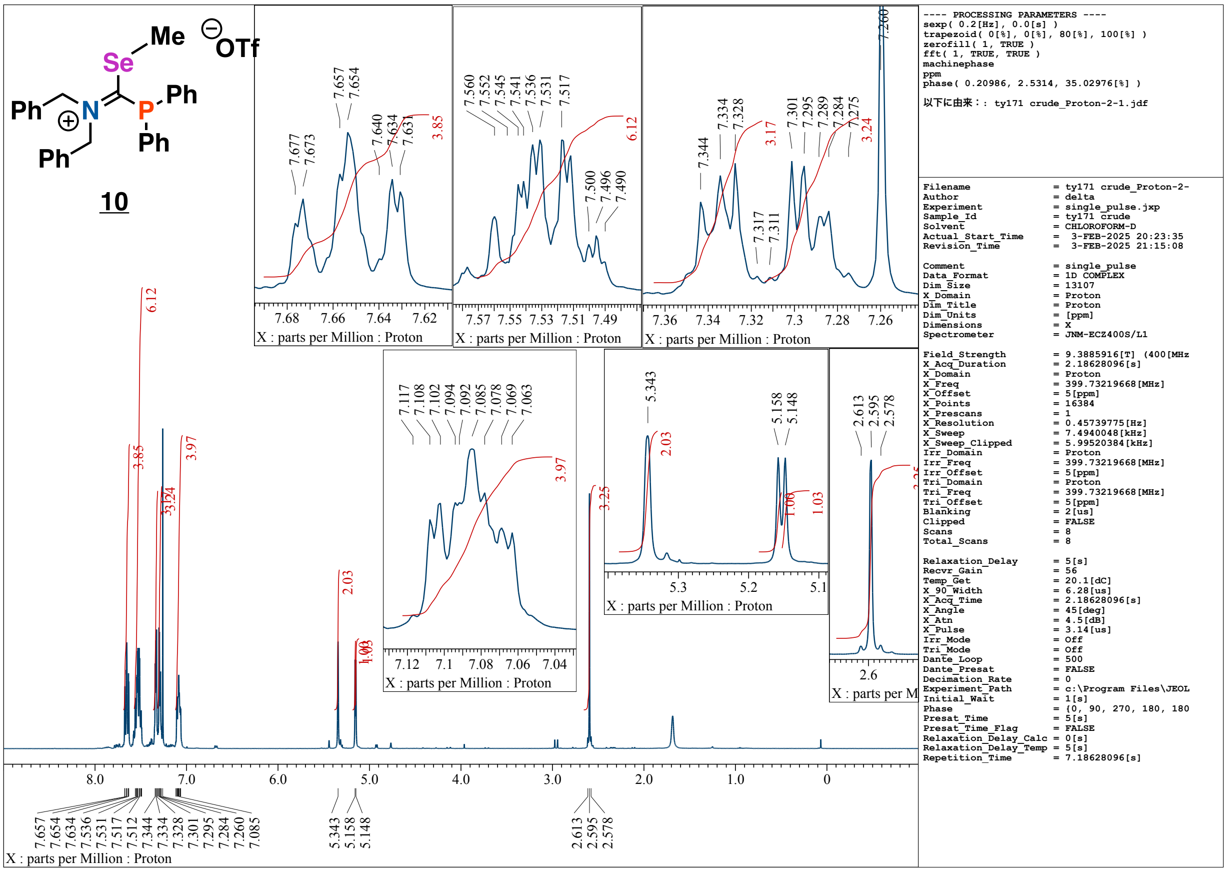
```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( -37.90755, 0, 43.51276[%] )

以下に由来：: ty036 c9_31P-1-1.jdf

Filename                    = ty036 c9_31P-1-3.j
Author                      = delta
Experiment                  = single_pulse_dec.j
Sample_Id                   = ty036 c9
Solvent                     = CHLOROFORM-D
Actual_Start_Time           = 22-JUN-2024 15:46:
Revision_Time               = 24-JAN-2025 15:46:

Comment                     = single pulse decou
Data_Format                 = 1D COMPLEX
Dim_Size                    = 26214
X_Domain                    = Phosphorus31
Dim_Title                   = Phosphorus31
Dim_Units                   = [ppm]
Dimensions                  = X
Spectrometer                = JNM-ECZ400S/L1

Field_Strength              = 9.3885916[T] (400[
X_Acq_Duration              = 0.23068672[s]
X_Domain                    = Phosphorus31
X_Freq                      = 161.81445204[MHz]
X_Offset                    = 0[ppm]
X_Points                    = 32768
X_Prescans                  = 4
X_Resolution                = 4.33488326[Hz]
X_Sweep                     = 142.04545455[kHz]
X_Sweep_Clipped             = 113.63636364[kHz]
Irr_Domain                  = Proton
Irr_Freq                    = 399.73219668[MHz]
Irr_Offset                  = 5[ppm]
Blanking                    = 5[us]
Clipped                     = FALSE
Scans                       = 128
Total_Scans                 = 128

Relaxation_Delay            = 2[s]
Recvr_Gain                  = 56
Temp_Get                    = 21.2[dC]
X_90_Width                  = 14.62[us]
X_Acq_Time                  = 0.23068672[s]
X_Angle                     = 30[deg]
X_Atn                       = 9[dB]
X_Pulse                     = 4.87333333[us]
Irr_Atn_Dec                 = 29.755[dB]
Irr_Atn_Dec_Calc            = 29.755[dB]
Irr_Atn_Dec_Default_Calc = 29.755[dB]
Irr_Atn_Noe                 = 29.755[dB]
Irr_Dec_Bandwidth_Hz        = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm       = 11.96453209[ppm]
Irr_Dec_Freq                = 399.73219668[MHz]
Irr_Dec_Merit_Factor        = 2.2
Irr_Decoupling              = TRUE
Irr_Noe                     = TRUE
Irr_Noise                   = WALTZ
Irr_Offset_Default          = 5[ppm]
Irr_Pwidth                  = 0.115[ms]
Irr_Pwidth_Default          = 0.115[ms]
Irr_Pwidth_Default_Calc     = 0.115[ms]
Irr_Pwidth_Temp1            = 0.115[ms]
Irr_Wurst                   = FALSE
Decimation_Rate             = 0
Experiment_Path             = c:\Program Files\J
Initial_Wait                = 1[s]
Noe_Time                    = 2[s]
Noe_Time_Flag               = FALSE
Relaxation_Delay_Calc       = 0[s]
Relaxation_Delay_Temp       = 2[s]
Repetition_Time             = 2.23068672[s]
```

Figure S46. $^{31}P\{^{1}H\}$ NMR spectrum (126 MHz, $CDCl_3$) of **9c**.



```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( -26.46445, -273.98148, 35.92492[%] )
thresh( 28.83989[%], 28.58533[%] )

以下に由来：: ty039 c6_77Se-1-1.jdf

Filename                    = ty039 c6_77Se-1-3.
Author                      = delta
Experiment                  = single_pulse_dec.j
Sample_Id                   = ty039 c6
Solvent                     = CHLOROFORM-D
Actual_Start_Time           =  1-JUL-2024 14:55:
Revision_Time               = 24-JAN-2025 15:49:

Comment                     = single pulse decou
Data_Format                 = 1D COMPLEX
Dim_Size                    = 26214
X_Domain                    = Selenium77
Dim_Title                   = Selenium77
Dim_Units                   = [ppm]
Dimensions                  = X
Spectrometer                = JNM-ECZ400S/L1

Field_Strength              = 9.3885916[T] (400[
X_Acq_Duration              = 0.23068672[s]
X_Domain                    = Selenium77
X_Freq                      = 76.23488685[MHz]
X_Offset                    = 750[ppm]
X_Points                    = 32768
X_Prescans                  = 4
X_Resolution                = 4.33488326[Hz]
X_Sweep                     = 142.04545455[kHz]
X_Sweep_Clipped             = 113.63636364[kHz]
Irr_Domain                  = Proton
Irr_Freq                    = 399.73219668[MHz]
Irr_Offset                  = 5[ppm]
Blanking                    = 5[us]
Clipped                     = FALSE
Scans                       = 648
Total_Scans                 = 648

Relaxation_Delay            = 2[s]
Recvr_Gain                  = 36
Temp_Get                    = 20.6[dC]
X_90_Width                  = 10.98[us]
X_Acq_Time                  = 0.23068672[s]
X_Angle                     = 30[deg]
X_Atn                       = 8[dB]
X_Pulse                     = 3.66[us]
Irr_Atn_Dec                 = 29.755[dB]
Irr_Atn_Dec_Calc            = 29.755[dB]
Irr_Atn_Dec_Default_Calc = 29.755[dB]
Irr_Atn_Noe                 = 29.755[dB]
Irr_Dec_Bandwidth_Hz        = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm       = 11.96453209[ppm]
Irr_Dec_Freq                = 399.73219668[MHz]
Irr_Dec_Merit_Factor        = 2.2
Irr_Decoupling              = TRUE
Irr_Noe                     = TRUE
Irr_Noise                   = WALTZ
Irr_Offset_Default          = 5[ppm]
Irr_Pwidth                  = 0.115[ms]
Irr_Pwidth_Default          = 0.115[ms]
Irr_Pwidth_Default_Calc     = 0.115[ms]
Irr_Pwidth_Temp1            = 0.115[ms]
Irr_Wurst                   = FALSE
Decimation_Rate             = 0
Experiment_Path             = c:\Program Files\J
Initial_Wait                = 1[s]
Noe_Time                    = 2[s]
Noe_Time_Flag               = FALSE
Relaxation_Delay_Calc       = 0[s]
Relaxation_Delay_Temp       = 2[s]
Repetition_Time             = 2.23068672[s]
```

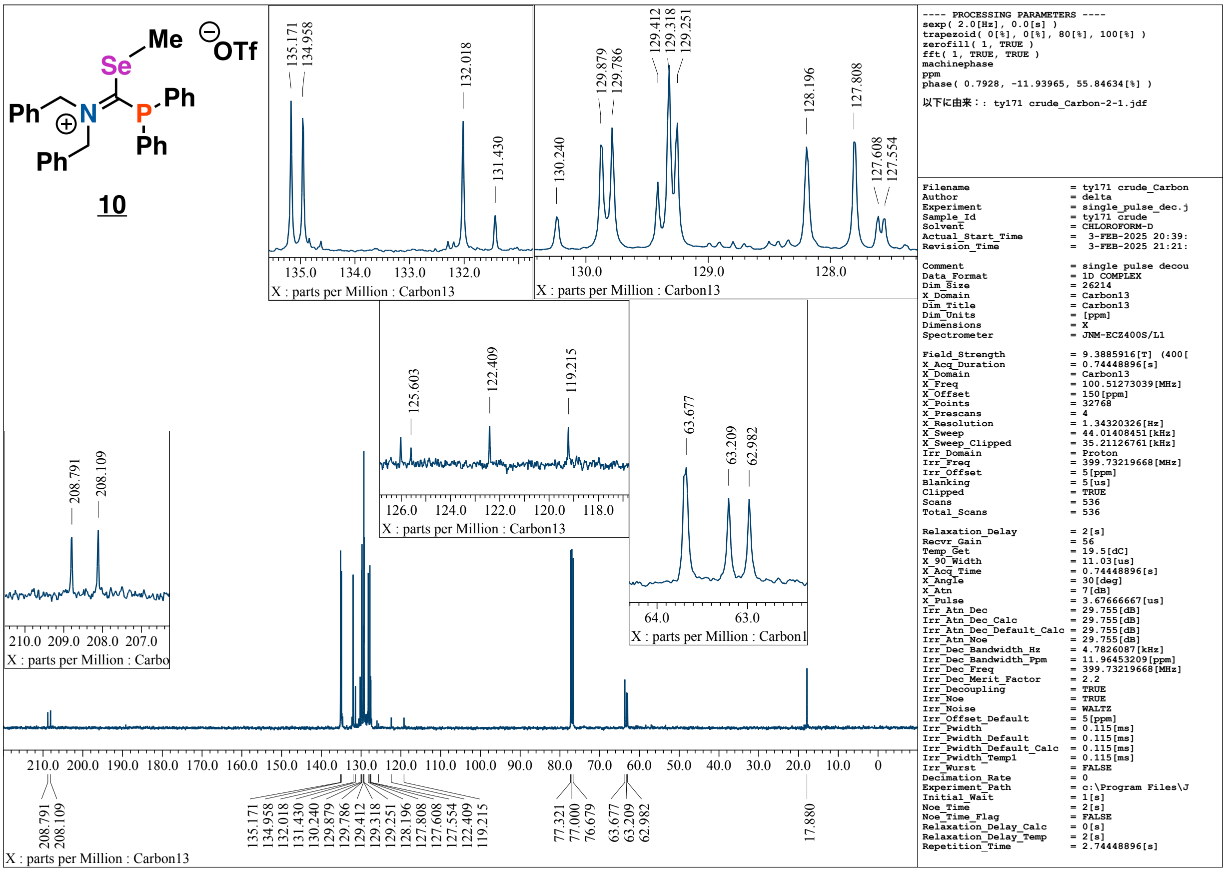
Figure S47. $^{77}Se\{^{1}H\}$ NMR spectrum (76 MHz, $CDCl_3$) of **9c**.



```
---- PROCESSING PARAMETERS ----
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( 0.20986, 2.5314, 35.02976[%] )

以下に由来：: ty171 crude_Proton-2-1.jdf
```

```
Filename                = ty171 crude_Proton-2-
Author                  = delta
Experiment              = single_pulse.jxp
Sample_Id               = ty171 crude
Solvent                 = CHLOROFORM-D
Actual_Start_Time       =  3-FEB-2025 20:23:35
Revision_Time           =  3-FEB-2025 21:15:08

Comment                 = single_pulse
Data_Format             = 1D COMPLEX
Dim_Size                = 13107
X_Domain                = Proton
Dim_Title               = Proton
Dim_Units               = [ppm]
Dimensions              = X
Spectrometer            = JNM-ECZ400S/L1

Field_Strength          = 9.3885916[T] (400[MHz
X_Acq_Duration          = 2.18628096[s]
X_Domain                = Proton
X_Freq                  = 399.73219668[MHz]
X_Offset                = 5[ppm]
X_Points                = 16384
X_Prescans              = 1
X_Resolution            = 0.45739775[Hz]
X_Sweep                 = 7.4940048[kHz]
X_Sweep_Clipped         = 5.99520384[kHz]
Irr_Domain              = Proton
Irr_Freq                = 399.73219668[MHz]
Irr_Offset              = 5[ppm]
Tri_Domain              = Proton
Tri_Freq                = 399.73219668[MHz]
Tri_Offset              = 5[ppm]
Blanking                = 2[us]
Clipped                 = FALSE
Scans                   = 8
Total_Scans             = 8

Relaxation_Delay        = 5[s]
Recvr_Gain              = 56
Temp_Get                = 20.1[dC]
X_90_Width              = 6.28[us]
X_Acq_Time              = 2.18628096[s]
X_Angle                 = 45[deg]
X_Atn                   = 4.5[dB]
X_Pulse                 = 3.14[us]
Irr_Mode                = Off
Tri_Mode                = Off
Dante_Loop              = 500
Dante_Presat            = FALSE
Decimation_Rate         = 0
Experiment_Path         = c:\Program Files\JEOL
Initial_Wait            = 1[s]
Phase                   = {0, 90, 270, 180, 180
Presat_Time             = 5[s]
Presat_Time_Flag        = FALSE
Relaxation_Delay_Calc   = 0[s]
Relaxation_Delay_Temp   = 5[s]
Repetition_Time         = 7.18628096[s]
```

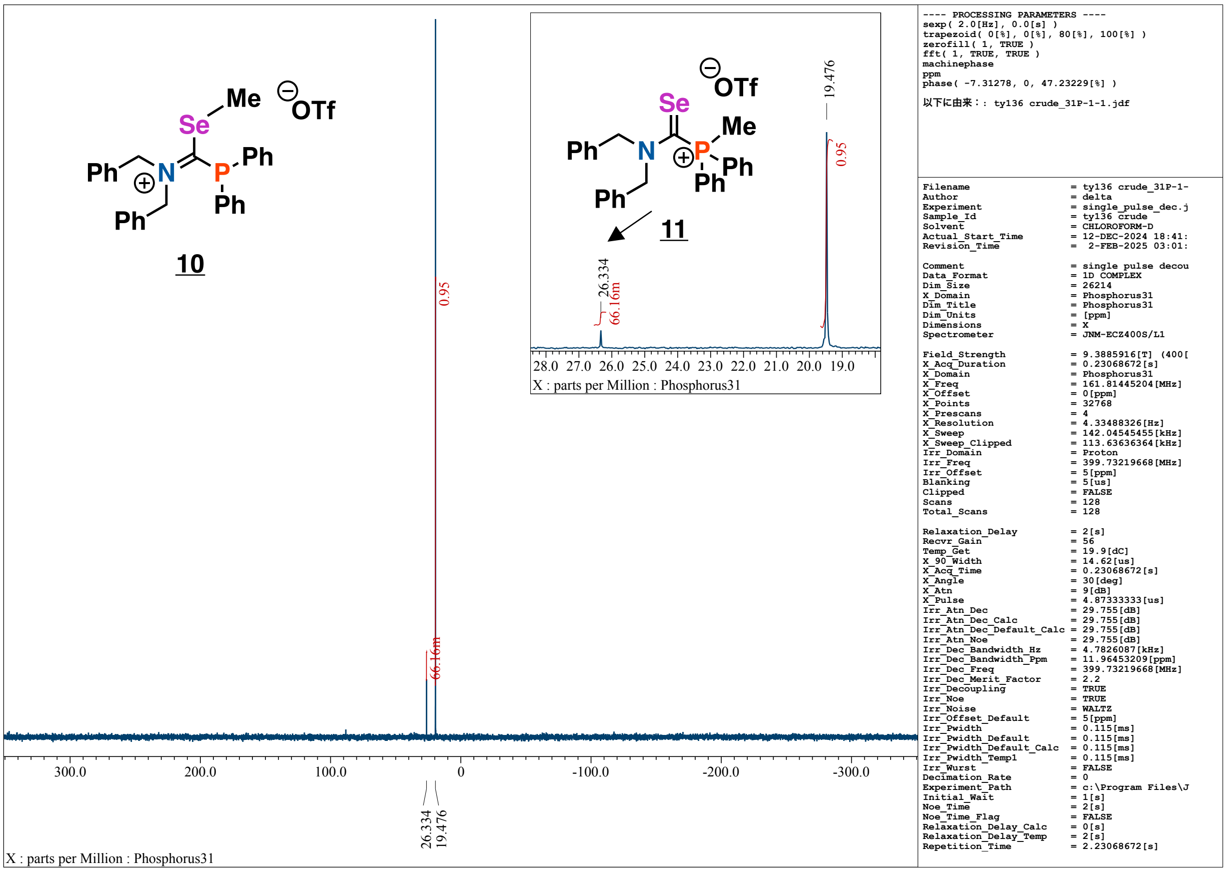
Figure S48. ^{1}H NMR spectrum (400 MHz, CDCl$_3$) of **10**.



```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( 0.7928, -11.93965, 55.84634[%] )

以下に由来：: ty171 crude_Carbon-2-1.jdf
```

```
Filename                   = ty171 crude_Carbon
Author                     = delta
Experiment                 = single_pulse_dec.j
Sample_Id                  = ty171 crude
Solvent                    = CHLOROFORM-D
Actual_Start_Time          =  3-FEB-2025 20:39:
Revision_Time              =  3-FEB-2025 21:21:

Comment                    = single pulse decou
Data_Format                = 1D COMPLEX
Dim_Size                   = 26214
X_Domain                   = Carbon13
Dim_Title                  = Carbon13
Dim_Units                  = [ppm]
Dimensions                 = X
Spectrometer               = JNM-ECZ400S/L1

Field_Strength             = 9.3885916[T] (400[
X_Acq_Duration             = 0.74448896[s]
X_Domain                   = Carbon13
X_Freq                     = 100.51273039[MHz]
X_Offset                   = 150[ppm]
X_Points                   = 32768
X_Prescans                 = 4
X_Resolution               = 1.34320326[Hz]
X_Sweep                    = 44.01408451[kHz]
X_Sweep_Clipped            = 35.21126761[kHz]
Irr_Domain                 = Proton
Irr_Freq                   = 399.73219668[MHz]
Irr_Offset                 = 5[ppm]
Blanking                   = 5[us]
Clipped                    = TRUE
Scans                      = 536
Total_Scans                = 536

Relaxation_Delay           = 2[s]
Recvr_Gain                 = 56
Temp_Get                   = 19.5[dC]
X_90_Width                 = 11.03[us]
X_Acq_Time                 = 0.74448896[s]
X_Angle                    = 30[deg]
X_Atn                      = 7[dB]
X_Pulse                    = 3.67666667[us]
Irr_Atn_Dec                = 29.755[dB]
Irr_Atn_Dec_Calc           = 29.755[dB]
Irr_Atn_Dec_Default_Calc = 29.755[dB]
Irr_Atn_Noe                = 29.755[dB]
Irr_Dec_Bandwidth_Hz       = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm      = 11.96453209[ppm]
Irr_Dec_Freq               = 399.73219668[MHz]
Irr_Dec_Merit_Factor       = 2.2
Irr_Decoupling             = TRUE
Irr_Noe                    = TRUE
Irr_Noise                  = WALTZ
Irr_Offset_Default         = 5[ppm]
Irr_Pwidth                 = 0.115[ms]
Irr_Pwidth_Default         = 0.115[ms]
Irr_Pwidth_Default_Calc    = 0.115[ms]
Irr_Pwidth_Temp1           = 0.115[ms]
Irr_Wurst                  = FALSE
Decimation_Rate            = 0
Experiment_Path            = c:\Program Files\J
Initial_Wait               = 1[s]
Noe_Time                   = 2[s]
Noe_Time_Flag              = FALSE
Relaxation_Delay_Calc      = 0[s]
Relaxation_Delay_Temp      = 2[s]
Repetition_Time            = 2.74448896[s]
```

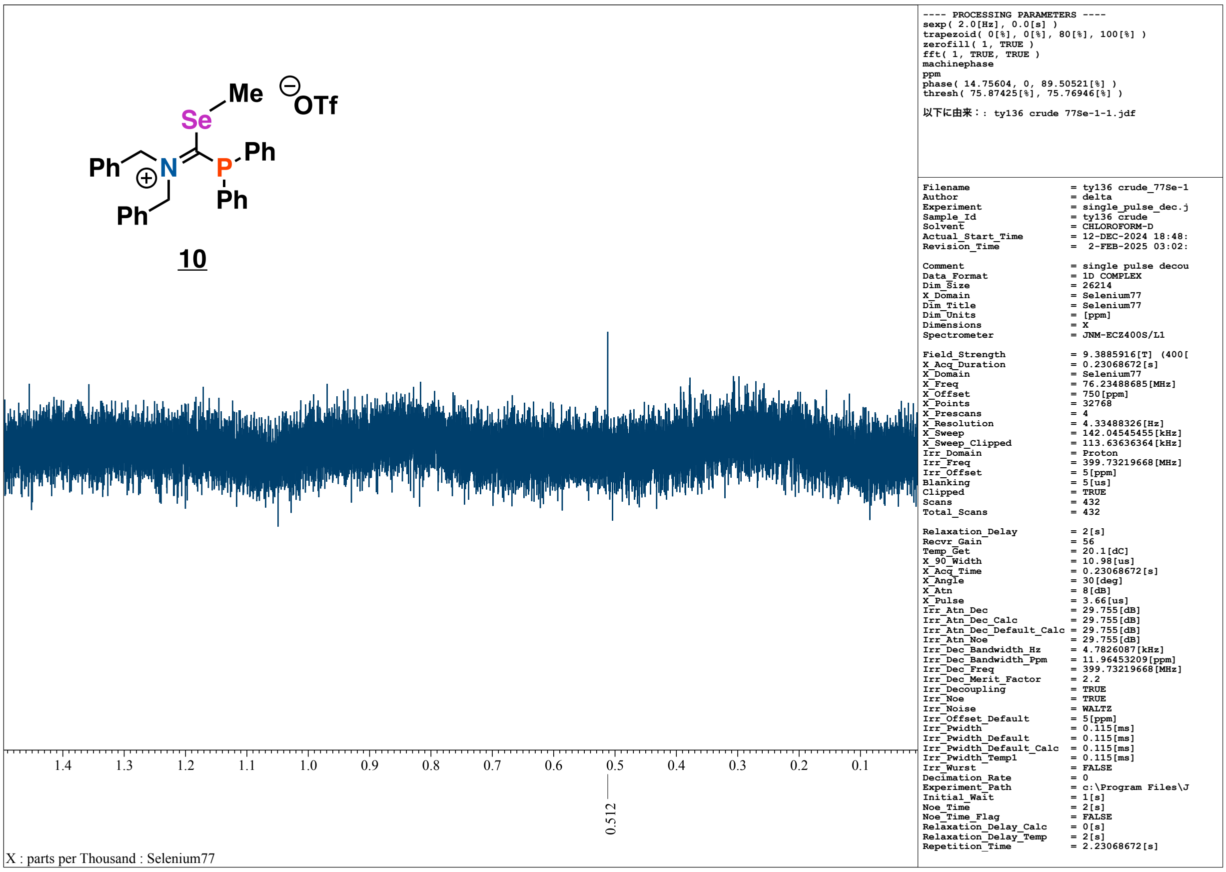
Figure S49. ^{13}C{^{1}H} NMR spectrum (100 MHz, CDCl$_3$) of **10**.



```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( -7.31278, 0, 47.23229[%] )

以下に由来：: ty136 crude_31P-1-1.jdf
```

```
Filename                   = ty136 crude_31P-1-
Author                     = delta
Experiment                 = single_pulse_dec.j
Sample_Id                  = ty136 crude
Solvent                    = CHLOROFORM-D
Actual_Start_Time          = 12-DEC-2024 18:41:
Revision_Time              =  2-FEB-2025 03:01:

Comment                    = single pulse decou
Data_Format                = 1D COMPLEX
Dim_Size                   = 26214
X_Domain                   = Phosphorus31
Dim_Title                  = Phosphorus31
Dim_Units                  = [ppm]
Dimensions                 = X
Spectrometer               = JNM-ECZ400S/L1

Field_Strength             = 9.3885916[T] (400[
X_Acq_Duration             = 0.23068672[s]
X_Domain                   = Phosphorus31
X_Freq                     = 161.81445204[MHz]
X_Offset                   = 0[ppm]
X_Points                   = 32768
X_Prescans                 = 4
X_Resolution               = 4.33488326[Hz]
X_Sweep                    = 142.04545455[kHz]
X_Sweep_Clipped            = 113.63636364[kHz]
Irr_Domain                 = Proton
Irr_Freq                   = 399.73219668[MHz]
Irr_Offset                 = 5[ppm]
Blanking                   = 5[us]
Clipped                    = FALSE
Scans                      = 128
Total_Scans                = 128

Relaxation_Delay           = 2[s]
Recvr_Gain                 = 56
Temp_Get                   = 19.9[dC]
X_90_Width                 = 14.62[us]
X_Acq_Time                 = 0.23068672[s]
X_Angle                    = 30[deg]
X_Atn                      = 9[dB]
X_Pulse                    = 4.87333333[us]
Irr_Atn_Dec                = 29.755[dB]
Irr_Atn_Dec_Calc           = 29.755[dB]
Irr_Atn_Dec_Default_Calc = 29.755[dB]
Irr_Atn_Noe                = 29.755[dB]
Irr_Dec_Bandwidth_Hz       = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm      = 11.96453209[ppm]
Irr_Dec_Freq               = 399.73219668[MHz]
Irr_Dec_Merit_Factor       = 2.2
Irr_Decoupling             = TRUE
Irr_Noe                    = TRUE
Irr_Noise                  = WALTZ
Irr_Offset_Default         = 5[ppm]
Irr_Pwidth                 = 0.115[ms]
Irr_Pwidth_Default         = 0.115[ms]
Irr_Pwidth_Default_Calc    = 0.115[ms]
Irr_Pwidth_Temp1           = 0.115[ms]
Irr_Wurst                  = FALSE
Decimation_Rate            = 0
Experiment_Path            = c:\Program Files\J
Initial_Wait               = 1[s]
Noe_Time                   = 2[s]
Noe_Time_Flag              = FALSE
Relaxation_Delay_Calc      = 0[s]
Relaxation_Delay_Temp      = 2[s]
Repetition_Time            = 2.23068672[s]
```

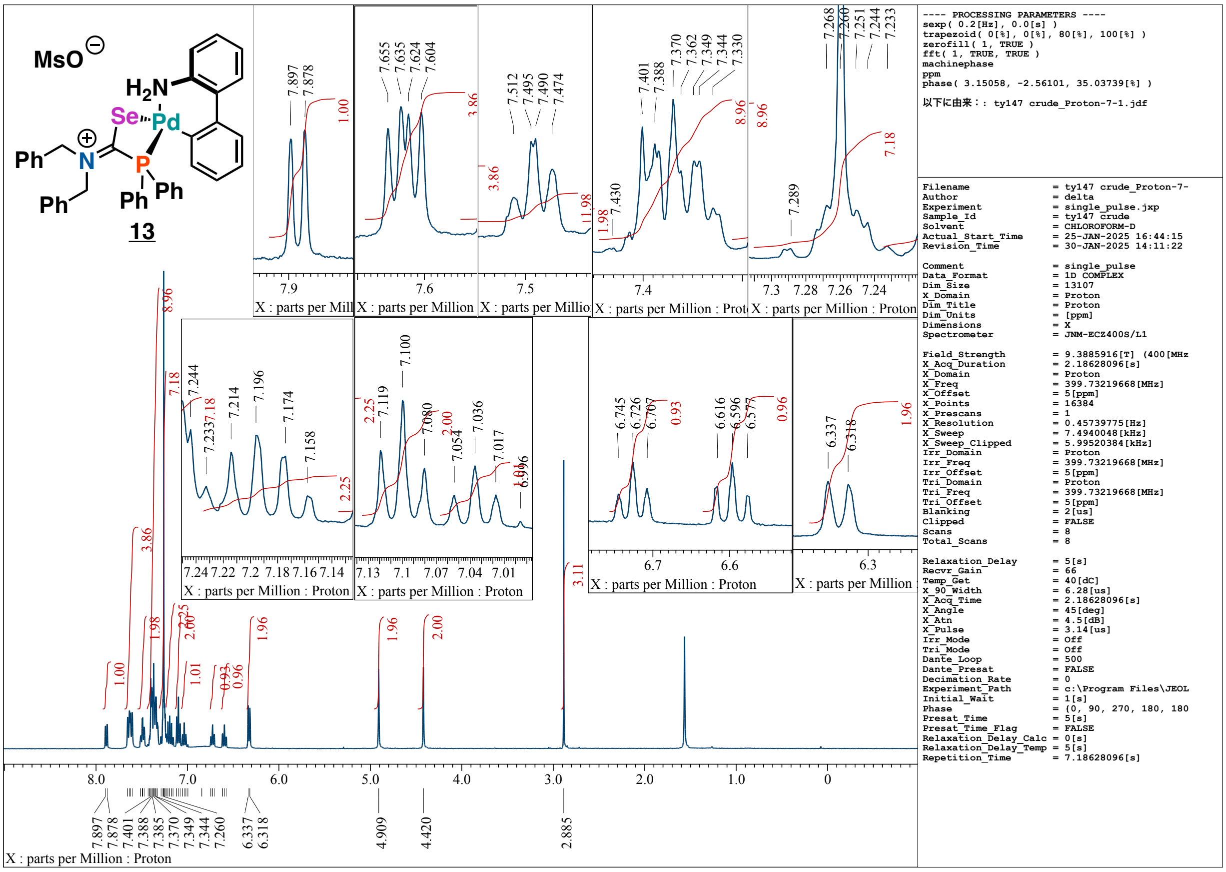
Figure S50. $^{31}P\{^{1}H\}$ NMR spectrum (126 MHz, $CDCl_3$) of **10**.



```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( 14.75604, 0, 89.50521[%] )
thresh( 75.87425[%], 75.76946[%] )

以下に由来：: ty136 crude 77Se-1-1.jdf
```

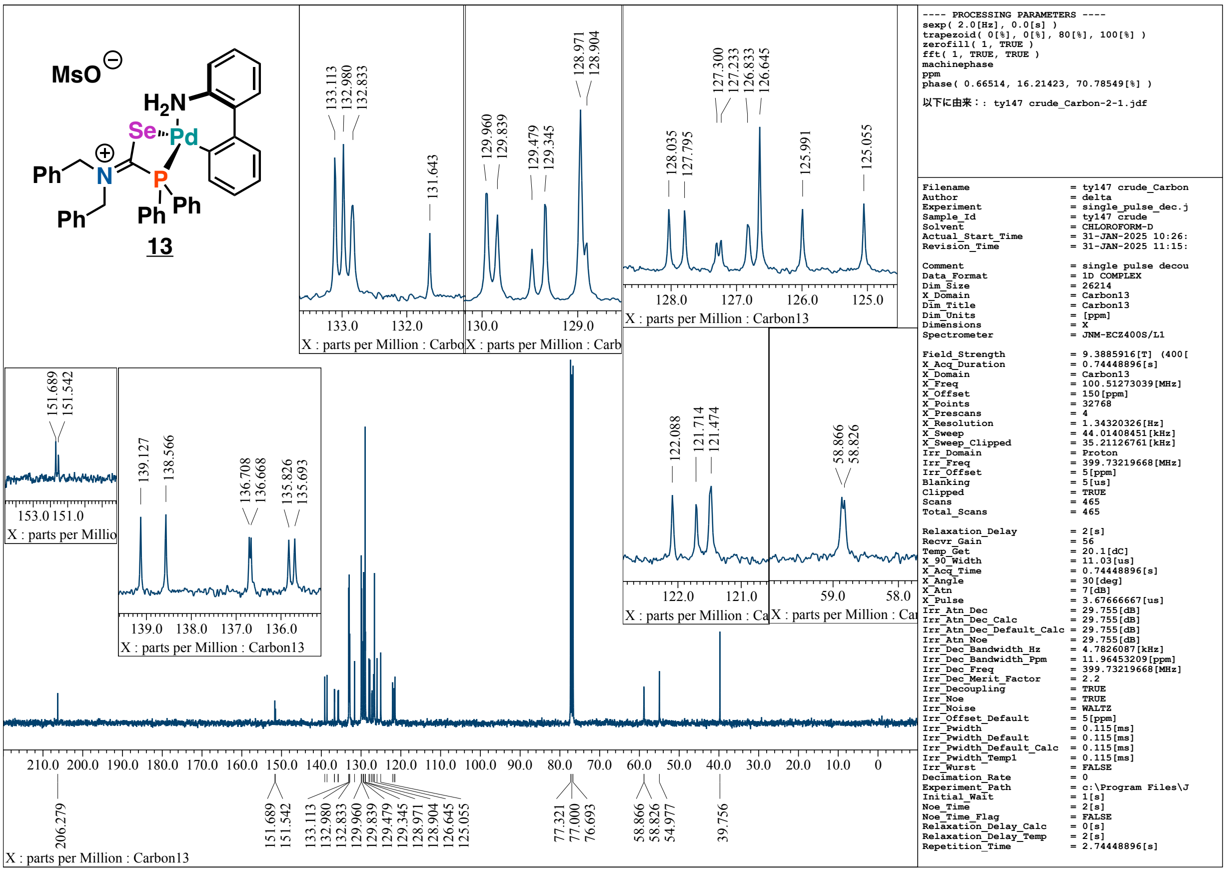
```
Filename                   = ty136 crude_77Se-1
Author                     = delta
Experiment                 = single_pulse_dec.j
Sample_Id                  = ty136 crude
Solvent                    = CHLOROFORM-D
Actual_Start_Time          = 12-DEC-2024 18:48:
Revision_Time              =  2-FEB-2025 03:02:

Comment                    = single pulse decou
Data_Format                = 1D COMPLEX
Dim_Size                   = 26214
X_Domain                   = Selenium77
Dim_Title                  = Selenium77
Dim_Units                  = [ppm]
Dimensions                 = X
Spectrometer               = JNM-ECZ400S/L1

Field_Strength             = 9.3885916[T] (400[
X_Acq_Duration             = 0.23068672[s]
X_Domain                   = Selenium77
X_Freq                     = 76.23488685[MHz]
X_Offset                   = 750[ppm]
X_Points                   = 32768
X_Prescans                 = 4
X_Resolution               = 4.33488326[Hz]
X_Sweep                    = 142.04545455[kHz]
X_Sweep_Clipped            = 113.63636364[kHz]
Irr_Domain                 = Proton
Irr_Freq                   = 399.73219668[MHz]
Irr_Offset                 = 5[ppm]
Blanking                   = 5[us]
Clipped                    = TRUE
Scans                      = 432
Total_Scans                = 432

Relaxation_Delay           = 2[s]
Recvr_Gain                 = 56
Temp_Get                   = 20.1[dC]
X_90_Width                 = 10.98[us]
X_Acq_Time                 = 0.23068672[s]
X_Angle                    = 30[deg]
X_Atn                      = 8[dB]
X_Pulse                    = 3.66[us]
Irr_Atn_Dec                = 29.755[dB]
Irr_Atn_Dec_Calc           = 29.755[dB]
Irr_Atn_Dec_Default_Calc = 29.755[dB]
Irr_Atn_Noe                = 29.755[dB]
Irr_Dec_Bandwidth_Hz       = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm      = 11.96453209[ppm]
Irr_Dec_Freq               = 399.73219668[MHz]
Irr_Dec_Merit_Factor       = 2.2
Irr_Decoupling             = TRUE
Irr_Noe                    = TRUE
Irr_Noise                  = WALTZ
Irr_Offset_Default         = 5[ppm]
Irr_Pwidth                 = 0.115[ms]
Irr_Pwidth_Default         = 0.115[ms]
Irr_Pwidth_Default_Calc    = 0.115[ms]
Irr_Pwidth_Temp1           = 0.115[ms]
Irr_Wurst                  = FALSE
Decimation_Rate            = 0
Experiment_Path            = c:\Program Files\J
Initial_Wait               = 1[s]
Noe_Time                   = 2[s]
Noe_Time_Flag              = FALSE
Relaxation_Delay_Calc      = 0[s]
Relaxation_Delay_Temp      = 2[s]
Repetition_Time            = 2.23068672[s]
```

Figure S51. $^{77}Se\{^{1}H\}$ NMR spectrum (76 MHz, $CDCl_3$) of **10**.



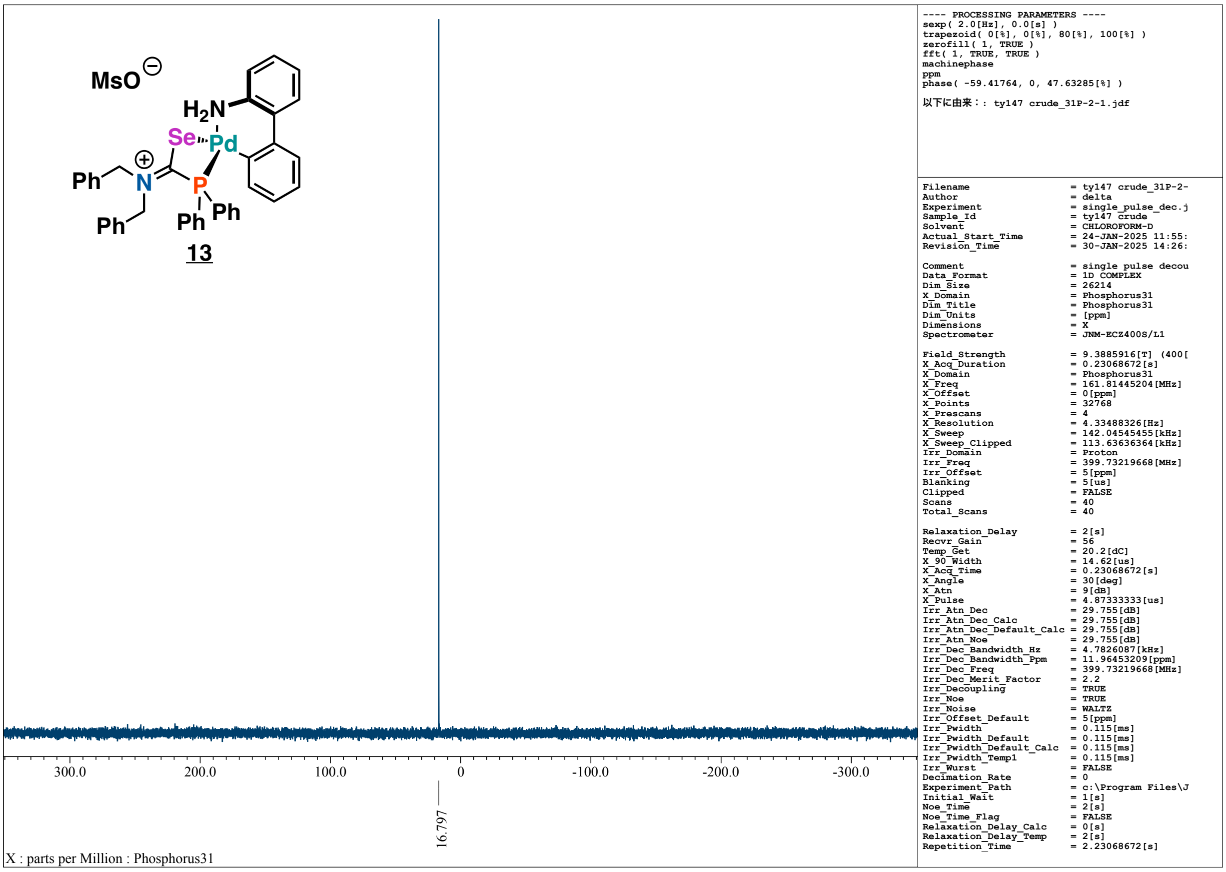
```
---- PROCESSING PARAMETERS ----
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( 3.15058, -2.56101, 35.03739[%] )

以下に由来：: ty147 crude_Proton-7-1.jdf

Filename                = ty147 crude_Proton-7-
Author                  = delta
Experiment              = single_pulse.jxp
Sample_Id               = ty147 crude
Solvent                 = CHLOROFORM-D
Actual_Start_Time       = 25-JAN-2025 16:44:15
Revision_Time           = 30-JAN-2025 14:11:22

Comment                 = single_pulse
Data_Format             = 1D COMPLEX
Dim_Size                = 13107
X_Domain                = Proton
Dim_Title               = Proton
Dim_Units               = [ppm]
Dimensions              = X
Spectrometer            = JNM-ECZ400S/L1

Field_Strength          = 9.3885916[T] (400[MHz
X_Acq_Duration          = 2.18628096[s]
X_Domain                = Proton
X_Freq                  = 399.73219668[MHz]
X_Offset                = 5[ppm]
X_Points                = 16384
X_Prescans              = 1
X_Resolution            = 0.45739775[Hz]
X_Sweep                 = 7.4940048[kHz]
X_Sweep_Clipped         = 5.99520384[kHz]
Irr_Domain              = Proton
Irr_Freq                = 399.73219668[MHz]
Irr_Offset              = 5[ppm]
Tri_Domain              = Proton
Tri_Freq                = 399.73219668[MHz]
Tri_Offset              = 5[ppm]
Blanking                = 2[us]
Clipped                 = FALSE
Scans                   = 8
Total_Scans             = 8

Relaxation_Delay        = 5[s]
Recvr_Gain              = 66
Temp_Get                = 40[dC]
X_90_Width              = 6.28[us]
X_Acq_Time              = 2.18628096[s]
X_Angle                 = 45[deg]
X_Atn                   = 4.5[dB]
X_Pulse                 = 3.14[us]
Irr_Mode                = Off
Tri_Mode                = Off
Dante_Loop              = 500
Dante_Presat            = FALSE
Decimation_Rate         = 0
Experiment_Path         = c:\Program Files\JEOL
Initial_Wait            = 1[s]
Phase                   = {0, 90, 270, 180, 180
Presat_Time             = 5[s]
Presat_Time_Flag        = FALSE
Relaxation_Delay_Calc   = 0[s]
Relaxation_Delay_Temp   = 5[s]
Repetition_Time         = 7.18628096[s]
```

Figure S52. ^{1}H NMR spectrum (400 MHz, $CDCl_3$, 40 °C) of **13**.



```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( 0.66514, 16.21423, 70.78549[%] )

以下に由来：: ty147 crude_Carbon-2-1.jdf

Filename                    = ty147 crude_Carbon
Author                      = delta
Experiment                  = single_pulse_dec.j
Sample_Id                   = ty147 crude
Solvent                     = CHLOROFORM-D
Actual_Start_Time           = 31-JAN-2025 10:26:
Revision_Time               = 31-JAN-2025 11:15:

Comment                     = single pulse decou
Data_Format                 = 1D COMPLEX
Dim_Size                    = 26214
X_Domain                    = Carbon13
Dim_Title                   = Carbon13
Dim_Units                   = [ppm]
Dimensions                  = X
Spectrometer                = JNM-ECZ400S/L1

Field_Strength              = 9.3885916[T] (400[
X_Acq_Duration              = 0.74448896[s]
X_Domain                    = Carbon13
X_Freq                      = 100.51273039[MHz]
X_Offset                    = 150[ppm]
X_Points                    = 32768
X_Prescans                  = 4
X_Resolution                = 1.34320326[Hz]
X_Sweep                     = 44.01408451[kHz]
X_Sweep_Clipped             = 35.21126761[kHz]
Irr_Domain                  = Proton
Irr_Freq                    = 399.73219668[MHz]
Irr_Offset                  = 5[ppm]
Blanking                    = 5[us]
Clipped                     = TRUE
Scans                       = 465
Total_Scans                 = 465

Relaxation_Delay            = 2[s]
Recvr_Gain                  = 56
Temp_Get                    = 20.1[dC]
X_90_Width                  = 11.03[us]
X_Acq_Time                  = 0.74448896[s]
X_Angle                     = 30[deg]
X_Atn                       = 7[dB]
X_Pulse                     = 3.67666667[us]
Irr_Atn_Dec                 = 29.755[dB]
Irr_Atn_Dec_Calc            = 29.755[dB]
Irr_Atn_Dec_Default_Calc    = 29.755[dB]
Irr_Atn_Noe                 = 29.755[dB]
Irr_Dec_Bandwidth_Hz        = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm       = 11.96453209[ppm]
Irr_Dec_Freq                = 399.73219668[MHz]
Irr_Dec_Merit_Factor        = 2.2
Irr_Decoupling              = TRUE
Irr_Noe                     = TRUE
Irr_Noise                   = WALTZ
Irr_Offset_Default          = 5[ppm]
Irr_Pwidth                  = 0.115[ms]
Irr_Pwidth_Default          = 0.115[ms]
Irr_Pwidth_Default_Calc     = 0.115[ms]
Irr_Pwidth_Temp1            = 0.115[ms]
Irr_Wurst                   = FALSE
Decimation_Rate             = 0
Experiment_Path             = c:\Program Files\J
Initial_Wait                = 1[s]
Noe_Time                    = 2[s]
Noe_Time_Flag               = FALSE
Relaxation_Delay_Calc       = 0[s]
Relaxation_Delay_Temp       = 2[s]
Repetition_Time             = 2.74448896[s]
```

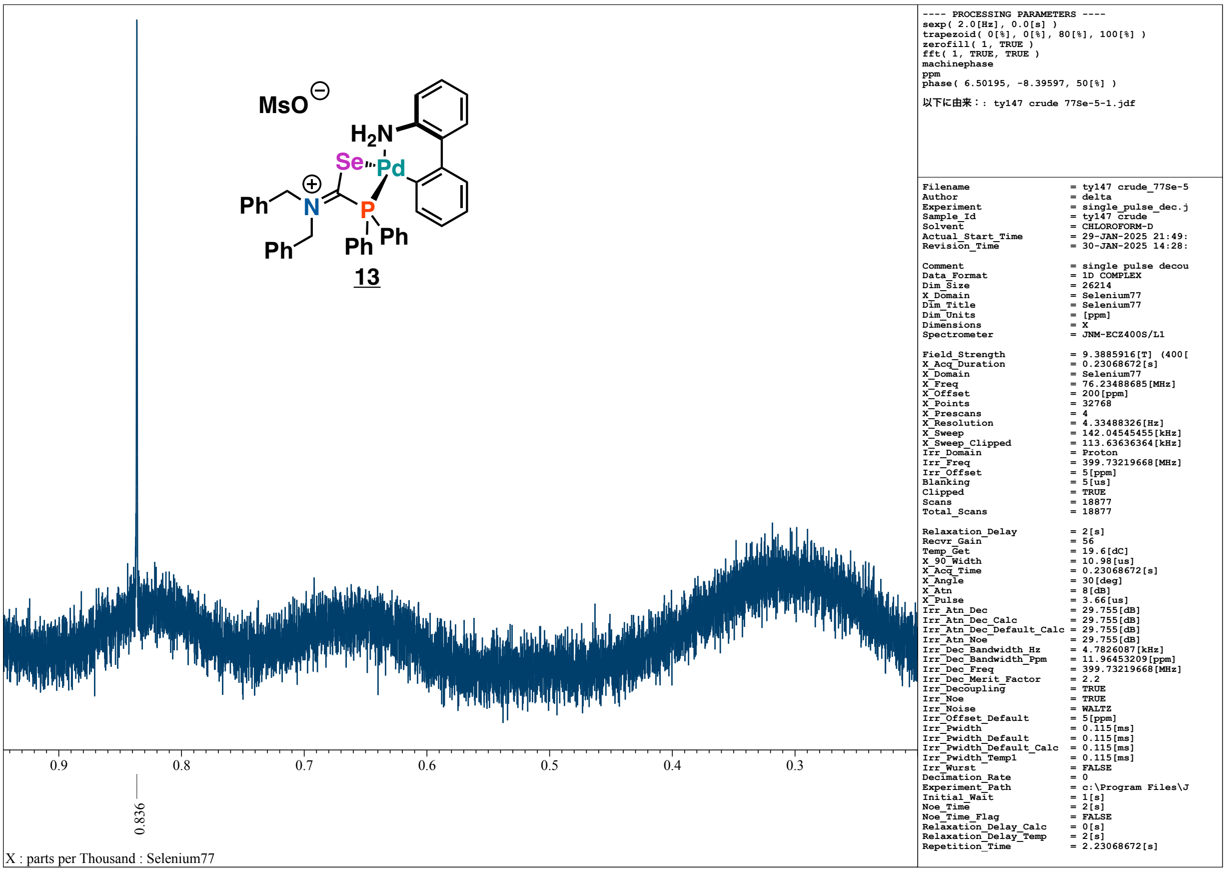
Figure S53. $^{13}C\{^{1}H\}$ NMR spectrum (100 MHz, $CDCl_3$) of **13**.



```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( -59.41764, 0, 47.63285[%] )

以下に由来：: ty147 crude_31P-2-1.jdf
```

```
Filename                   = ty147 crude_31P-2-
Author                     = delta
Experiment                 = single_pulse_dec.j
Sample_Id                  = ty147 crude
Solvent                    = CHLOROFORM-D
Actual_Start_Time          = 24-JAN-2025 11:55:
Revision_Time              = 30-JAN-2025 14:26:

Comment                    = single pulse decou
Data_Format                = 1D COMPLEX
Dim_Size                   = 26214
X_Domain                   = Phosphorus31
Dim_Title                  = Phosphorus31
Dim_Units                  = [ppm]
Dimensions                 = X
Spectrometer               = JNM-ECZ400S/L1

Field_Strength             = 9.3885916[T] (400[
X_Acq_Duration             = 0.23068672[s]
X_Domain                   = Phosphorus31
X_Freq                     = 161.81445204[MHz]
X_Offset                   = 0[ppm]
X_Points                   = 32768
X_Prescans                 = 4
X_Resolution               = 4.33488326[Hz]
X_Sweep                    = 142.04545455[kHz]
X_Sweep_Clipped            = 113.63636364[kHz]
Irr_Domain                 = Proton
Irr_Freq                   = 399.73219668[MHz]
Irr_Offset                 = 5[ppm]
Blanking                   = 5[us]
Clipped                    = FALSE
Scans                      = 40
Total_Scans                = 40

Relaxation_Delay           = 2[s]
Recvr_Gain                 = 56
Temp_Get                   = 20.2[dC]
X_90_Width                 = 14.62[us]
X_Acq_Time                 = 0.23068672[s]
X_Angle                    = 30[deg]
X_Atn                      = 9[dB]
X_Pulse                    = 4.87333333[us]
Irr_Atn_Dec                = 29.755[dB]
Irr_Atn_Dec_Calc           = 29.755[dB]
Irr_Atn_Dec_Default_Calc = 29.755[dB]
Irr_Atn_Noe                = 29.755[dB]
Irr_Dec_Bandwidth_Hz       = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm      = 11.96453209[ppm]
Irr_Dec_Freq               = 399.73219668[MHz]
Irr_Dec_Merit_Factor       = 2.2
Irr_Decoupling             = TRUE
Irr_Noe                    = TRUE
Irr_Noise                  = WALTZ
Irr_Offset_Default         = 5[ppm]
Irr_Pwidth                 = 0.115[ms]
Irr_Pwidth_Default         = 0.115[ms]
Irr_Pwidth_Default_Calc    = 0.115[ms]
Irr_Pwidth_Temp1           = 0.115[ms]
Irr_Wurst                  = FALSE
Decimation_Rate            = 0
Experiment_Path            = c:\Program Files\J
Initial_Wait               = 1[s]
Noe_Time                   = 2[s]
Noe_Time_Flag              = FALSE
Relaxation_Delay_Calc      = 0[s]
Relaxation_Delay_Temp      = 2[s]
Repetition_Time            = 2.23068672[s]
```

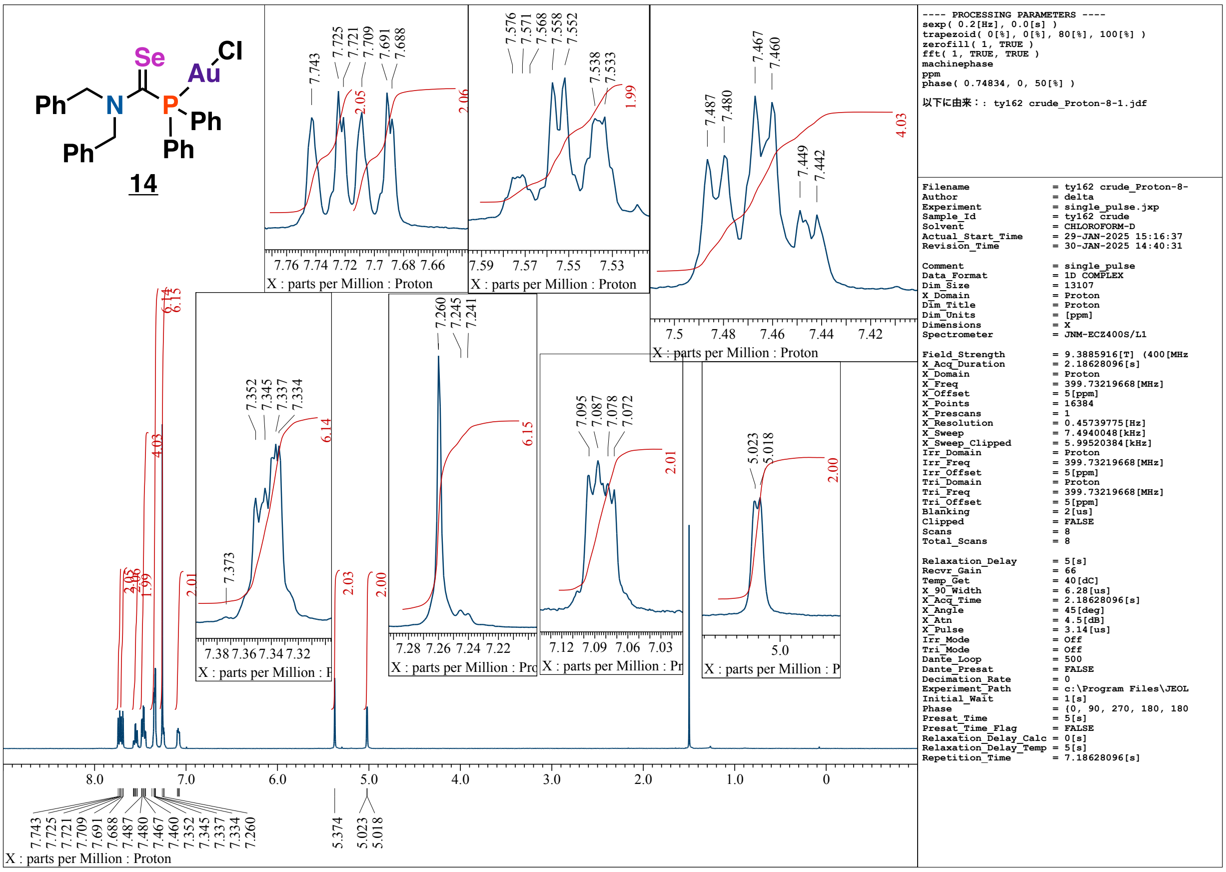
Figure S54. $^{31}P\{^1H\}$ NMR spectrum (126 MHz, $CDCl_3$) of **13**.



```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( 6.50195, -8.39597, 50[%] )

以下に由来：: ty147 crude 77Se-5-1.jdf
```

```
Filename                   = ty147 crude_77Se-5
Author                     = delta
Experiment                 = single_pulse_dec.j
Sample_Id                  = ty147 crude
Solvent                    = CHLOROFORM-D
Actual_Start_Time          = 29-JAN-2025 21:49:
Revision_Time              = 30-JAN-2025 14:28:

Comment                    = single pulse decou
Data_Format                = 1D COMPLEX
Dim_Size                   = 26214
X_Domain                   = Selenium77
Dim_Title                  = Selenium77
Dim_Units                  = [ppm]
Dimensions                 = X
Spectrometer               = JNM-ECZ400S/L1

Field_Strength             = 9.3885916[T] (400[
X_Acq_Duration             = 0.23068672[s]
X_Domain                   = Selenium77
X_Freq                     = 76.23488685[MHz]
X_Offset                   = 200[ppm]
X_Points                   = 32768
X_Prescans                 = 4
X_Resolution               = 4.33488326[Hz]
X_Sweep                    = 142.04545455[kHz]
X_Sweep_Clipped            = 113.63636364[kHz]
Irr_Domain                 = Proton
Irr_Freq                   = 399.73219668[MHz]
Irr_Offset                 = 5[ppm]
Blanking                   = 5[us]
Clipped                    = TRUE
Scans                      = 18877
Total_Scans                = 18877

Relaxation_Delay           = 2[s]
Recvr_Gain                 = 56
Temp_Get                   = 19.6[dC]
X_90_Width                 = 10.98[us]
X_Acq_Time                 = 0.23068672[s]
X_Angle                    = 30[deg]
X_Atn                      = 8[dB]
X_Pulse                    = 3.66[us]
Irr_Atn_Dec                = 29.755[dB]
Irr_Atn_Dec_Calc           = 29.755[dB]
Irr_Atn_Dec_Default_Calc = 29.755[dB]
Irr_Atn_Noe                = 29.755[dB]
Irr_Dec_Bandwidth_Hz       = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm      = 11.96453209[ppm]
Irr_Dec_Freq               = 399.73219668[MHz]
Irr_Dec_Merit_Factor       = 2.2
Irr_Decoupling             = TRUE
Irr_Noe                    = TRUE
Irr_Noise                  = WALTZ
Irr_Offset_Default         = 5[ppm]
Irr_Pwidth                 = 0.115[ms]
Irr_Pwidth_Default         = 0.115[ms]
Irr_Pwidth_Default_Calc    = 0.115[ms]
Irr_Pwidth_Temp1           = 0.115[ms]
Irr_Wurst                  = FALSE
Decimation_Rate            = 0
Experiment_Path            = c:\Program Files\J
Initial_Wait               = 1[s]
Noe_Time                   = 2[s]
Noe_Time_Flag              = FALSE
Relaxation_Delay_Calc      = 0[s]
Relaxation_Delay_Temp      = 2[s]
Repetition_Time            = 2.23068672[s]
```

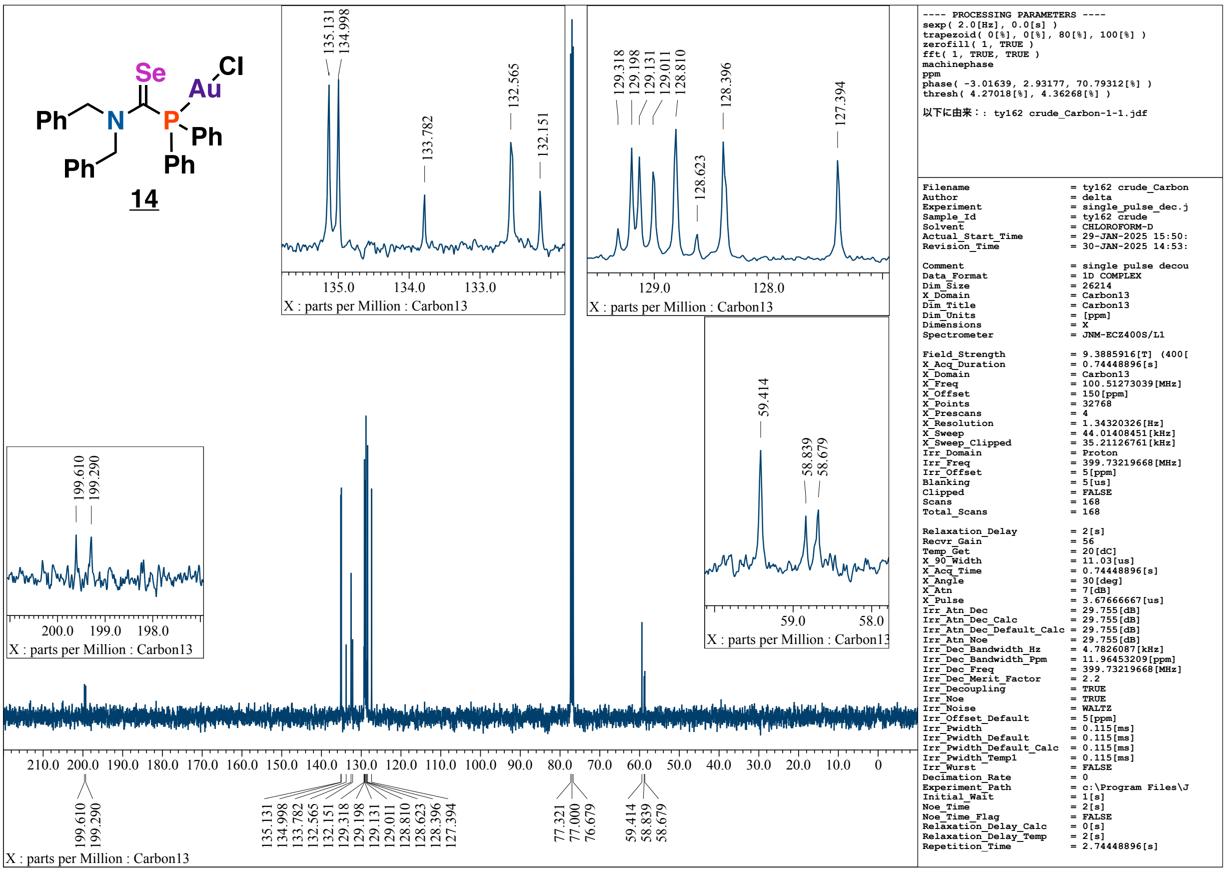
Figure S55. $^{77}Se\{^1H\}$ NMR spectrum (76 MHz, $CDCl_3$) of **13**.



```
---- PROCESSING PARAMETERS ----
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( 0.74834, 0, 50[%] )

以下に由来：: ty162 crude_Proton-8-1.jdf
```

```
Filename               = ty162 crude_Proton-8-
Author                 = delta
Experiment             = single_pulse.jxp
Sample_Id              = ty162 crude
Solvent                = CHLOROFORM-D
Actual_Start_Time      = 29-JAN-2025 15:16:37
Revision_Time          = 30-JAN-2025 14:40:31

Comment                = single_pulse
Data_Format            = 1D COMPLEX
Dim_Size               = 13107
X_Domain               = Proton
Dim_Title              = Proton
Dim_Units              = [ppm]
Dimensions             = X
Spectrometer           = JNM-ECZ400S/L1

Field_Strength         = 9.3885916[T] (400[MHz
X_Acq_Duration         = 2.18628096[s]
X_Domain               = Proton
X_Freq                 = 399.73219668[MHz]
X_Offset               = 5[ppm]
X_Points               = 16384
X_Prescans             = 1
X_Resolution           = 0.45739775[Hz]
X_Sweep                = 7.4940048[kHz]
X_Sweep_Clipped        = 5.99520384[kHz]
Irr_Domain             = Proton
Irr_Freq               = 399.73219668[MHz]
Irr_Offset             = 5[ppm]
Tri_Domain             = Proton
Tri_Freq               = 399.73219668[MHz]
Tri_Offset             = 5[ppm]
Blanking               = 2[us]
Clipped                = FALSE
Scans                  = 8
Total_Scans            = 8

Relaxation_Delay       = 5[s]
Recvr_Gain             = 66
Temp_Get               = 40[dC]
X_90_Width             = 6.28[us]
X_Acq_Time             = 2.18628096[s]
X_Angle                = 45[deg]
X_Atn                  = 4.5[dB]
X_Pulse                = 3.14[us]
Irr_Mode               = Off
Tri_Mode               = Off
Dante_Loop             = 500
Dante_Presat           = FALSE
Decimation_Rate        = 0
Experiment_Path        = c:\Program Files\JEOL
Initial_Wait           = 1[s]
Phase                  = {0, 90, 270, 180, 180
Presat_Time            = 5[s]
Presat_Time_Flag       = FALSE
Relaxation_Delay_Calc  = 0[s]
Relaxation_Delay_Temp  = 5[s]
Repetition_Time        = 7.18628096[s]
```

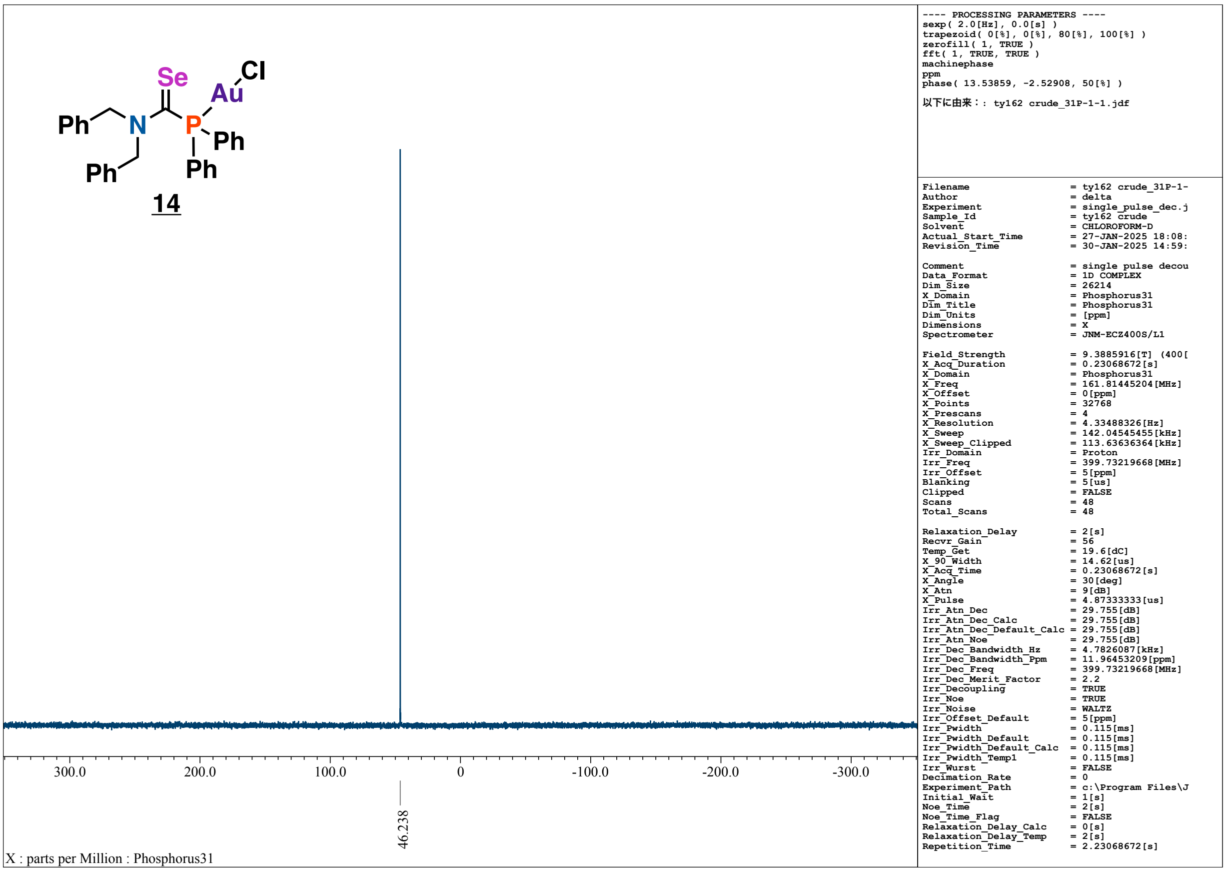
Figure S56. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **14**.



```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( -3.01639, 2.93177, 70.79312[%] )
thresh( 4.27018[%], 4.36268[%] )

以下に由来：: ty162 crude_Carbon-1-1.jdf
```

```
Filename                  = ty162 crude_Carbon
Author                    = delta
Experiment                = single_pulse_dec.j
Sample_Id                 = ty162 crude
Solvent                   = CHLOROFORM-D
Actual_Start_Time         = 29-JAN-2025 15:50:
Revision_Time             = 30-JAN-2025 14:53:

Comment                   = single pulse decou
Data_Format               = 1D COMPLEX
Dim_Size                  = 26214
X_Domain                  = Carbon13
Dim_Title                 = Carbon13
Dim_Units                 = [ppm]
Dimensions                = X
Spectrometer              = JNM-ECZ400S/L1

Field_Strength            = 9.3885916[T] (400[
X_Acq_Duration            = 0.74448896[s]
X_Domain                  = Carbon13
X_Freq                    = 100.51273039[MHz]
X_Offset                  = 150[ppm]
X_Points                  = 32768
X_Prescans                = 4
X_Resolution              = 1.34320326[Hz]
X_Sweep                   = 44.01408451[kHz]
X_Sweep_Clipped           = 35.21126761[kHz]
Irr_Domain                = Proton
Irr_Freq                  = 399.73219668[MHz]
Irr_Offset                = 5[ppm]
Blanking                  = 5[us]
Clipped                   = FALSE
Scans                     = 168
Total_Scans               = 168

Relaxation_Delay          = 2[s]
Recvr_Gain                = 56
Temp_Get                  = 20[dC]
X_90_Width                = 11.03[us]
X_Acq_Time                = 0.74448896[s]
X_Angle                   = 30[deg]
X_Atn                     = 7[dB]
X_Pulse                   = 3.67666667[us]
Irr_Atn_Dec               = 29.755[dB]
Irr_Atn_Dec_Calc          = 29.755[dB]
Irr_Atn_Dec_Default_Calc  = 29.755[dB]
Irr_Atn_Noe               = 29.755[dB]
Irr_Dec_Bandwidth_Hz      = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm     = 11.96453209[ppm]
Irr_Dec_Freq              = 399.73219668[MHz]
Irr_Dec_Merit_Factor      = 2.2
Irr_Decoupling            = TRUE
Irr_Noe                   = TRUE
Irr_Noise                 = WALTZ
Irr_Offset_Default        = 5[ppm]
Irr_Pwidth                = 0.115[ms]
Irr_Pwidth_Default        = 0.115[ms]
Irr_Pwidth_Default_Calc   = 0.115[ms]
Irr_Pwidth_Temp1          = 0.115[ms]
Irr_Wurst                 = FALSE
Decimation_Rate           = 0
Experiment_Path           = c:\Program Files\J
Initial_Wait              = 1[s]
Noe_Time                  = 2[s]
Noe_Time_Flag             = FALSE
Relaxation_Delay_Calc     = 0[s]
Relaxation_Delay_Temp     = 2[s]
Repetition_Time           = 2.74448896[s]
```

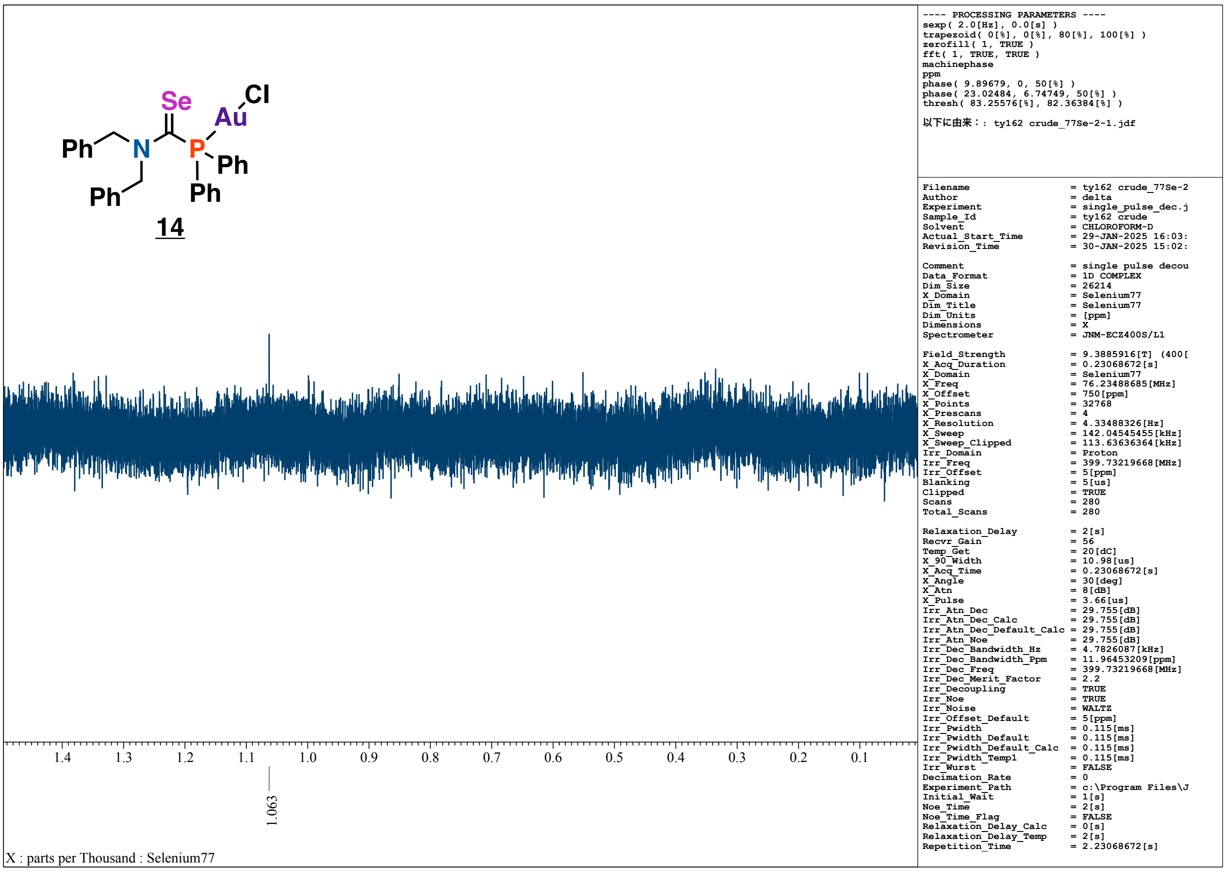
Figure S57. $^{13}C\{^{1}H\}$ NMR spectrum (100 MHz, $CDCl_3$) of **14**.



```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( 13.53859, -2.52908, 50[%] )

以下に由来：: ty162 crude_31P-1-1.jdf
```

```
Filename                    = ty162 crude_31P-1-
Author                      = delta
Experiment                  = single_pulse_dec.j
Sample_Id                   = ty162 crude
Solvent                     = CHLOROFORM-D
Actual_Start_Time           = 27-JAN-2025 18:08:
Revision_Time               = 30-JAN-2025 14:59:

Comment                     = single pulse decou
Data_Format                 = 1D COMPLEX
Dim_Size                    = 26214
X_Domain                    = Phosphorus31
Dim_Title                   = Phosphorus31
Dim_Units                   = [ppm]
Dimensions                  = X
Spectrometer                = JNM-ECZ400S/L1

Field_Strength              = 9.3885916[T] (400[
X_Acq_Duration              = 0.23068672[s]
X_Domain                    = Phosphorus31
X_Freq                      = 161.81445204[MHz]
X_Offset                    = 0[ppm]
X_Points                    = 32768
X_Prescans                  = 4
X_Resolution                = 4.33488326[Hz]
X_Sweep                     = 142.04545455[kHz]
X_Sweep_Clipped             = 113.63636364[kHz]
Irr_Domain                  = Proton
Irr_Freq                    = 399.73219668[MHz]
Irr_Offset                  = 5[ppm]
Blanking                    = 5[us]
Clipped                     = FALSE
Scans                       = 48
Total_Scans                 = 48

Relaxation_Delay            = 2[s]
Recvr_Gain                  = 56
Temp_Get                    = 19.6[dC]
X_90_Width                  = 14.62[us]
X_Acq_Time                  = 0.23068672[s]
X_Angle                     = 30[deg]
X_Atn                       = 9[dB]
X_Pulse                     = 4.87333333[us]
Irr_Atn_Dec                 = 29.755[dB]
Irr_Atn_Dec_Calc            = 29.755[dB]
Irr_Atn_Dec_Default_Calc = 29.755[dB]
Irr_Atn_Noe                 = 29.755[dB]
Irr_Dec_Bandwidth_Hz        = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm       = 11.96453209[ppm]
Irr_Dec_Freq                = 399.73219668[MHz]
Irr_Dec_Merit_Factor        = 2.2
Irr_Decoupling              = TRUE
Irr_Noe                     = TRUE
Irr_Noise                   = WALTZ
Irr_Offset_Default          = 5[ppm]
Irr_Pwidth                  = 0.115[ms]
Irr_Pwidth_Default          = 0.115[ms]
Irr_Pwidth_Default_Calc     = 0.115[ms]
Irr_Pwidth_Temp1            = 0.115[ms]
Irr_Wurst                   = FALSE
Decimation_Rate             = 0
Experiment_Path             = c:\Program Files\J
Initial_Wait                = 1[s]
Noe_Time                    = 2[s]
Noe_Time_Flag               = FALSE
Relaxation_Delay_Calc       = 0[s]
Relaxation_Delay_Temp       = 2[s]
Repetition_Time             = 2.23068672[s]
```

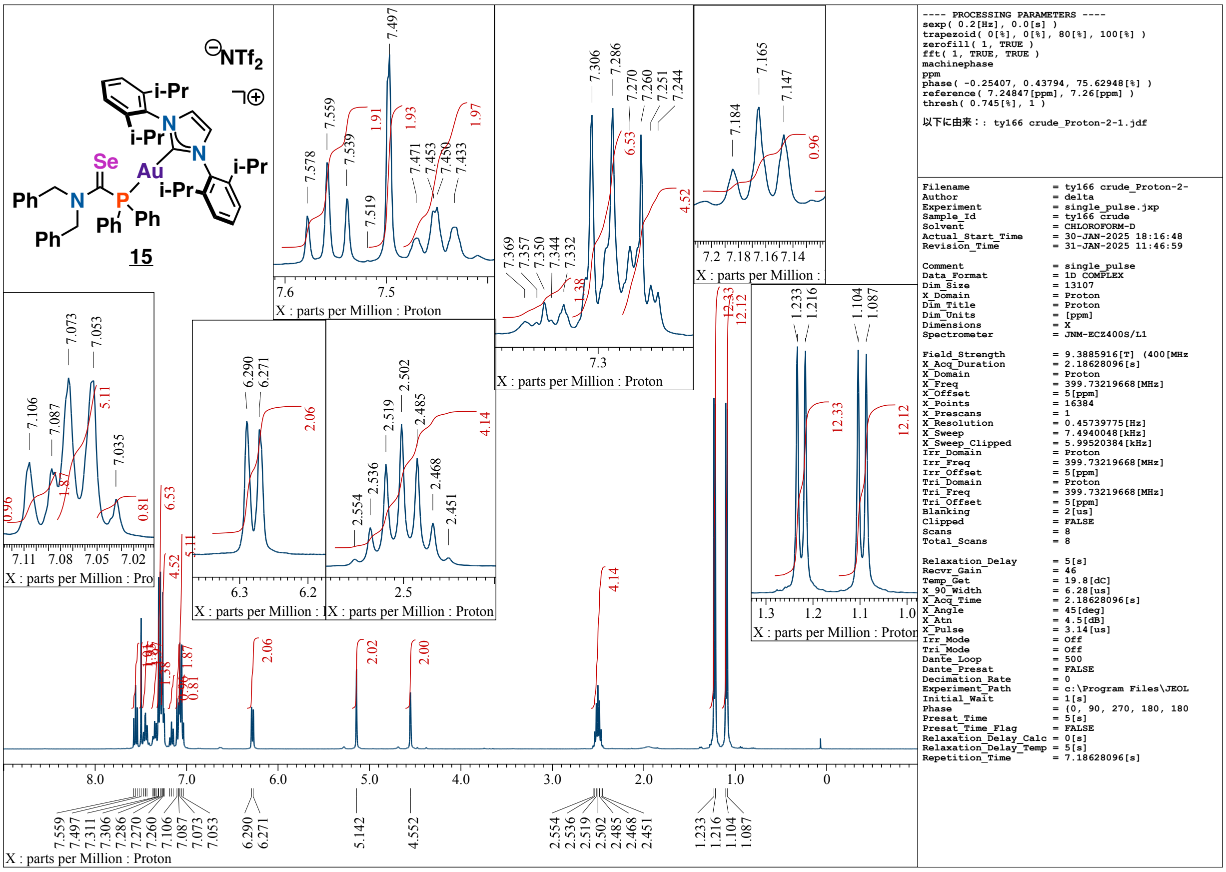
Figure S58. $^{31}P\{^1H\}$ NMR spectrum (126 MHz, $CDCl_3$) of **14**.



```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( 9.89679, 0, 50[%] )
phase( 23.02484, 6.74749, 50[%] )
thresh( 83.25576[%], 82.36384[%] )

以下に由来：: ty162 crude_77Se-2-1.jdf
```

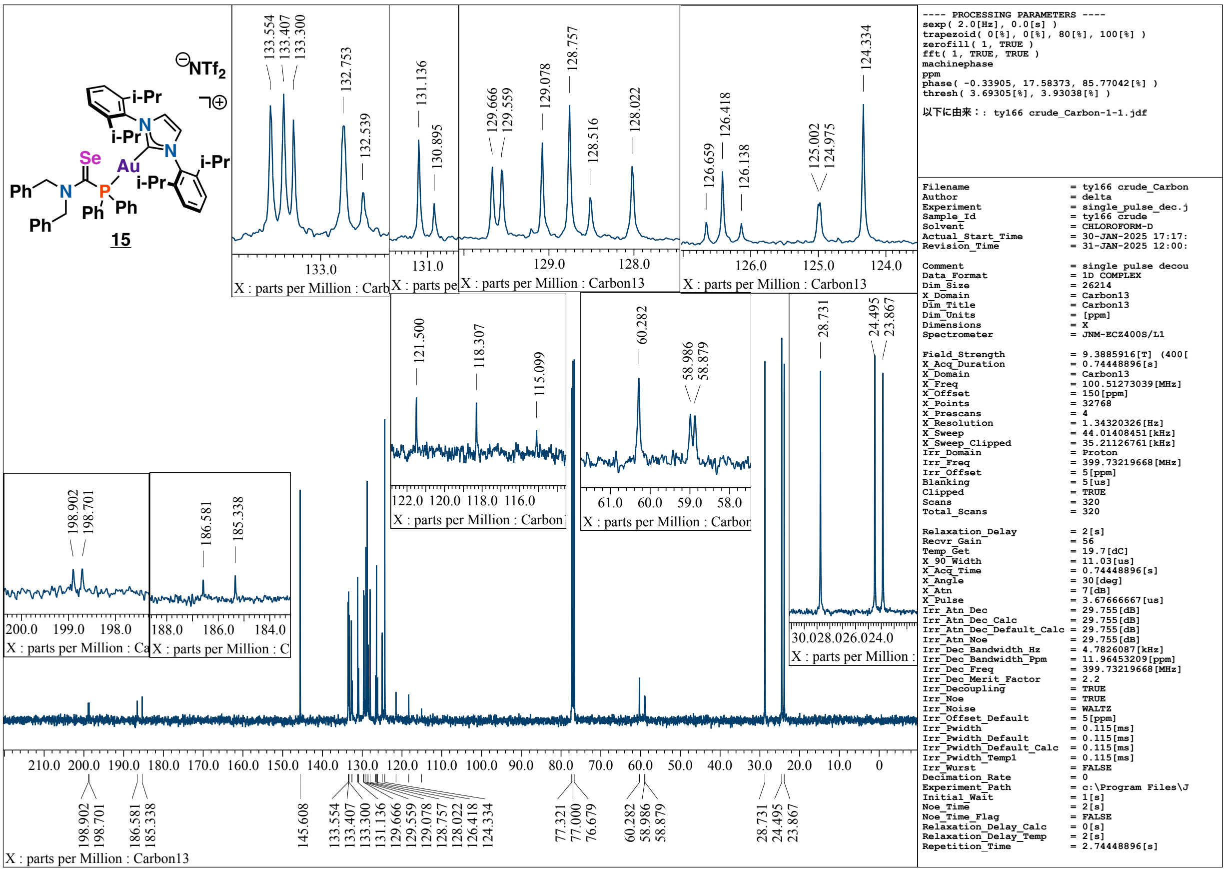
```
Filename                    = ty162 crude_77Se-2
Author                      = delta
Experiment                  = single_pulse_dec.j
Sample_Id                   = ty162 crude
Solvent                     = CHLOROFORM-D
Actual_Start_Time           = 29-JAN-2025 16:03:
Revision_Time               = 30-JAN-2025 15:02:

Comment                     = single pulse decou
Data_Format                 = 1D COMPLEX
Dim_Size                    = 26214
X_Domain                    = Selenium77
Dim_Title                   = Selenium77
Dim_Units                   = [ppm]
Dimensions                  = X
Spectrometer                = JNM-ECZ400S/L1

Field_Strength              = 9.3885916[T] (400[
X_Acq_Duration              = 0.23068672[s]
X_Domain                    = Selenium77
X_Freq                      = 76.23488685[MHz]
X_Offset                    = 750[ppm]
X_Points                    = 32768
X_Prescans                  = 4
X_Resolution                = 4.33488326[Hz]
X_Sweep                     = 142.04545455[kHz]
X_Sweep_Clipped             = 113.63636364[kHz]
Irr_Domain                  = Proton
Irr_Freq                    = 399.73219668[MHz]
Irr_Offset                  = 5[ppm]
Blanking                    = 5[us]
Clipped                     = TRUE
Scans                       = 280
Total_Scans                 = 280

Relaxation_Delay            = 2[s]
Recvr_Gain                  = 56
Temp_Get                    = 20[dC]
X_90_Width                  = 10.98[us]
X_Acq_Time                  = 0.23068672[s]
X_Angle                     = 30[deg]
X_Atn                       = 8[dB]
X_Pulse                     = 3.66[us]
Irr_Atn_Dec                 = 29.755[dB]
Irr_Atn_Dec_Calc            = 29.755[dB]
Irr_Atn_Dec_Default_Calc = 29.755[dB]
Irr_Atn_Noe                 = 29.755[dB]
Irr_Dec_Bandwidth_Hz        = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm       = 11.96453209[ppm]
Irr_Dec_Freq                = 399.73219668[MHz]
Irr_Dec_Merit_Factor        = 2.2
Irr_Decoupling              = TRUE
Irr_Noe                     = TRUE
Irr_Noise                   = WALTZ
Irr_Offset_Default          = 5[ppm]
Irr_Pwidth                  = 0.115[ms]
Irr_Pwidth_Default          = 0.115[ms]
Irr_Pwidth_Default_Calc     = 0.115[ms]
Irr_Pwidth_Temp1            = 0.115[ms]
Irr_Wurst                   = FALSE
Decimation_Rate             = 0
Experiment_Path             = c:\Program Files\J
Initial_Wait                = 1[s]
Noe_Time                    = 2[s]
Noe_Time_Flag               = FALSE
Relaxation_Delay_Calc       = 0[s]
Relaxation_Delay_Temp       = 2[s]
Repetition_Time             = 2.23068672[s]
```

Figure S59. $^{77}Se\{^1H\}$ NMR spectrum (76 MHz, $CDCl_3$) of **14**.



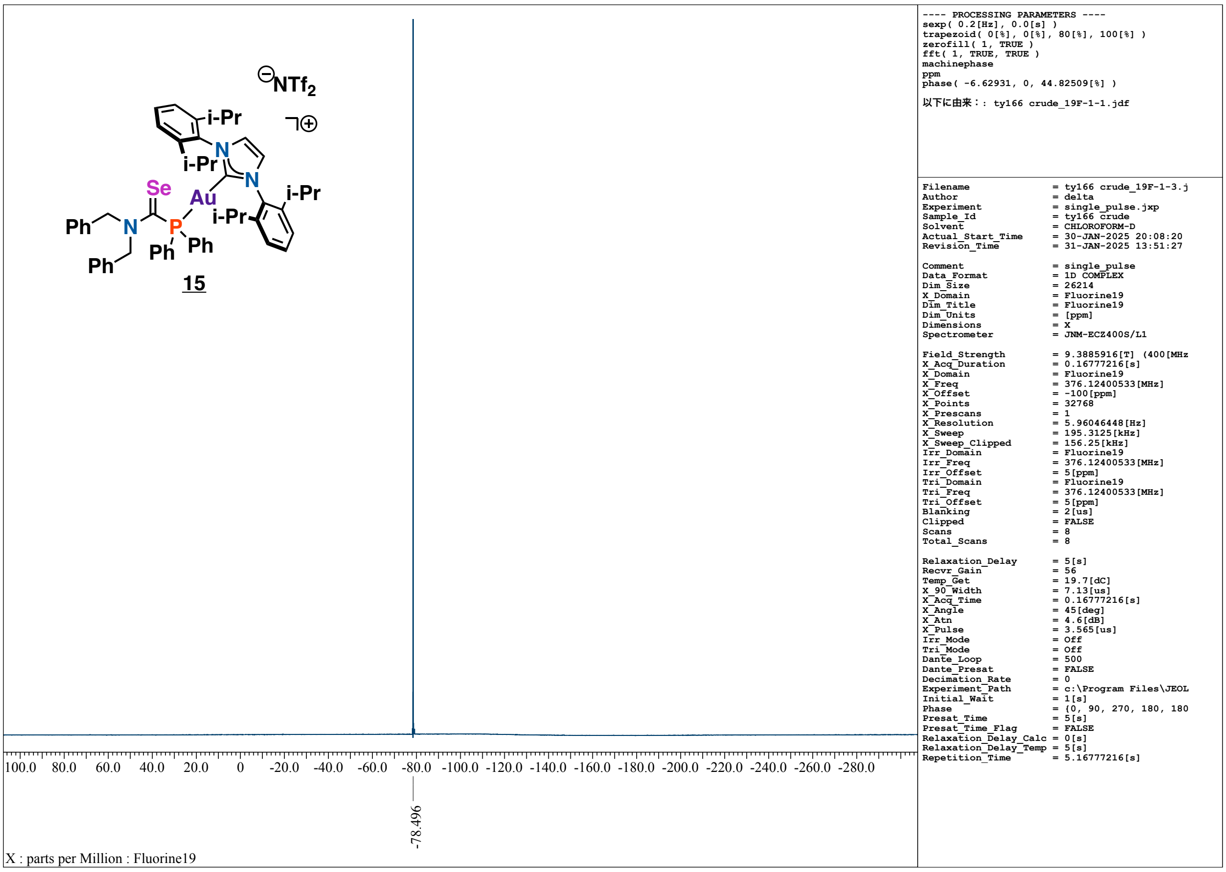
```
---- PROCESSING PARAMETERS ----
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( -0.25407, 0.43794, 75.62948[%] )
reference( 7.24847[ppm], 7.26[ppm] )
thresh( 0.745[%], 1 )

以下に由来：: ty166 crude_Proton-2-1.jdf

Filename               = ty166 crude_Proton-2-
Author                 = delta
Experiment             = single_pulse.jxp
Sample_Id              = ty166 crude
Solvent                = CHLOROFORM-D
Actual_Start_Time      = 30-JAN-2025 18:16:48
Revision_Time          = 31-JAN-2025 11:46:59

Comment                = single_pulse
Data_Format            = 1D COMPLEX
Dim_Size               = 13107
X_Domain               = Proton
Dim_Title              = Proton
Dim_Units              = [ppm]
Dimensions             = X
Spectrometer           = JNM-ECZ400S/L1

Field_Strength         = 9.3885916[T] (400[MHz
X_Acq_Duration         = 2.18628096[s]
X_Domain               = Proton
X_Freq                 = 399.73219668[MHz]
X_Offset               = 5[ppm]
X_Points               = 16384
X_Prescans             = 1
X_Resolution           = 0.45739775[Hz]
X_Sweep                = 7.4940048[kHz]
X_Sweep_Clipped        = 5.99520384[kHz]
Irr_Domain             = Proton
Irr_Freq               = 399.73219668[MHz]
Irr_Offset             = 5[ppm]
Tri_Domain             = Proton
Tri_Freq               = 399.73219668[MHz]
Tri_Offset             = 5[ppm]
Blanking               = 2[us]
Clipped                = FALSE
Scans                  = 8
Total_Scans            = 8

Relaxation_Delay       = 5[s]
Recvr_Gain             = 46
Temp_Get               = 19.8[dC]
X_90_Width             = 6.28[us]
X_Acq_Time             = 2.18628096[s]
X_Angle                = 45[deg]
X_Atn                  = 4.5[dB]
X_Pulse                = 3.14[us]
Irr_Mode               = Off
Tri_Mode               = Off
Dante_Loop             = 500
Dante_Presat           = FALSE
Decimation_Rate        = 0
Experiment_Path        = c:\Program Files\JEOL
Initial_Wait           = 1[s]
Phase                  = {0, 90, 270, 180, 180
Presat_Time            = 5[s]
Presat_Time_Flag       = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 5[s]
Repetition_Time        = 7.18628096[s]
```

Figure S60. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **15**.



```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( -0.33905, 17.58373, 85.77042[%] )
thresh( 3.69305[%], 3.93038[%] )

以下に由来：: ty166 crude_Carbon-1-1.jdf

Filename                    = ty166 crude_Carbon
Author                      = delta
Experiment                  = single_pulse_dec.j
Sample_Id                   = ty166 crude
Solvent                     = CHLOROFORM-D
Actual_Start_Time           = 30-JAN-2025 17:17:
Revision_Time               = 31-JAN-2025 12:00:

Comment                     = single pulse decou
Data_Format                 = 1D COMPLEX
Dim_Size                    = 26214
X_Domain                    = Carbon13
Dim_Title                   = Carbon13
Dim_Units                   = [ppm]
Dimensions                  = X
Spectrometer                = JNM-ECZ400S/L1

Field_Strength              = 9.3885916[T] (400[
X_Acq_Duration              = 0.74448896[s]
X_Domain                    = Carbon13
X_Freq                      = 100.51273039[MHz]
X_Offset                    = 150[ppm]
X_Points                    = 32768
X_Prescans                  = 4
X_Resolution                = 1.34320326[Hz]
X_Sweep                     = 44.01408451[kHz]
X_Sweep_Clipped             = 35.21126761[kHz]
Irr_Domain                  = Proton
Irr_Freq                    = 399.73219668[MHz]
Irr_Offset                  = 5[ppm]
Blanking                    = 5[us]
Clipped                     = TRUE
Scans                       = 320
Total_Scans                 = 320

Relaxation_Delay            = 2[s]
Recvr_Gain                  = 56
Temp_Get                    = 19.7[dC]
X_90_Width                  = 11.03[us]
X_Acq_Time                  = 0.74448896[s]
X_Angle                     = 30[deg]
X_Atn                       = 7[dB]
X_Pulse                     = 3.67666667[us]
Irr_Atn_Dec                 = 29.755[dB]
Irr_Atn_Dec_Calc            = 29.755[dB]
Irr_Atn_Dec_Default_Calc = 29.755[dB]
Irr_Atn_Noe                 = 29.755[dB]
Irr_Dec_Bandwidth_Hz        = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm       = 11.96453209[ppm]
Irr_Dec_Freq                = 399.73219668[MHz]
Irr_Dec_Merit_Factor        = 2.2
Irr_Decoupling              = TRUE
Irr_Noe                     = TRUE
Irr_Noise                   = WALTZ
Irr_Offset_Default          = 5[ppm]
Irr_Pwidth                  = 0.115[ms]
Irr_Pwidth_Default          = 0.115[ms]
Irr_Pwidth_Default_Calc     = 0.115[ms]
Irr_Pwidth_Temp1            = 0.115[ms]
Irr_Wurst                   = FALSE
Decimation_Rate             = 0
Experiment_Path             = c:\Program Files\J
Initial_Wait                = 1[s]
Noe_Time                    = 2[s]
Noe_Time_Flag               = FALSE
Relaxation_Delay_Calc       = 0[s]
Relaxation_Delay_Temp       = 2[s]
Repetition_Time             = 2.74448896[s]
```

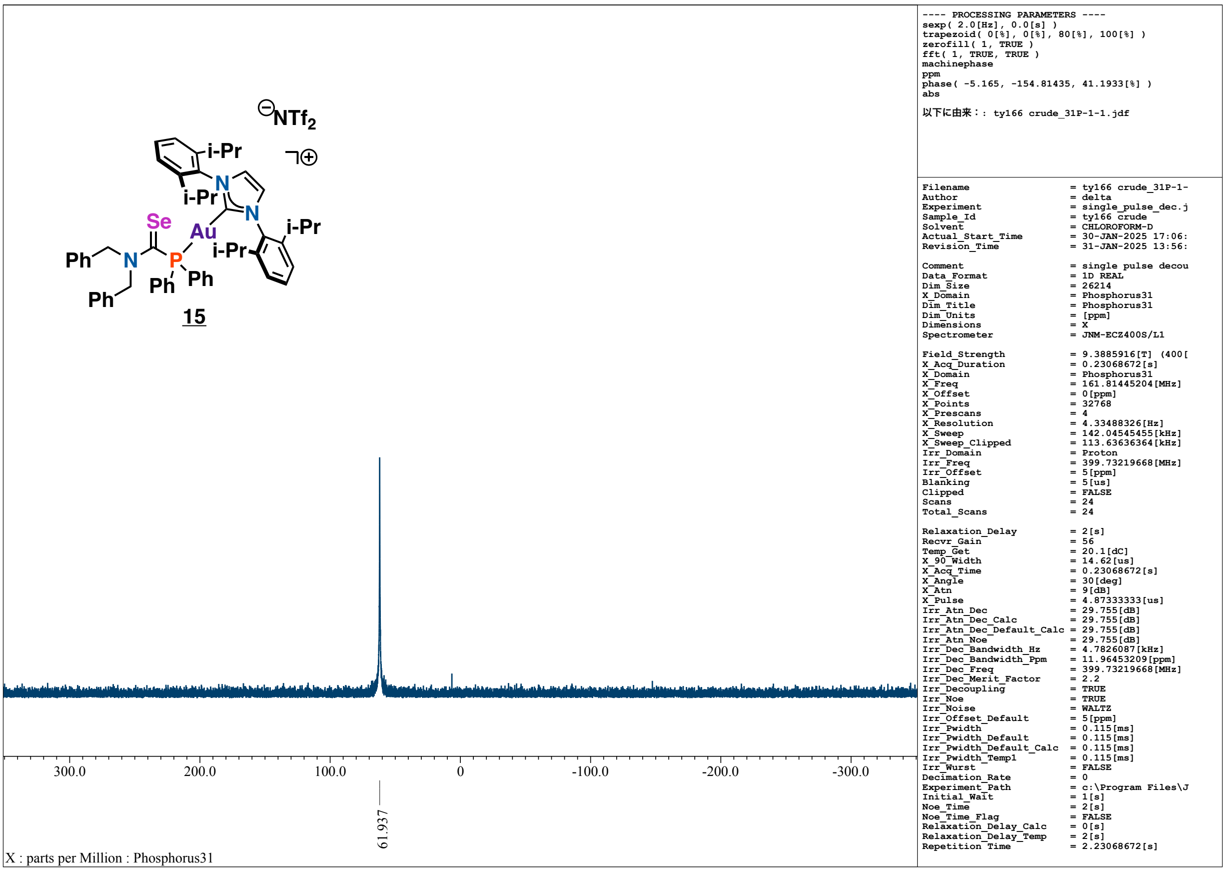
Figure S61. $^{13}C\{^1H\}$ NMR spectrum (100 MHz, $CDCl_3$) of **15**.



```
---- PROCESSING PARAMETERS ----
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( -6.62931, 0, 44.82509[%] )

以下に由来：: ty166 crude_19F-1-1.jdf
```

```
Filename              = ty166 crude_19F-1-3.j
Author                = delta
Experiment            = single_pulse.jxp
Sample_Id             = ty166 crude
Solvent               = CHLOROFORM-D
Actual_Start_Time     = 30-JAN-2025 20:08:20
Revision_Time         = 31-JAN-2025 13:51:27

Comment               = single_pulse
Data_Format           = 1D COMPLEX
Dim_Size              = 26214
X_Domain              = Fluorine19
Dim_Title             = Fluorine19
Dim_Units             = [ppm]
Dimensions            = X
Spectrometer          = JNM-ECZ400S/L1

Field_Strength        = 9.3885916[T] (400[MHz
X_Acq_Duration        = 0.16777216[s]
X_Domain              = Fluorine19
X_Freq                = 376.12400533[MHz]
X_Offset              = -100[ppm]
X_Points              = 32768
X_Prescans            = 1
X_Resolution          = 5.96046448[Hz]
X_Sweep               = 195.3125[kHz]
X_Sweep_Clipped       = 156.25[kHz]
Irr_Domain            = Fluorine19
Irr_Freq              = 376.12400533[MHz]
Irr_Offset            = 5[ppm]
Tri_Domain            = Fluorine19
Tri_Freq              = 376.12400533[MHz]
Tri_Offset            = 5[ppm]
Blanking              = 2[us]
Clipped               = FALSE
Scans                 = 8
Total_Scans           = 8

Relaxation_Delay      = 5[s]
Recvr_Gain            = 56
Temp_Get              = 19.7[dC]
X_90_Width            = 7.13[us]
X_Acq_Time            = 0.16777216[s]
X_Angle               = 45[deg]
X_Atn                 = 4.6[dB]
X_Pulse               = 3.565[us]
Irr_Mode              = Off
Tri_Mode              = Off
Dante_Loop            = 500
Dante_Presat          = FALSE
Decimation_Rate       = 0
Experiment_Path       = c:\Program Files\JEOL
Initial_Wait          = 1[s]
Phase                 = {0, 90, 270, 180, 180
Presat_Time           = 5[s]
Presat_Time_Flag      = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 5[s]
Repetition_Time       = 5.16777216[s]
```

Figure S62. ^{19}F{^{1}H} NMR spectrum (376 MHz, CDCl$_3$) of **15**.



```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( -5.165, -154.81435, 41.1933[%] )
abs

以下に由来：: ty166 crude_31P-1-1.jdf
```

```
Filename                 = ty166 crude_31P-1-
Author                   = delta
Experiment               = single_pulse_dec.j
Sample_Id                = ty166 crude
Solvent                  = CHLOROFORM-D
Actual_Start_Time        = 30-JAN-2025 17:06:
Revision_Time            = 31-JAN-2025 13:56:

Comment                  = single pulse decou
Data_Format              = 1D REAL
Dim_Size                 = 26214
X_Domain                 = Phosphorus31
Dim_Title                = Phosphorus31
Dim_Units                = [ppm]
Dimensions               = X
Spectrometer             = JNM-ECZ400S/L1

Field_Strength           = 9.3885916[T] (400[
X_Acq_Duration           = 0.23068672[s]
X_Domain                 = Phosphorus31
X_Freq                   = 161.81445204[MHz]
X_Offset                 = 0[ppm]
X_Points                 = 32768
X_Prescans               = 4
X_Resolution             = 4.33488326[Hz]
X_Sweep                  = 142.04545455[kHz]
X_Sweep_Clipped          = 113.63636364[kHz]
Irr_Domain               = Proton
Irr_Freq                 = 399.73219668[MHz]
Irr_Offset               = 5[ppm]
Blanking                 = 5[us]
Clipped                  = FALSE
Scans                    = 24
Total_Scans              = 24

Relaxation_Delay         = 2[s]
Recvr_Gain               = 56
Temp_Get                 = 20.1[dC]
X_90_Width               = 14.62[us]
X_Acq_Time               = 0.23068672[s]
X_Angle                  = 30[deg]
X_Atn                    = 9[dB]
X_Pulse                  = 4.87333333[us]
Irr_Atn_Dec              = 29.755[dB]
Irr_Atn_Dec_Calc         = 29.755[dB]
Irr_Atn_Dec_Default_Calc = 29.755[dB]
Irr_Atn_Noe              = 29.755[dB]
Irr_Dec_Bandwidth_Hz     = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm    = 11.96453209[ppm]
Irr_Dec_Freq             = 399.73219668[MHz]
Irr_Dec_Merit_Factor     = 2.2
Irr_Decoupling           = TRUE
Irr_Noe                  = TRUE
Irr_Noise                = WALTZ
Irr_Offset_Default       = 5[ppm]
Irr_Pwidth               = 0.115[ms]
Irr_Pwidth_Default       = 0.115[ms]
Irr_Pwidth_Default_Calc  = 0.115[ms]
Irr_Pwidth_Temp1         = 0.115[ms]
Irr_Wurst                = FALSE
Decimation_Rate          = 0
Experiment_Path          = c:\Program Files\J
Initial_Wait             = 1[s]
Noe_Time                 = 2[s]
Noe_Time_Flag            = FALSE
Relaxation_Delay_Calc    = 0[s]
Relaxation_Delay_Temp    = 2[s]
Repetition_Time          = 2.23068672[s]
```

Figure S63. ^{31}P{^{1}H} NMR spectrum (126 MHz, CDCl$_3$) of **15**.

5. Reference

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