

Synthesis and Reactivity of a Bulky Indium Anion

by

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Supplementary Information

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1. Materials and Methods

1.1 Experimental Procedures

The experimental procedures were conducted under a dry argon atmosphere using standard Schenk–line techniques or in a conventional nitrogen–filled glovebox. Diethyl ether (Et₂O), tetrahydrofuran (THF), toluene and hexane solvents were obtained from a PureSolv MD 5 system and were stored over activated 5 Å molecular sieves for 24 hours before use. The alkali metals used in the experiments were obtained as chunks and stored under mineral oil. Before their use, the metals were washed with hexane to remove the mineral oil, and any oxidised surfaces were mechanically removed under an inert atmosphere. Unless indicated otherwise, all other chemicals utilised in the experiments were purchased from Sigma–Aldrich and used without additional purification. The synthesised compounds were characterised using 2D NMR spectra which were acquired using a JOEL 500 MHz (11.747 Tesla) spectrometer equipped with a ROYAL digital auto–tune probe S. The spectrometer operated at frequencies of 500.1 MHz for proton (¹H) NMR and 125.8 MHz for carbon (¹³C) NMR. Spectra were recorded at a temperature of 294 K. Proton and carbon chemical shifts were referenced internally to residual solvent resonances. Coupling constants were reported in Hz. Data processing was performed using Mestrenova software suites. Crystals were prepared and mounted by Sophie G. Unsworth and modelled by Scott Cameron. Single crystal X–ray diffraction was collected using an Agilent diffractometer system located at the Victoria University of Wellington.

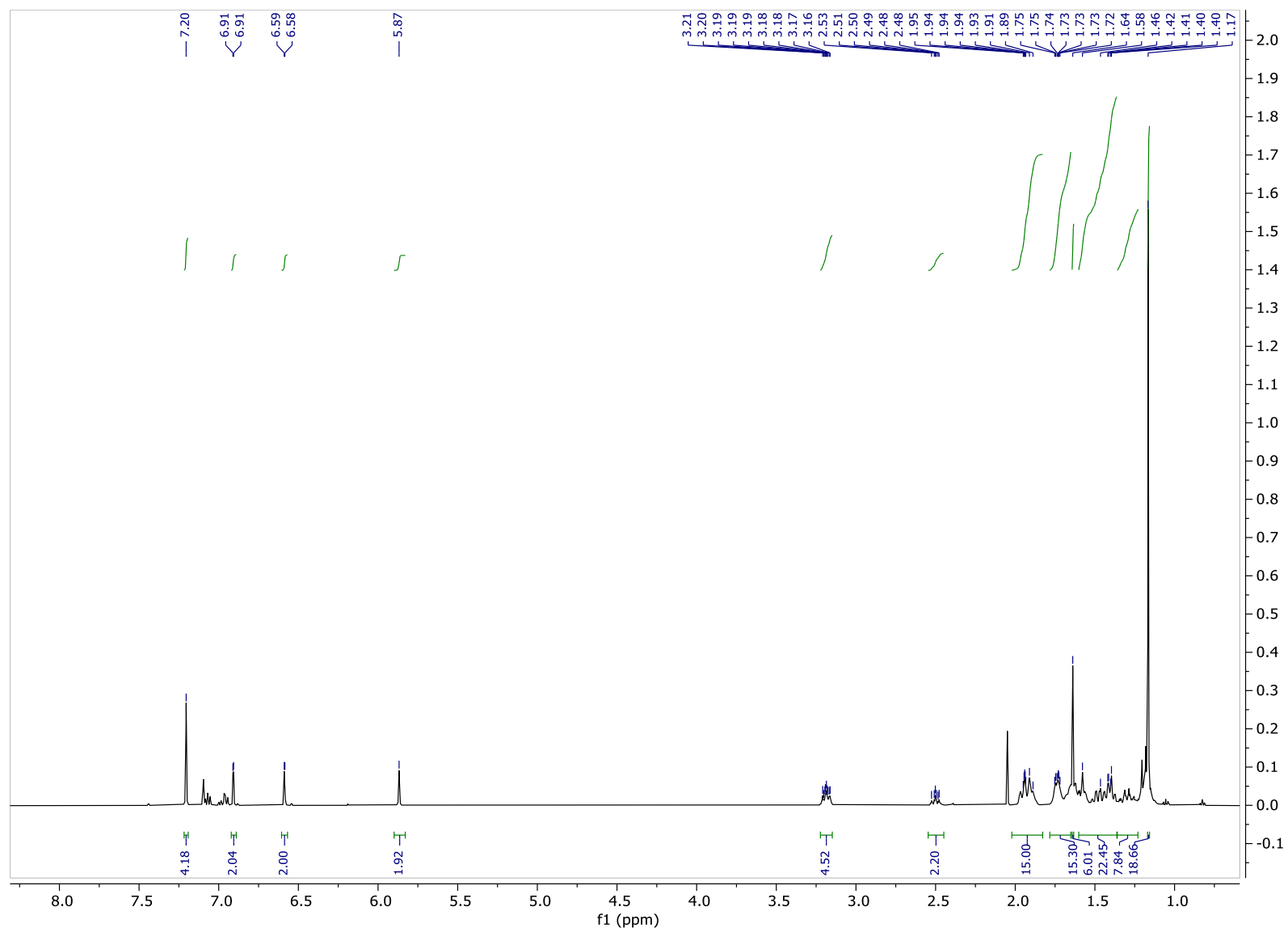
1.2 Synthetic and Spectroscopic for new compounds.

Preparation of (xNON^{TCHP})H₂ (37).

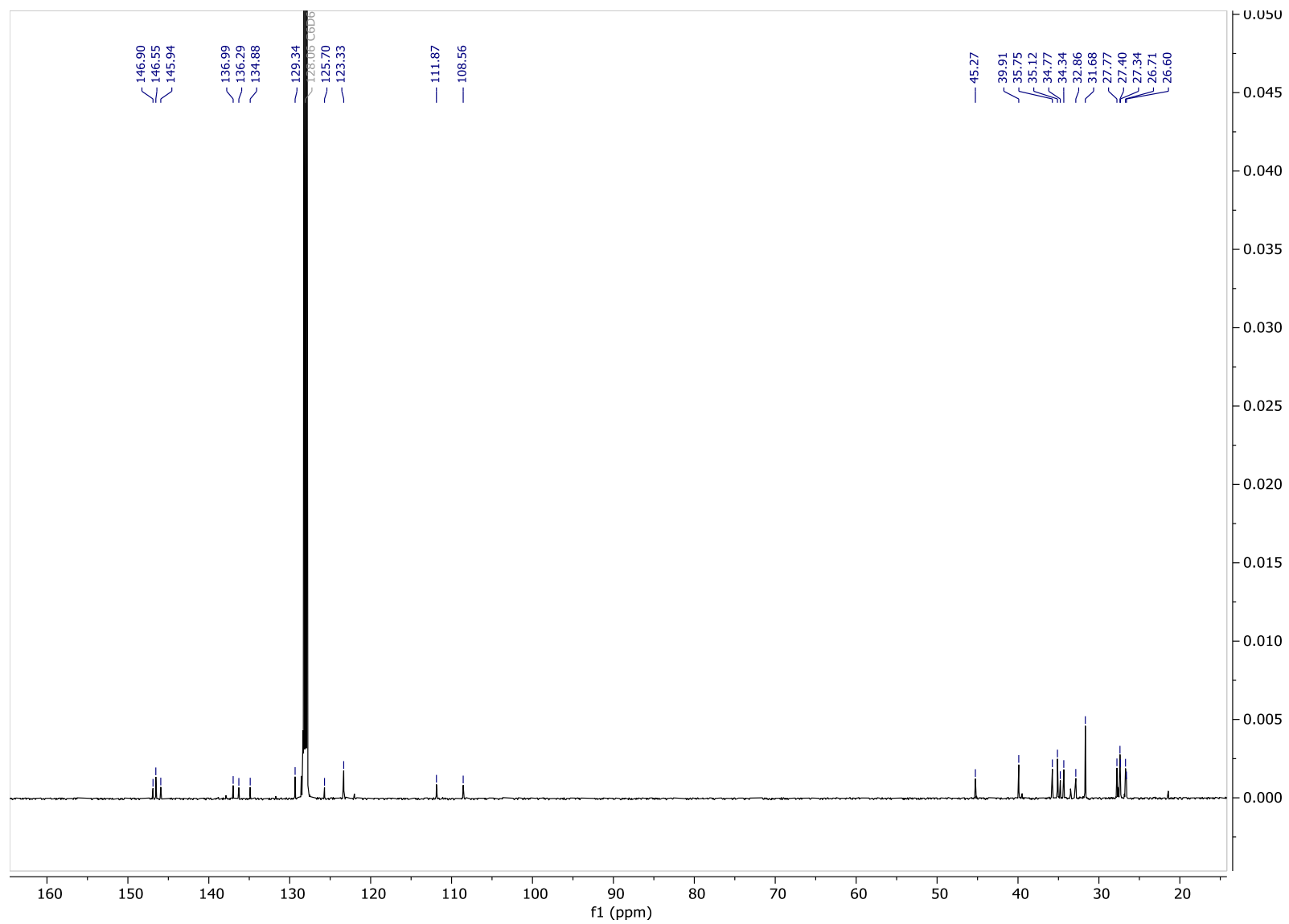
Sodium tert-butoxide (311.4 mg, 3.24 mmol), palladium(II) acetate (5.4 mg, 0.02 mmol), Oxydi-2, 1-phenylene)bis(diphenylphosphine (18.6 mg, 0.03 mmol), 4,5-dibromo-2,7-di-tert-butyl-9,9-dimethyl-9H-xanthene (526.2 mg, 1.17 mmol) and TCHP amine (794.2mg, 2.34 mmol) were added in a toluene solution and stirred for 5 days at 100°C. The resulting solution was quenched with water, extracted into toluene (3 x 50 mL), dried over MgSO₄, and concentrated to approximately 10 mL. Recrystallisation was achieved from hot toluene to obtain off-white crystals. Yield 987.7 mg, 84.7 %.

¹H NMR (500 MHz, C₆D₆) δ 7.20 (s, 4H, ArH), 6.91 (d, *J* = 2.2 Hz, 2H, XA-*p*-CH), 6.59 (d, *J* = 2.2 Hz, 2H, XA-*o*-CH), 5.87 (s, 2H, NH), 3.19 (tt, *J* = 8.1, 3.2 Hz, 4H, *o*-CyH), 2.55 – 2.44 (m, 2H, *p*-CyH), 2.00 – 1.83 (m, 15H, CyH₂), 1.79 – 1.70 (m, 15H, CyH₂), 1.64 (s, 6H, C(CH₃)₂), 1.60 – 1.38 (m, 22H, CyH₂), 1.35 – 1.22 (m, 8H, CyH₂), 1.17 (s, 18H, C(CH₃)₃).

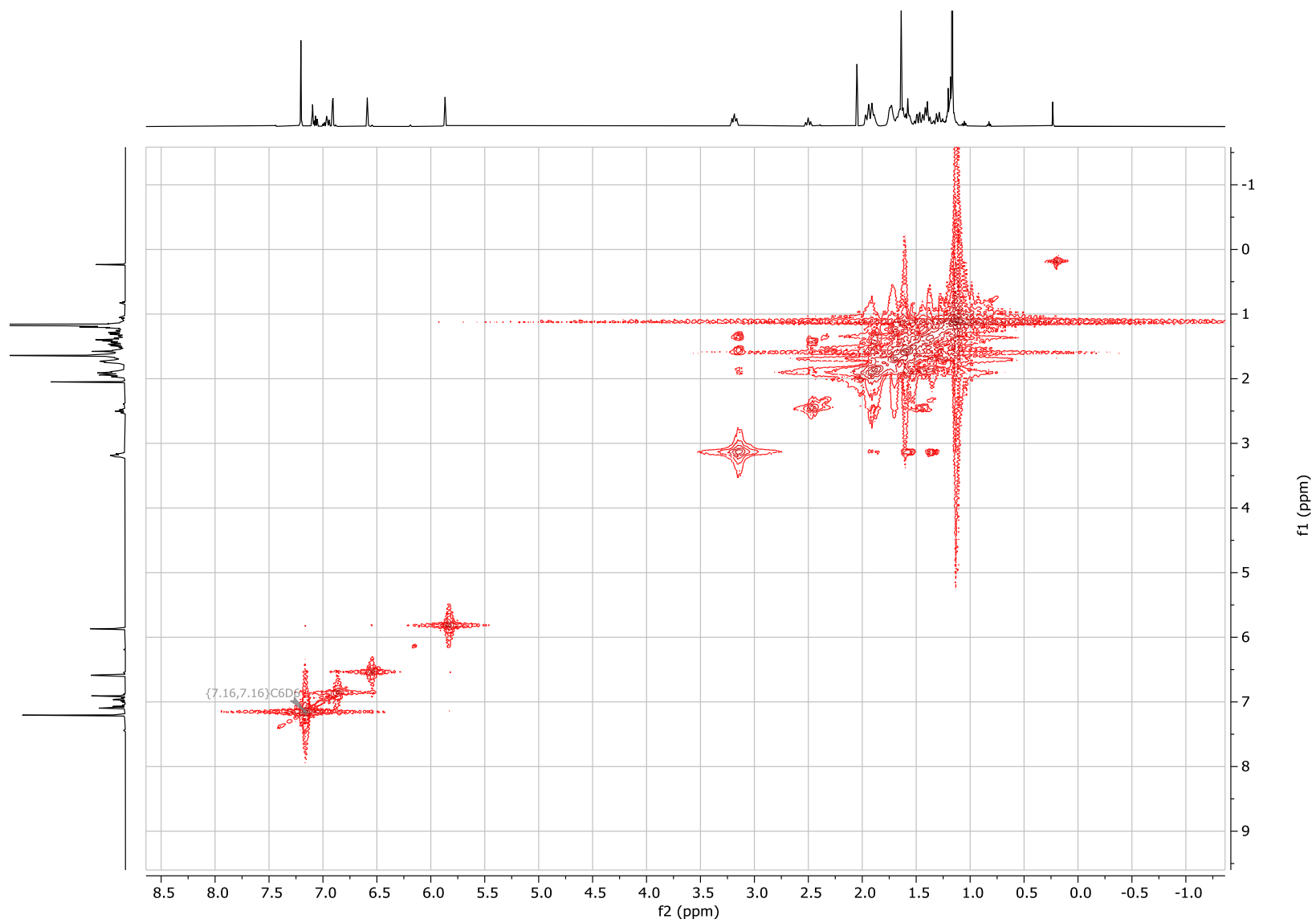
¹³C{¹H} NMR (126 MHz, C₆D₆) δ 146.9, 146.6, 145.9, 136.99, 136.3, 134.9, 129.3, 125.7, 123.3, 111.9, 108.6 (ArC), 45.3 (*p*-CyC), 39.9 (*o*-CyC), 35.8, 35.1, 34.8, 34.3 (C(CH₃)₂, C(CH₃)₃, CyCH₂), 32.9 C(CH₃)₂, 31.7 (C(CH₃)₃), 27.8, 27.4, 27.3, 26.7, 26.6 (C(CH₃)₂, C(CH₃)₃, CyCH₂).



Supplementary figure 1. ^1H NMR spectrum (500 MHz, C_6D_6) of $(\text{x})\text{NON}^{\text{TCHP}}\text{H}_2$ (**37**).



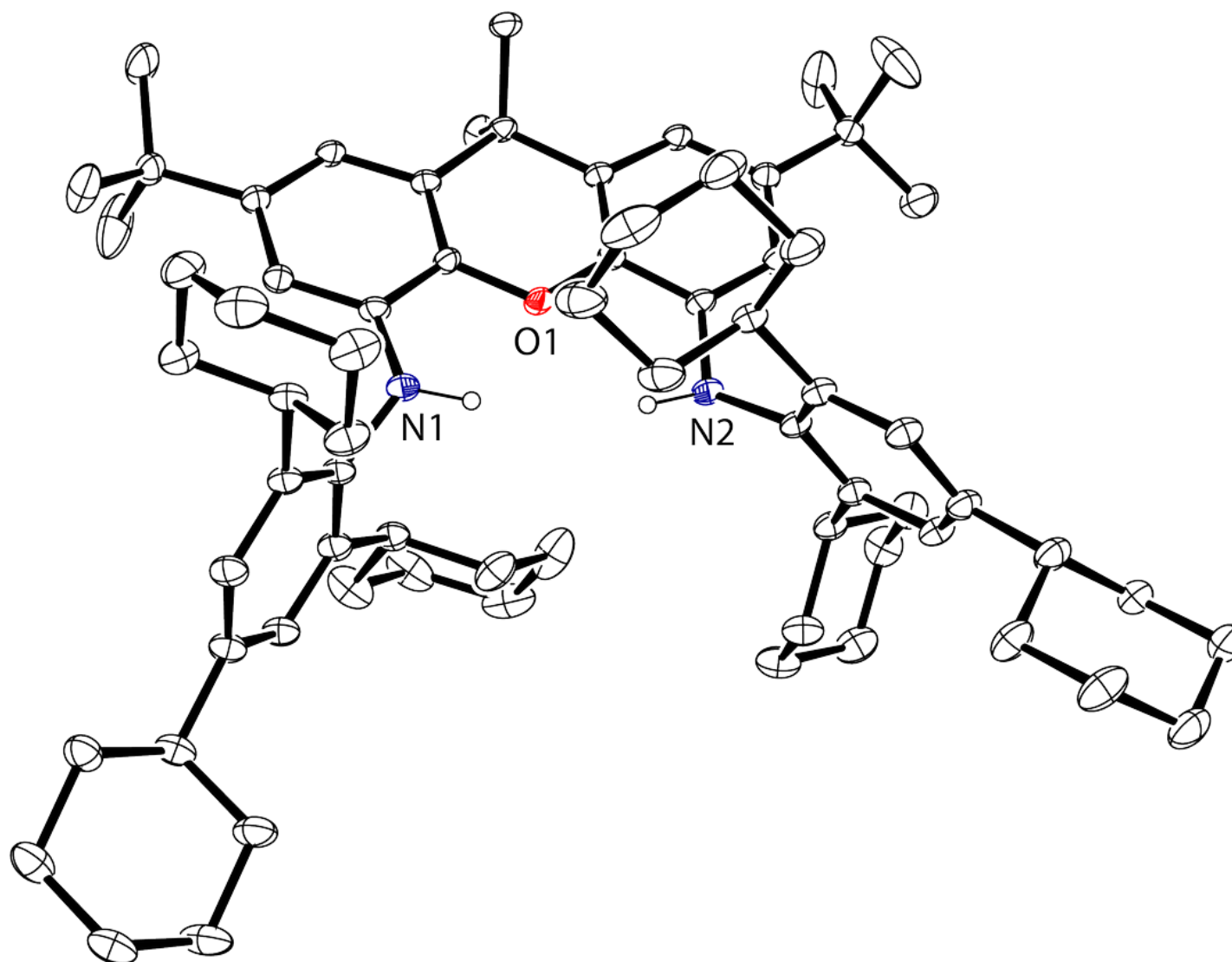
Supplementary figure 2. ¹³C NMR spectrum (126 MHz, C₆D₆) of (xNON^{TCHP})H₂ (**37**).



Supplementary figure 3. ^1H - ^1H COSY NMR spectrum (500 MHz, C_6D_6) of $(\text{xNON}^{\text{TCHP}})\text{H}_2$ (**37**).



Supplementary figure 4. ^1H - ^{13}C HSQC NMR spectrum (500 MHz, C_6D_6) of $(\text{xNON}^{\text{TCHP}})_2$ (**37**).



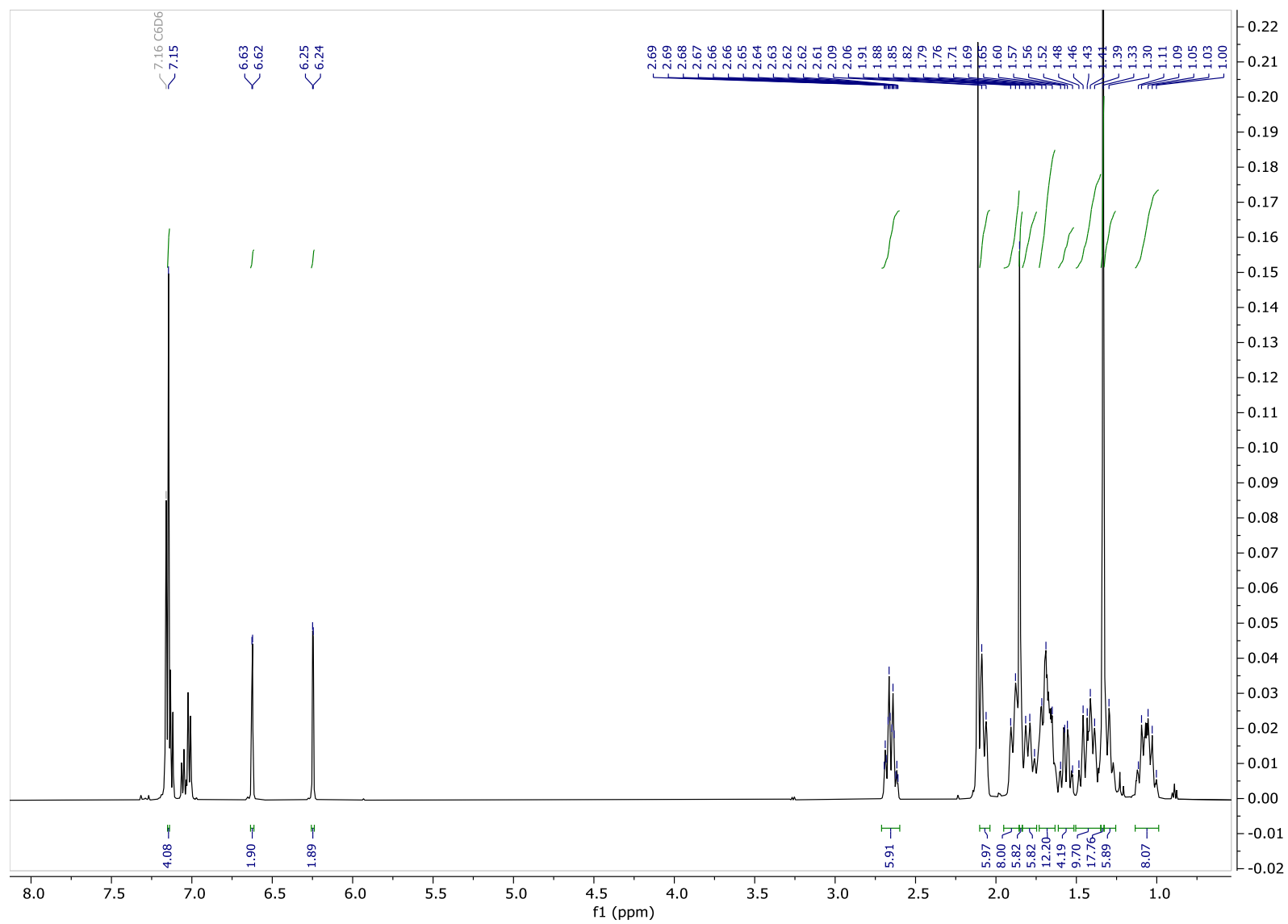
Supplementary figure 5. Ortep representation (ellipsoid 30% probability) of (xNON^{TCHP})H₂, **37**. Hydrogen atoms have been omitted for clarity.

Preparation of (xNON^{TCHP})K₂, 38

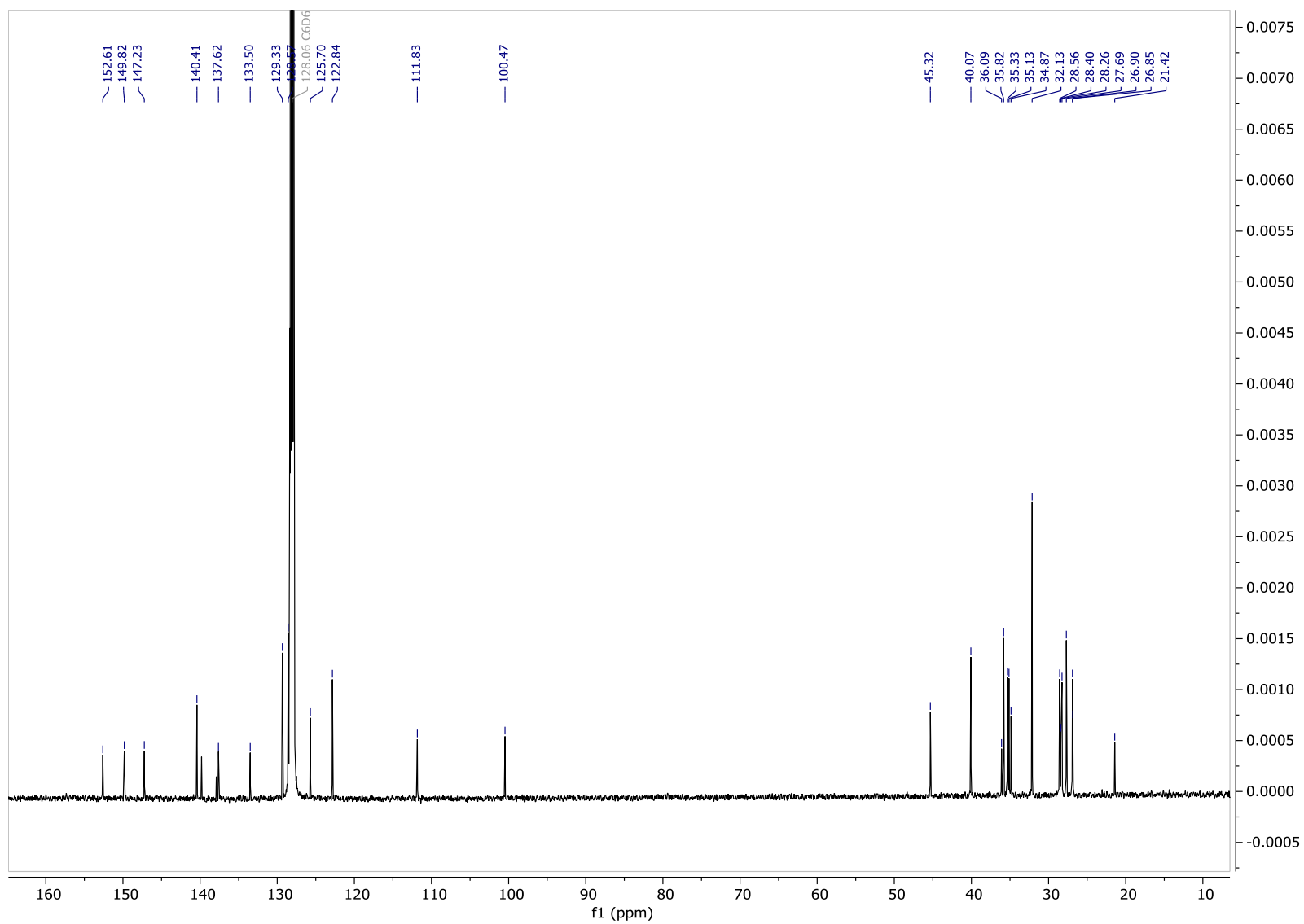
A solution of (xNON^{TCHP})H₂ (412.7 mg, 0.414 mmol) in toluene was added to an excess of potassium hydride (36.5 mg, 0.919 mmol) in toluene and stirred for 48 hours under nitrogen at 80°C. The mixture was filtered *via* a cannula to give a green solution. The solvent was removed *in vacuo* and the resulting green powder was heated under vacuum (10⁻² mbar) to remove the coordinated toluene yielding “(xNON^{TCHP})K₂”. Recrystallisation was achieved from a saturated toluene solution obtained light green crystals. Yield 425 mg, 97.0 %.

¹H NMR (500 MHz, C₆D₆) δ 7.15 (s, 4H, ArH), 6.62 (d, *J* = 2.1 Hz, 2H, XA-*p*-CH), 6.25 (d, *J* = 2.1 Hz, 2H, XA-*o*-CH), 2.65 (qt, *J* = 12.2, 3.2 Hz, 6H, CyH), 2.07 (d, *J* = 13.6 Hz, 6H, CyH₂), 1.89 (d, *J* = 15.0 Hz, 8H, CyH₂), 1.85 (s, 6H, C(CH₃)₂), 1.79 (t, *J* = 13.8 Hz, 6H, CyH₂), 1.69 (m, 12H, CyH₂), 1.58 (m, 4H, CyH₂), 1.43 (m, 10H, CyH₂), 1.33 (s, 18H, C(CH₃)₃), 1.30 (s, 6H, CyH₂), 1.07 (m, 8H, CyH₂).

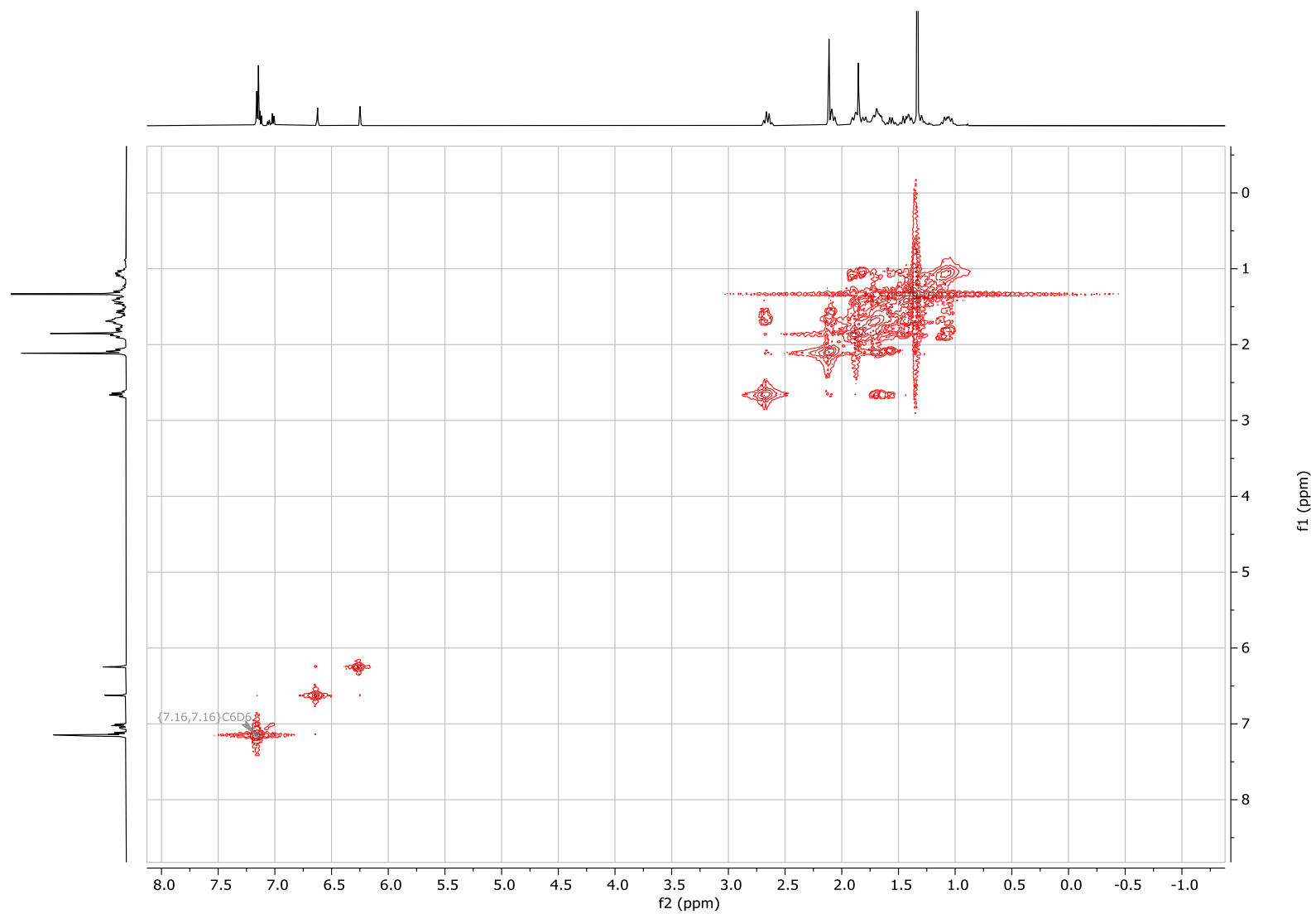
¹³C{¹H} NMR (126 MHz, C₆D₆) δ 152.6, 149.8, 147.2, 140.4, 137.6, 133.5, 129.3, 128.6, (Ar), 125.7 (C(CH₃)₂), 122.8 (ArH), 111.8 (XA-*p*-CH), 100.5 (XA-*o*-CH), 45.3 (*p*-CyH), 40.1 (*o*-CyH), 36.1, 35.8, 35.3, 33.1 (CyH₂), 34.9 (C(CH₃)₂), 32.1 (C(CH₃)₃), 28.6 (CyH₂), 28.4 (CH₃), 28.3 (CyH₂), 27.7, 26.9, 26.9 21.4 (C(CH₃)₂, C(CH₃)₃, CyCH₂).



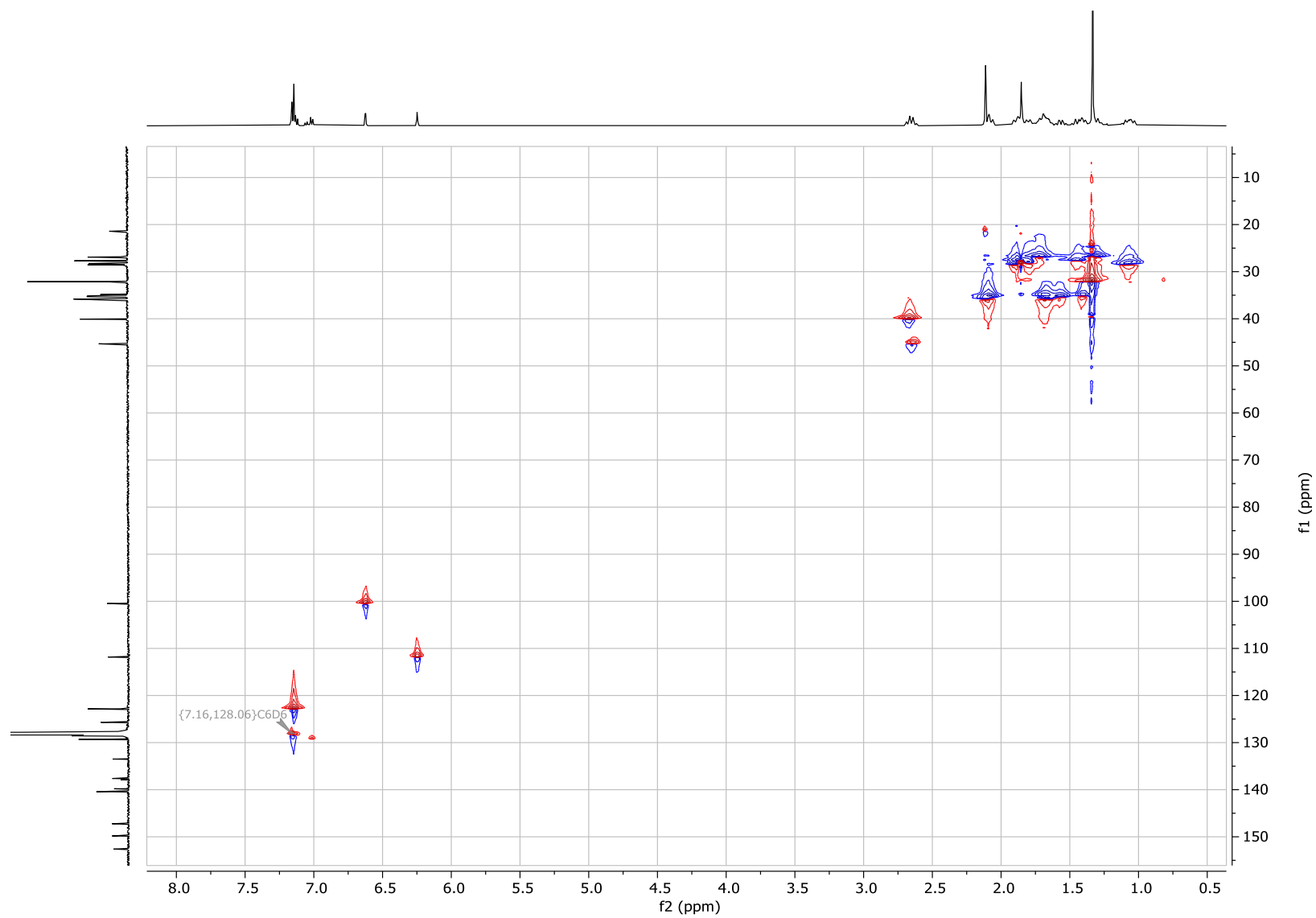
Supplementary figure 6. ^1H NMR spectrum (500 MHz, C_6D_6) of $(\text{xNON}^{\text{TCHP}})\text{K}_2$, (**38**).



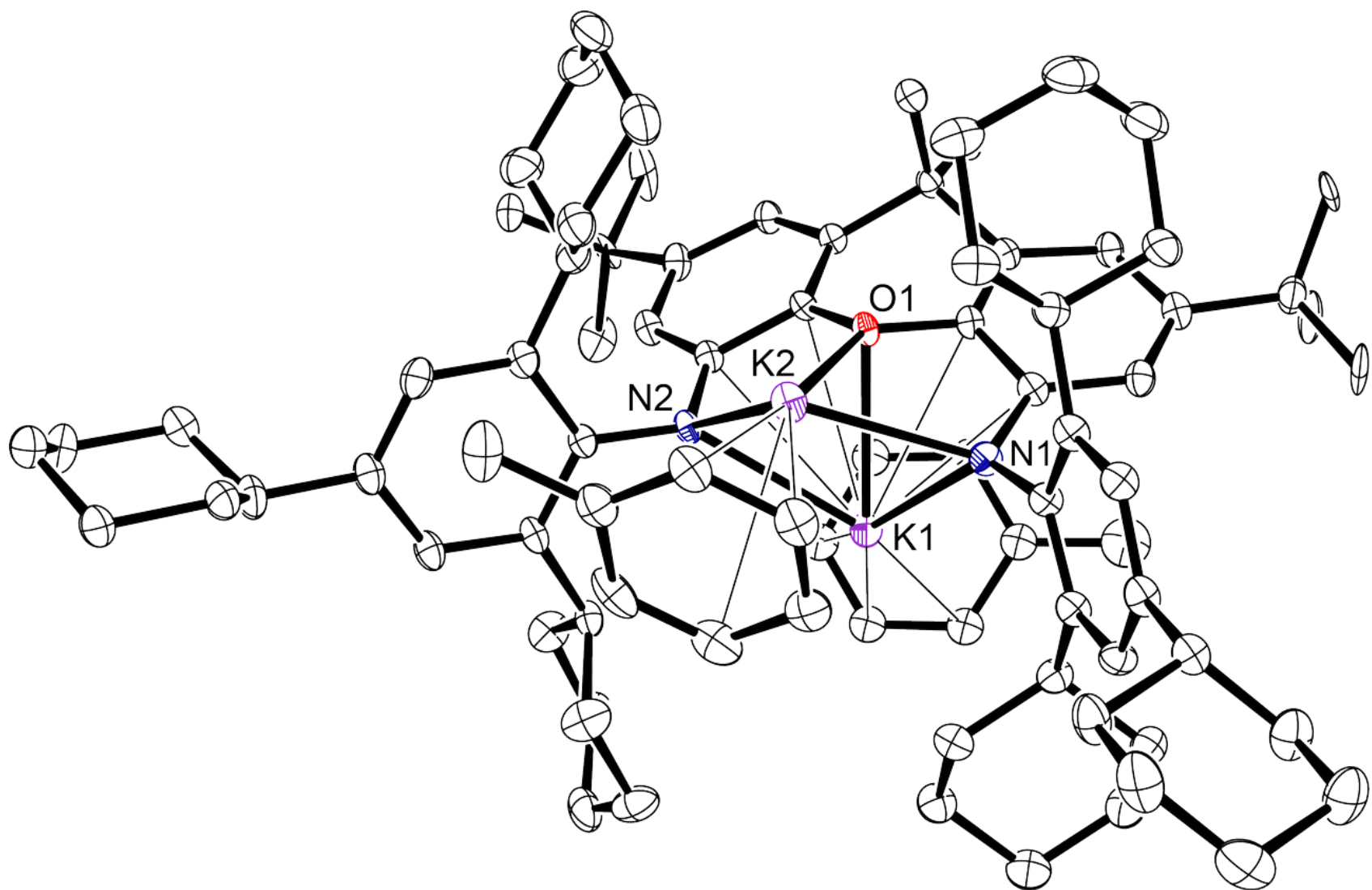
Supplementary figure 7. ¹³C NMR spectrum (126 MHz, C₆D₆) of (xNON^{TCHP})K₂ (**38**).



Supplementary figure 8. ^1H - ^1H COSY NMR spectrum (500 MHz, C_6D_6) of $(\text{xNON}^{\text{TCHP}})\text{K}_2$ (**38**).



Supplementary figure 9. ^1H - ^{13}C HSQC NMR spectrum (500 MHz, C_6D_6) of $(\text{xNON}^{\text{TCHP}})\text{K}_2$ (**38**)



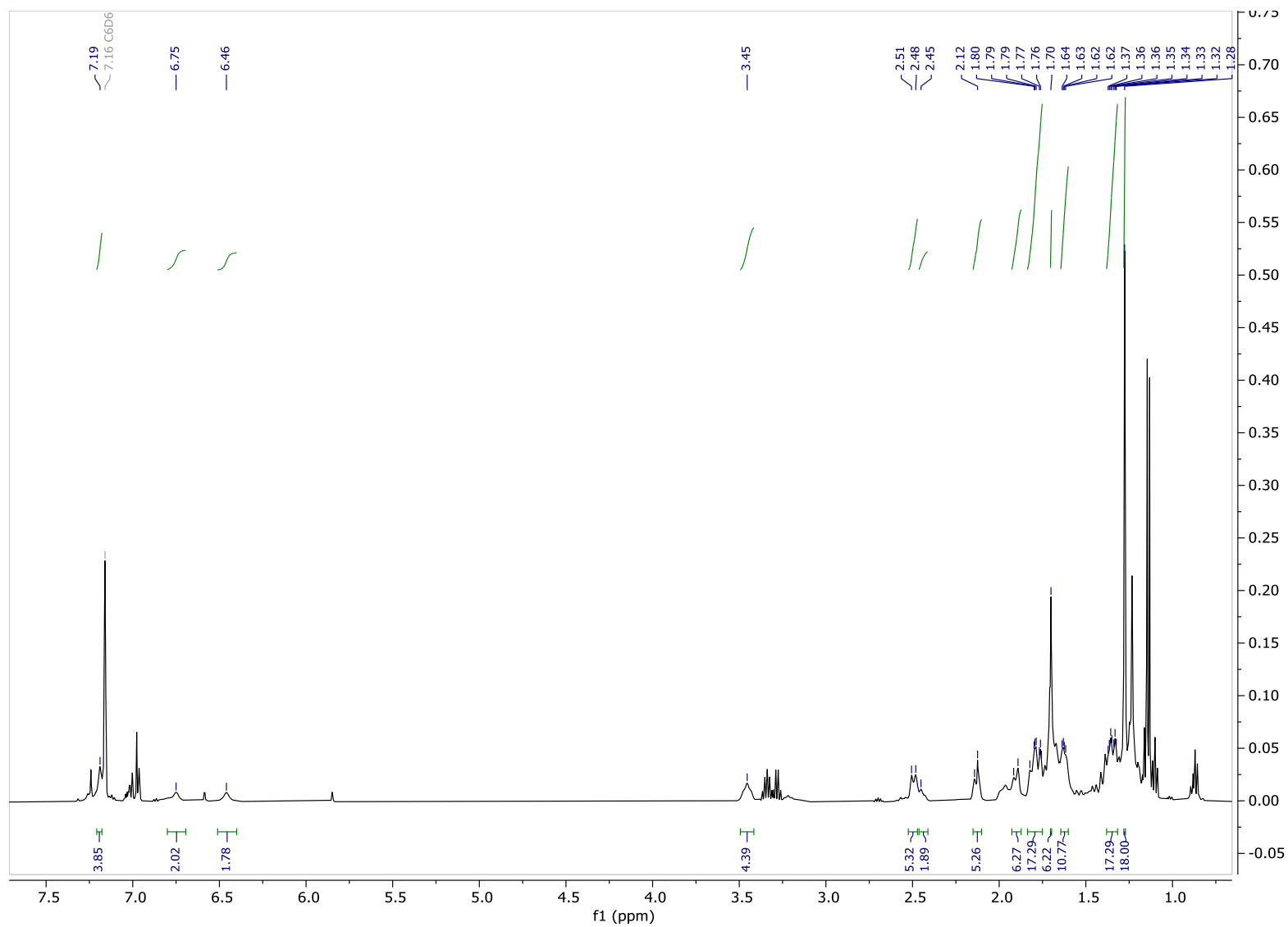
Supplementary figure 10. Ortep representation (ellipsoid 30% probability) of $(x\text{NON}^{\text{TCHP}})\text{K}_2$ (**38**). Hydrogen atoms have been omitted for clarity.

Preparation of $(x\text{NON}^{\text{TCHP}})\text{InI}_2\text{K}$, (39)

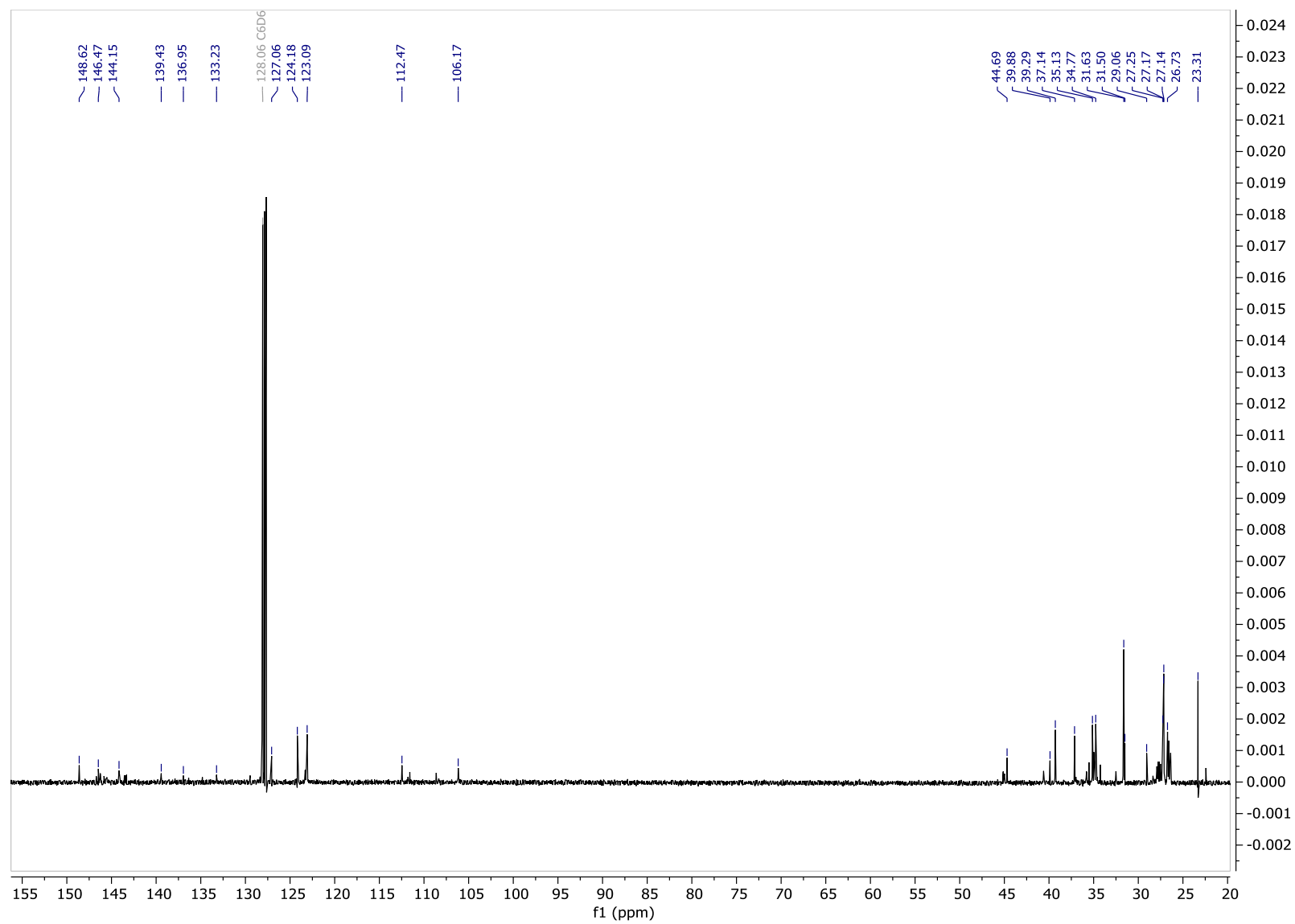
A solution of $x\text{NON}^{\text{TCHP}}\text{K}_2$ (425 mg, 0.400 mmol) in a 1:5 hexane: diethyl ether solution was added to InI_3 (198.7 mg, 0.400 mmol) in a 1:5 hexane: diethyl ether solution and stirred for 18 hours under nitrogen at room temperature. The mixture was filtered *via* a cannula to give a yellow/green solution. The solvent was removed *in vacuo* and the resulting green powder was heated under vacuum (10^{-2} mbar) to remove the coordinated diethyl ether and hexane yielding “ $(x\text{NON}^{\text{TCHP}})\text{InI}_2\text{K}$ ”. Recrystallisation was achieved from a saturated toluene solution to give light green crystals. Yield 504.1 mg, 92.1 %.

^1H NMR (500 MHz, C_6D_6) δ 7.19 (s, 4H, TCHP–ArH), 6.75 (s, 2H, XA–*p*–CH), 6.46 (s, 2H, XA–*o*–CH), 3.45 (s, 4H, Phen–*p*–CH), 2.50 (d, $J = 12.2$ Hz, 5H, CyH₂), 2.45 (s, 2H, Phen–*m*–CH), 2.13 (d, $J = 8.9$ Hz, 5H, CyH₂), 1.90 (d, $J = 12.6$ Hz, 6H, CyH₂), 1.82 – 1.76 (m, 17H, CyH₂), 1.70 (s, 6H, C(CH₃)₂), 1.64 – 1.62 (m, 10H, CyH₂), 1.34 (dt, $J = 12.6, 3.7$ Hz, 17H, CyH₂), 1.28 (s, 18H, C(CH₃)₃).

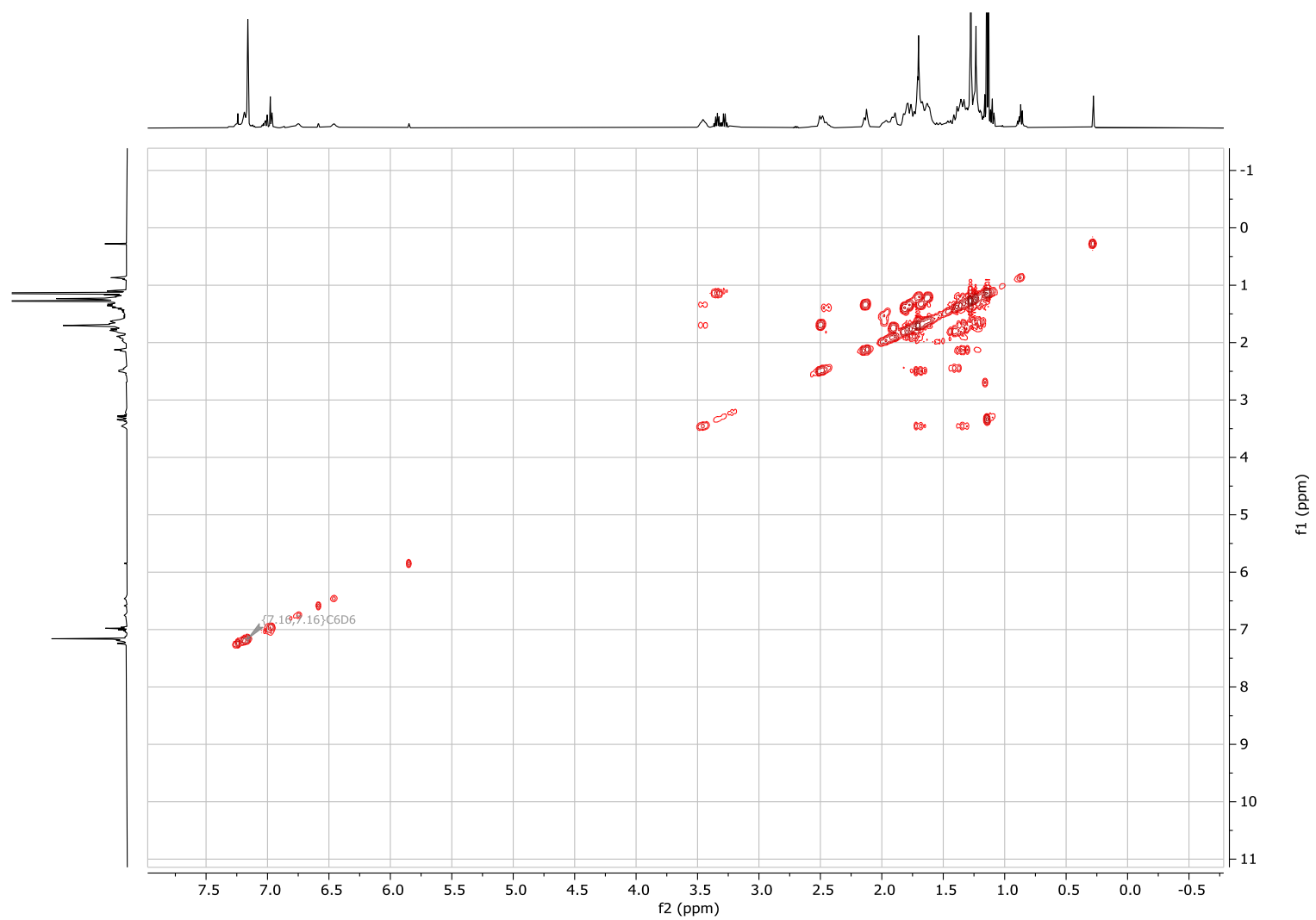
$^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, C_6D_6) δ 148.62, 146.47, 144.15, 139.43, 136.95, 133.23, 127.06, 124.18 (ArC), 123.09 (TCHP–ArC), 112.47 (XA–*o*–CH), 106.17 (XA–*p*–CH), 44.69 (TCHP–*p*–CyH), 39.88 (CyH₂), 39.29 (TCHP–*o*–CyH), 37.14, 35.13, 34.77 (CyCH₂), 31.63 C(CH₃)₃, 31.50, 29.06 (C(CH₃)₃, CyCH₂), 27.25 C(CH₃)₂, 27.17, 27.14, 26.73, 23.31 (C(CH₃)₃, CyCH₂, C(CH₃)₂).



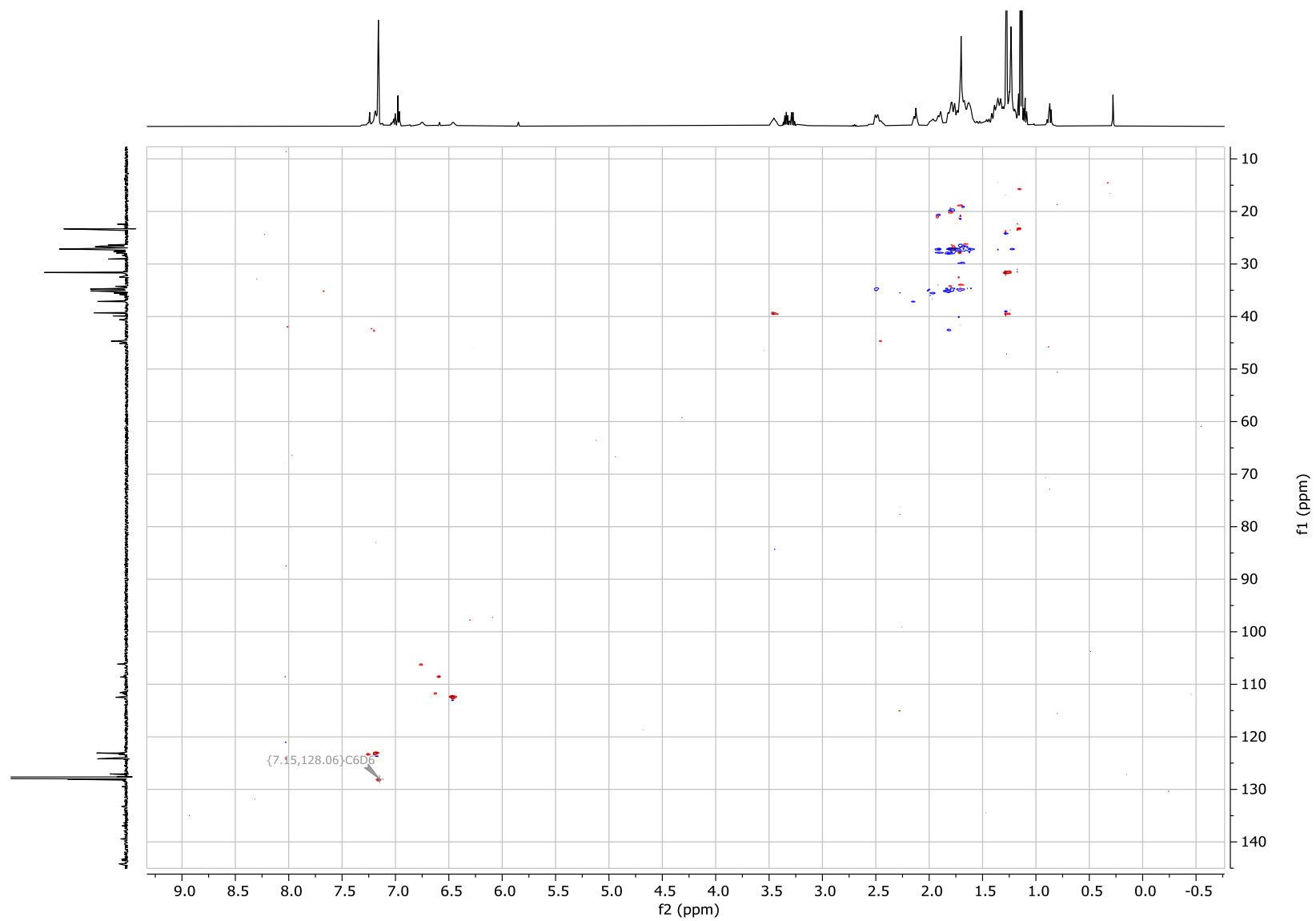
Supplementary figure 11. ^1H NMR spectrum (500 MHz, C_6D_6) of $(\text{xNON}^{\text{TCHP}})\text{InI}_2\text{K}$, (**39**).



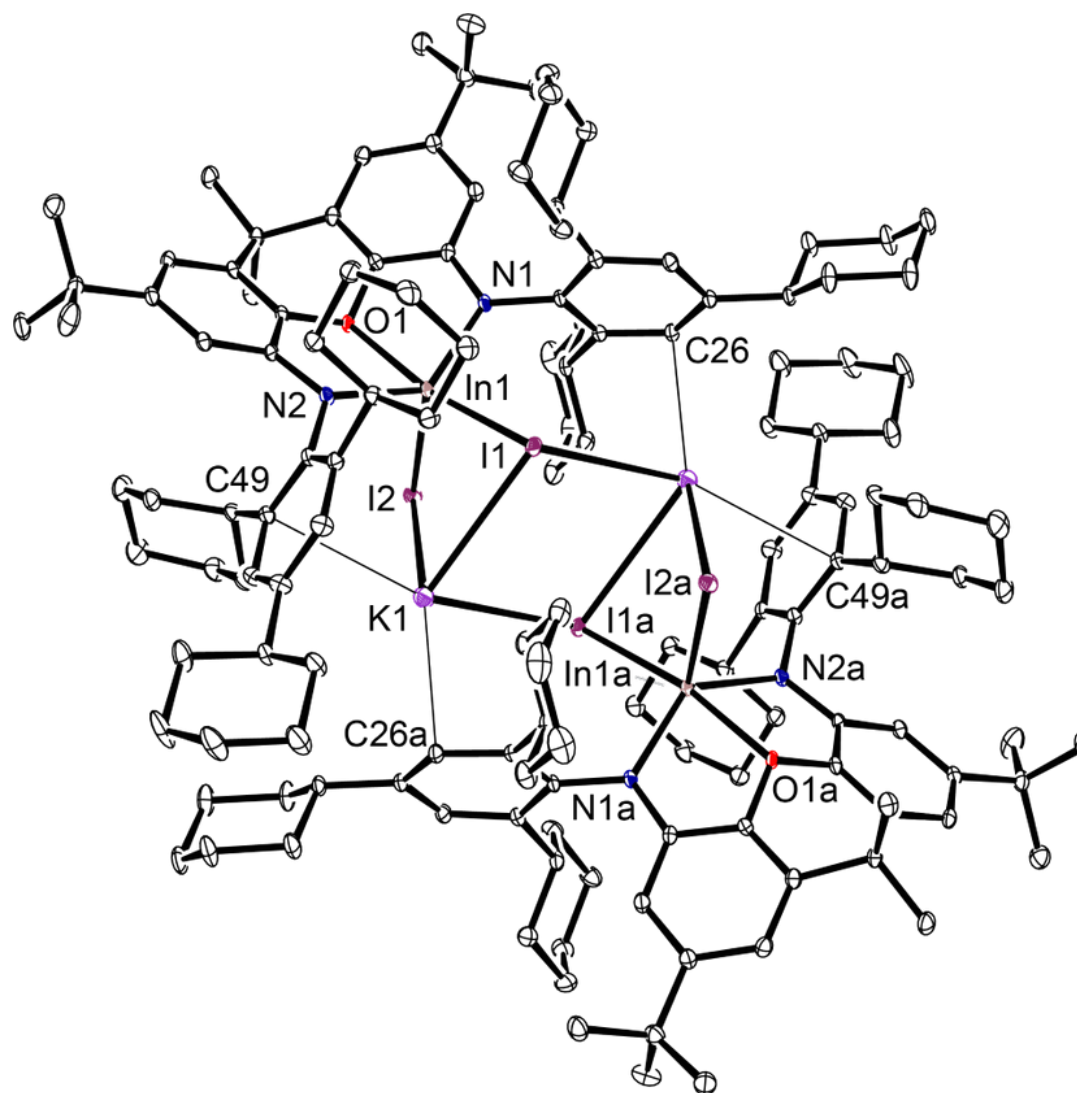
Supplementary figure 12. ¹³C NMR spectrum (126 MHz, C₆D₆) of (xNON^{TCHP})InI₂K (**39**).



Supplementary figure 13. ^1H - ^1H COSY NMR spectrum (500 MHz, C_6D_6) of $(\text{xNON}^{\text{TCHP}})\text{InI}_2\text{K}$ (**39**).



Supplementary figure 14. ^1H - ^{13}C HSQC NMR spectrum (500 MHz, C_6D_6) of $(x)\text{NON}^{\text{TCHP}}\text{InI}_2\text{K}$ (**39**).



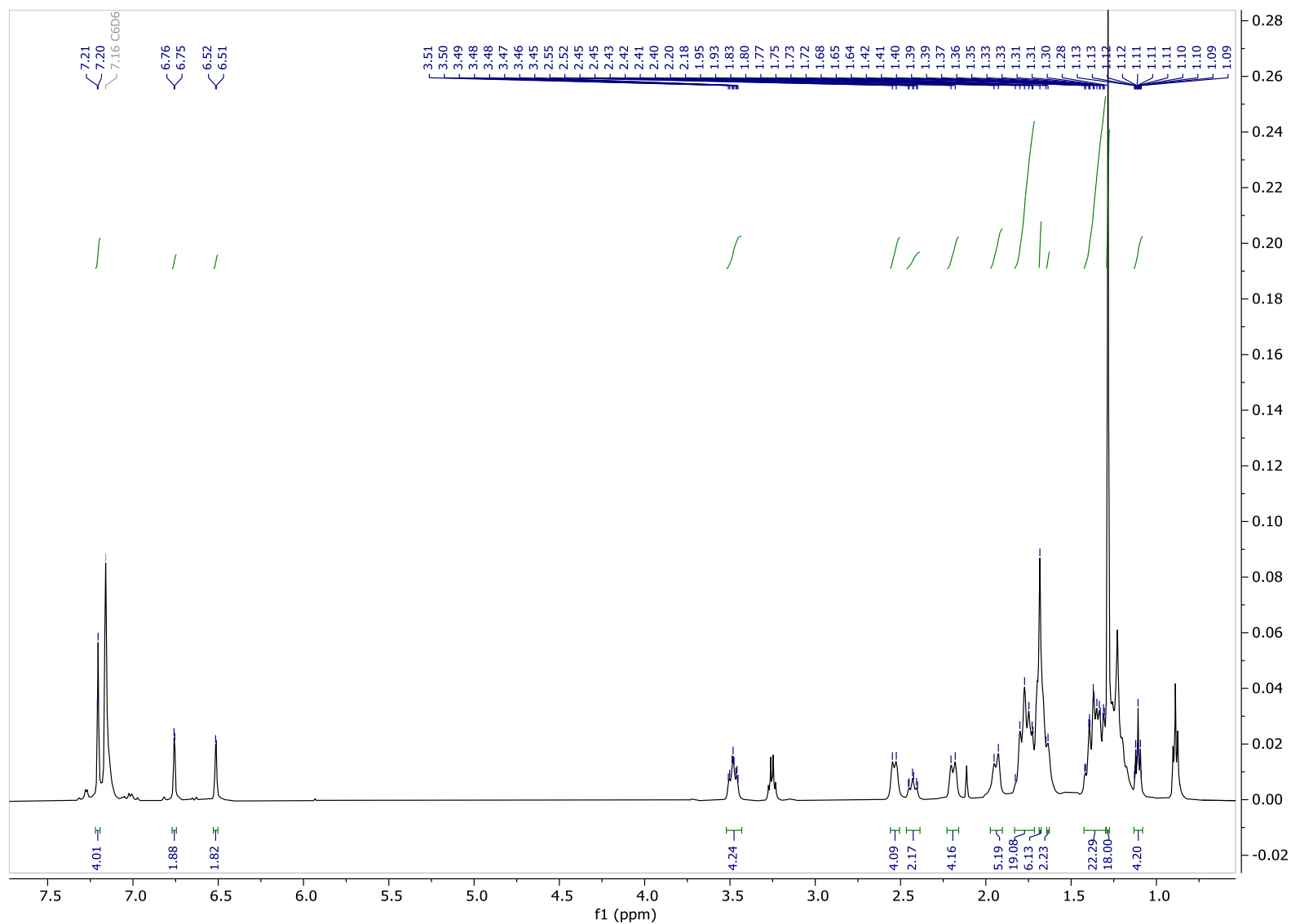
Supplementary figure 15. Ortep representation (ellipsoid 30% probability) of $(x\text{NON}^{\text{TCHP}})\text{InI}_2\text{K}$ (**39**). Hydrogen atoms have been omitted for clarity.

Preparation of (xNON^{TCHP})InK(Et₂O)₂(THF)₂, (40)

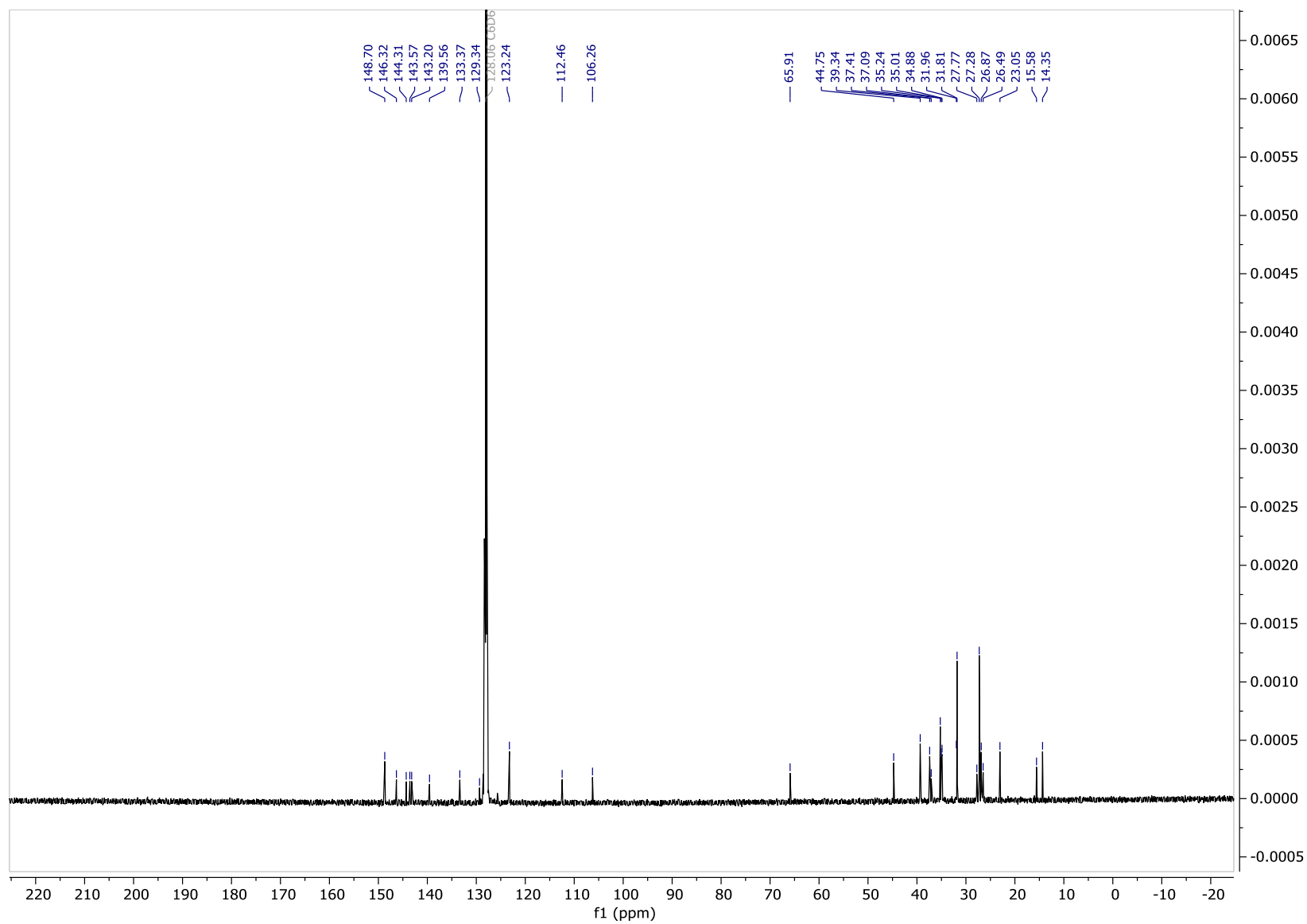
A solution of (xNON^{TCHP})InI₂K (500 mg, 0.360 mmol) in a 1:1 hexane: diethyl ether solution was added to KC₈ (97.3 mg, 0.720 mmol) in a 1:1 hexane: diethyl ether solution and stirred for 18 hours under nitrogen at room temperature. The solution was filtered through celite, the solvent was removed *in vacuo* and the residue was washed three times in hexane. The resulting light-yellow powder and hexane solution were concentrated *in vacuo* and the powder was extracted with THF and left to crystallise at room temperature, giving bright yellow crystals suitable for an X-ray diffraction experiment. Yield 251.9 mg, 53.5 %.

¹H NMR (500 MHz, C₆D₆) δ 7.21 (s, 4H, Ar-*H*), 6.76 (d, J = 2.1 Hz, 2H, XA-*p*-CH), 6.51 (d, J = 2.2 Hz, 2H, XA-*o*-CH), 3.48 (tt, J = 8.9, 3.6 Hz, 4H, *p*-CyH), 2.54 (d, J = 11.4 Hz, 4H), 2.46 – 2.38 (m, 2H, *o*-CyH), 2.19 (d, J = 12.5 Hz, 4H, CyH₂), 1.94 (d, J = 12.7 Hz, 5H, CyH₂), 1.76 (q, J = 11.6 Hz, 19H, CyH₂), 1.68 (s, 6H, CH₃), 1.65 – 1.62 (m, 2H, CyH₂), 1.44 – 1.30 (m, 22H, CyH₂), 1.28 (s, 18H, CH(CH₃)₃), 1.11 (ddt, J = 9.5, 6.7, 2.3 Hz, 4H, CyH₂).

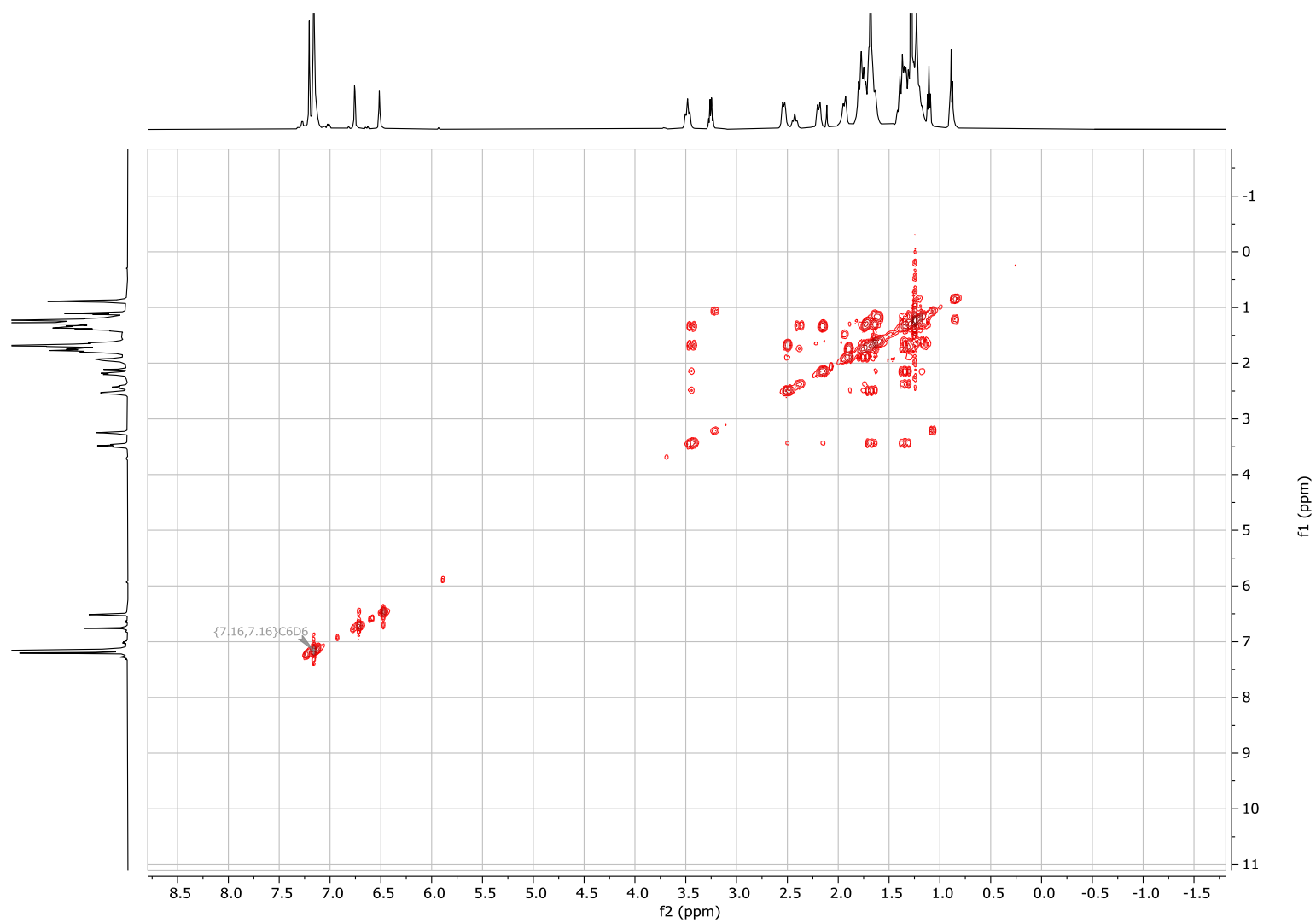
¹³C{¹H} NMR (126 MHz, C₆D₆) δ 148.39, 146.01, 144.00, 143.26, 142.89, 139.25, 133.06, 129.03 (*Ar*), 122.93 (*ArC*), 112.15 (XA-*p*-CH), 105.95 (XA-*o*-CH), 65.60 (*Cy*), 44.44 (*p*-CyH), 39.03 (*o*-CyH), 37.10, 36.78, 34.93, 34.70, 34.57 (C(CH₃)₂, C(CH₃)₃, CyCH₂), 31.65 (C(CH₃)₃), 31.50, 27.46, 26.97 (C(CH₃)₂), 26.56, 22.74, 15.27, 14.04 (C(CH₃)₂, C(CH₃)₃, CyCH₂).



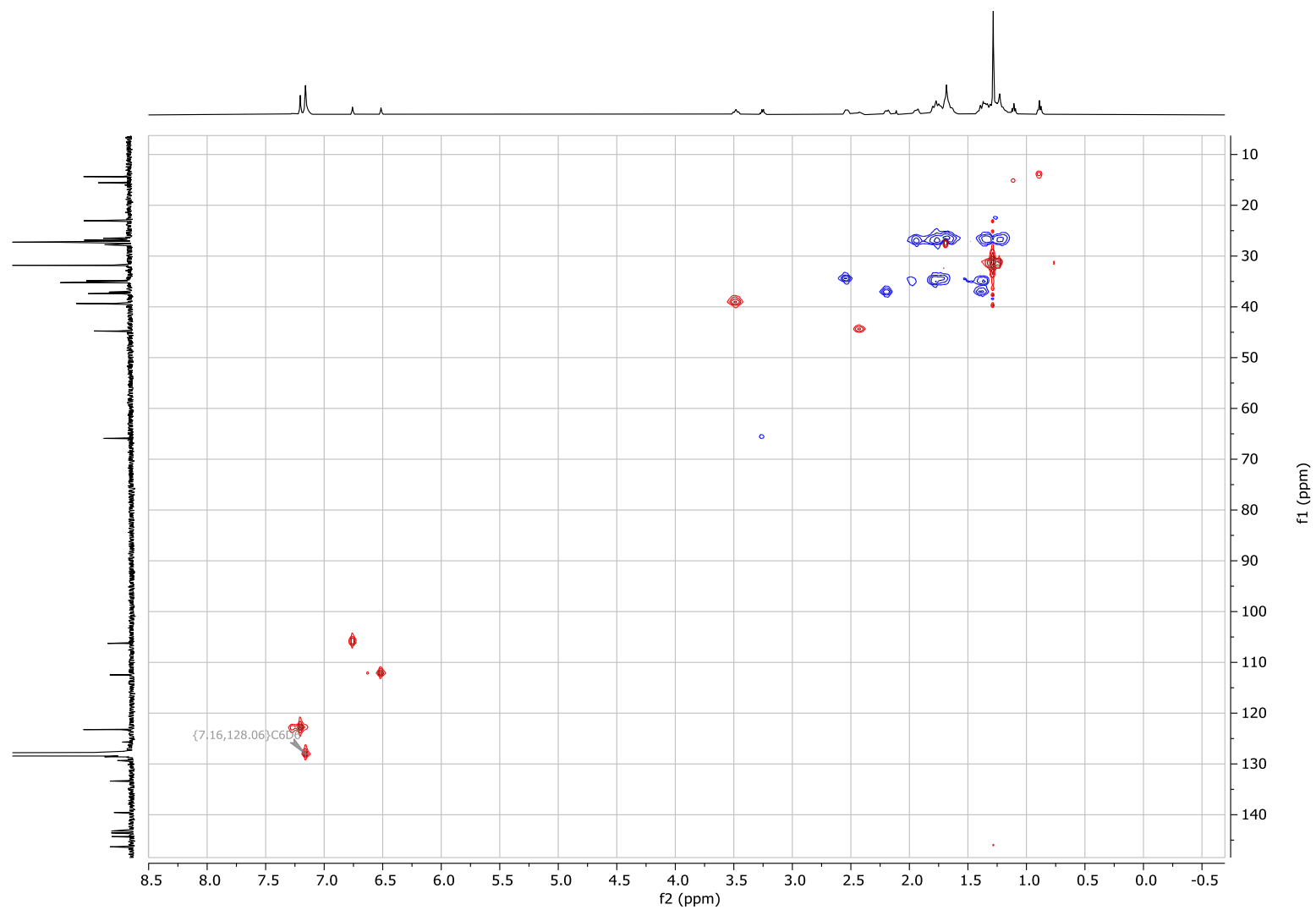
Supplementary figure 16. ^1H NMR spectrum (500 MHz, C_6D_6) of $(\text{xNON}^{\text{TCHP}})\text{K}(\text{Et}_2\text{O})_2(\text{THF})_2$ (**40**).



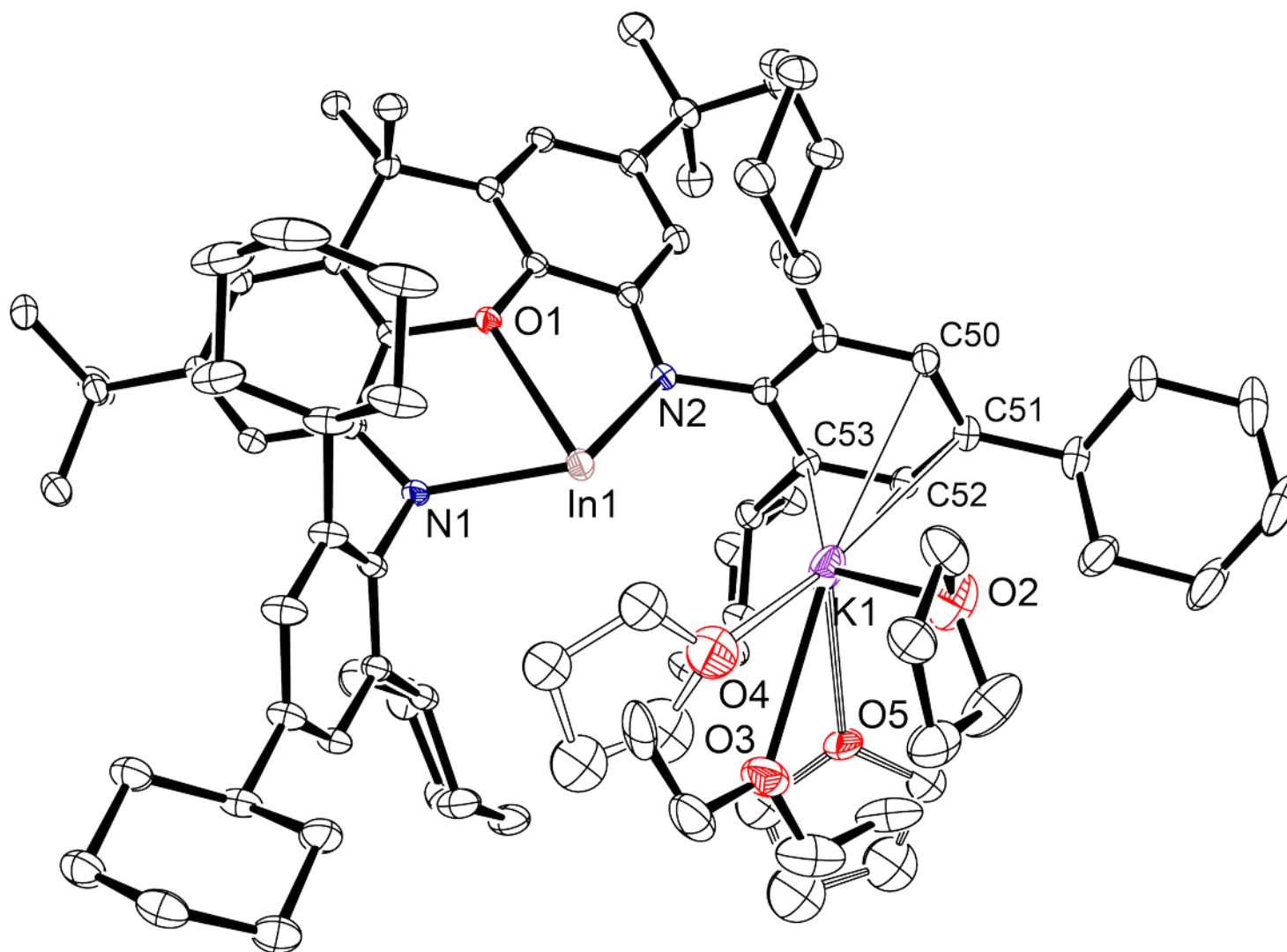
Supplementary figure 17. ¹³C NMR spectrum (126 MHz, C₆D₆) of (xNON^{TCHP})K(Et₂O)₂(THF)₂ (**40**).



Supplementary figure 18. ^1H - ^1H COSY NMR spectrum (500 MHz, C_6D_6) of $(\text{xNON}^{\text{TCHP}})\text{K}(\text{Et}_2\text{O})_2(\text{THF})_2$ (**40**).



Supplementary figure 19. ^1H - ^{13}C HSQC NMR spectrum (500 MHz, C_6D_6) of $(\text{xNON}^{\text{TCHP}})\text{K}(\text{Et}_2\text{O})_2(\text{THF})_2$ (**40**).



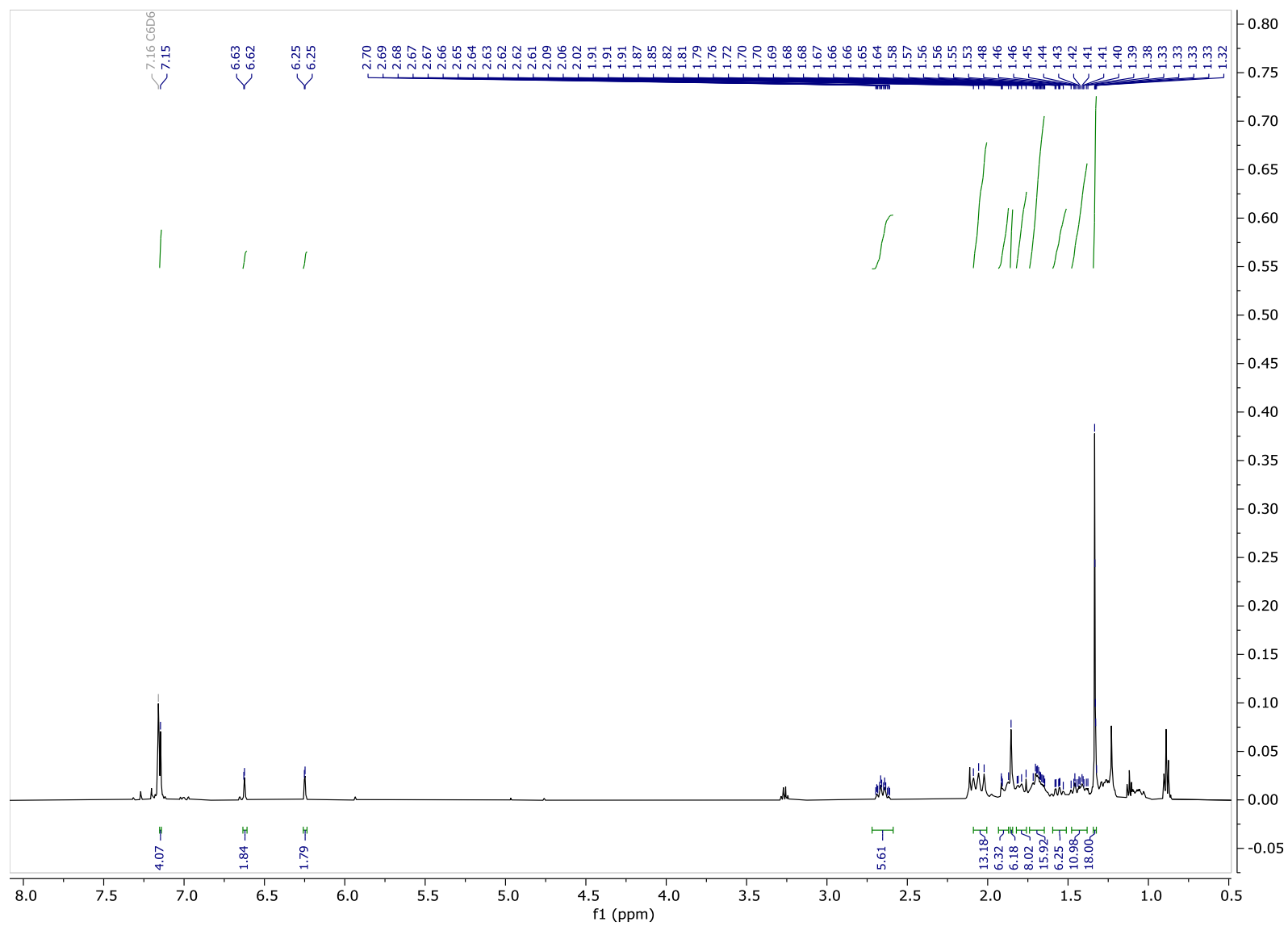
Supplementary figure 20. Ortep representation (ellipsoid 30% probability) of $(x\text{NON}^{\text{TCHP}})\text{K}(\text{Et}_2\text{O})_2(\text{THF})_2$. (**40**). Hydrogen atoms have been omitted for clarity.

Preparation of (xNON^{TCHP})InK(Cp^{4*}), (46)

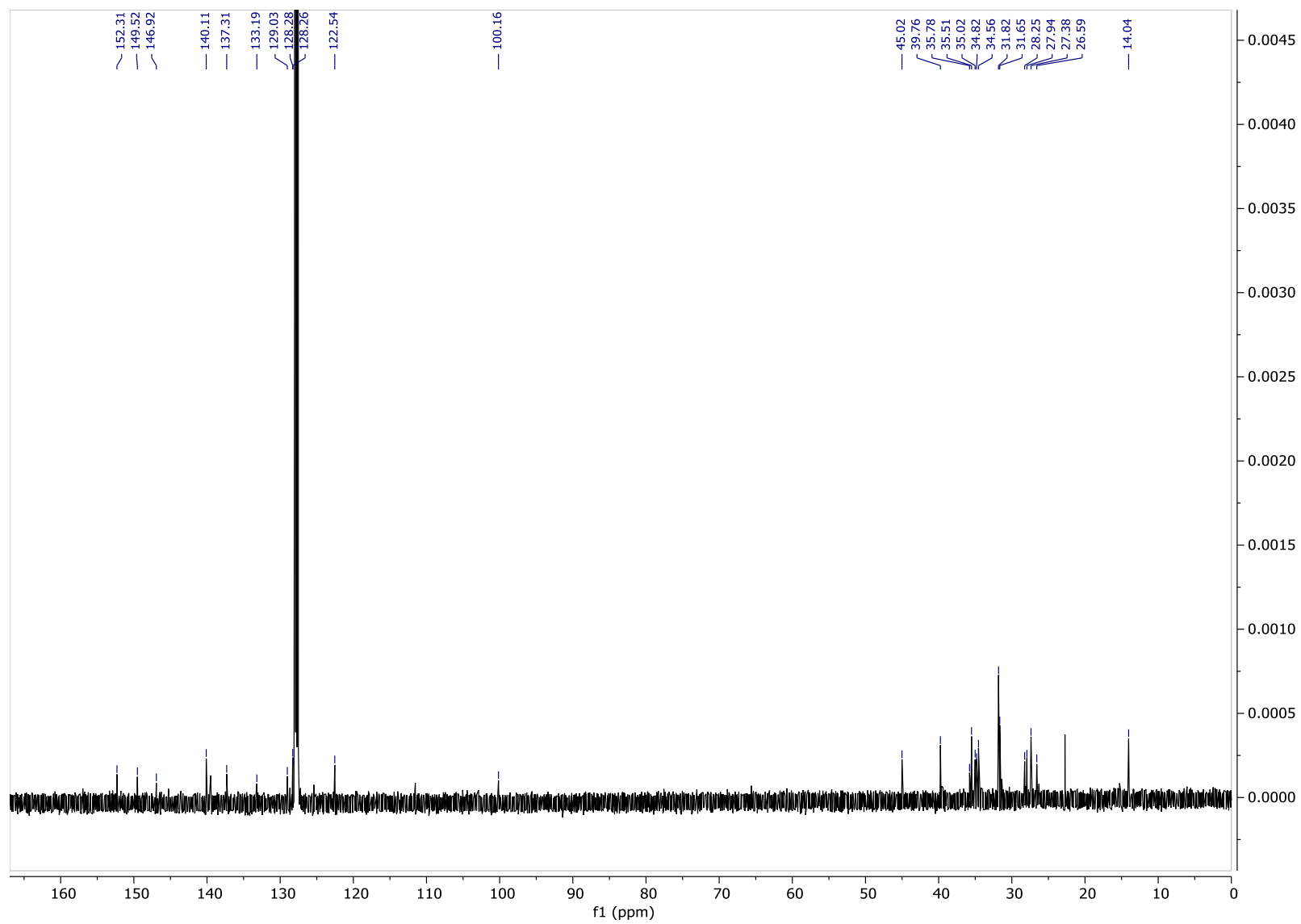
A deuterated benzene (C₆D₆) solution of Cp^{4*}In (10 mg, 0.04 mmol) was added to a scintillation vial containing (xNON^{TCHP})K₂ (45 mg, 0.04 mmol) in C₆D₆ and transferred to an *J Youngs* NMR tube to be heated at 60°C for 2 hours under nitrogen. The solvent was removed *in vacuo* and the residue was extracted with toluene. The solution was filtered through celite, concentrated, and left to crystallise at room temperature, giving bright yellow crystals suitable for an X-ray diffraction experiment. Yield 13.4 mg, 24.1 %.

¹H NMR (500 MHz, C₆D₆) δ 7.15 (s, 4H, ArH), 6.63 (d, *J* = 2.2 Hz, 2H, XA-*p*-CH), 6.25 (d, *J* = 2.2 Hz, 2H, XA-*o*-CH), 2.65 (m, 6H, CyH), 2.09 – 2.02 (m, 13H, CyH₂), 1.92 – 1.87 (m, 6H, CyH₂), 1.85 (s, 6H, C(CH₃)₂), 1.83 – 1.76 (m, 8H, CyH₂), 1.75 – 1.61 (m, 16H, CyH₂), 1.61 – 1.50 (m, 6H, CyH₂), 1.49 – 1.37 (m, 11H, CyH₂), 1.33 (s, 18H, C(CH₃)₃).

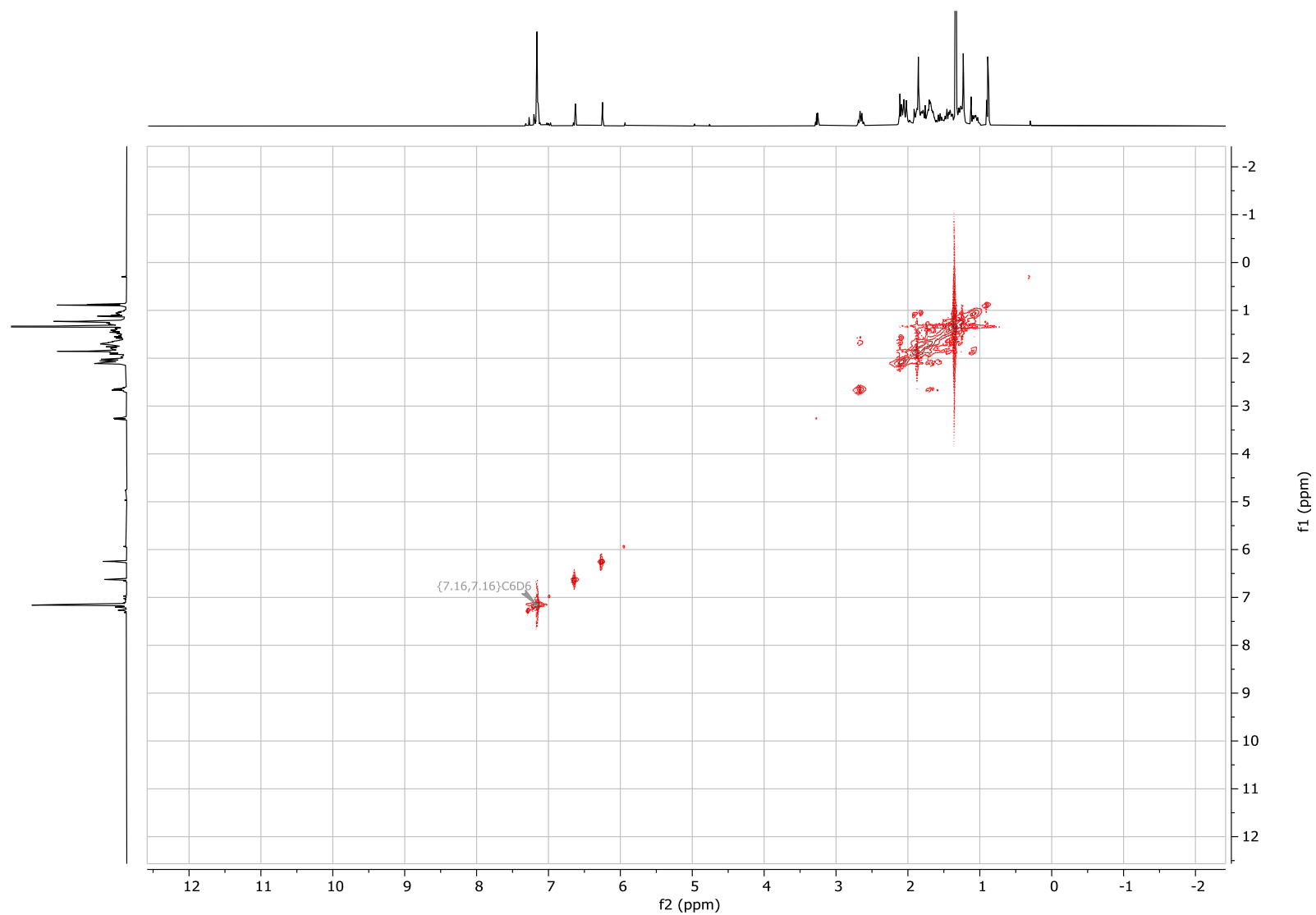
¹³C {¹H} NMR (126 MHz, C₆D₆) δ 152.3, 149.5, 146.9, 140.1, 137.3, 133.2, 129.0, 128.3, 128.3 (*Ar*), 122.4 (*ArH*), 111.5 (XA-*p*-CH), 100.2 (XA-*o*-CH), 45.0, 39.8 (CyH), 35.8, 35.5, 35.0, 34.8, 34.6 (C(CH₃)₂, C(CH₃)₃, CyCH₂), 31.8 C(CH₃)₃, 31.7, 28.3, 27.9, 27.4, 26.4, 14.0 (C(CH₃)₂, C(CH₃)₃, CyCH₂).



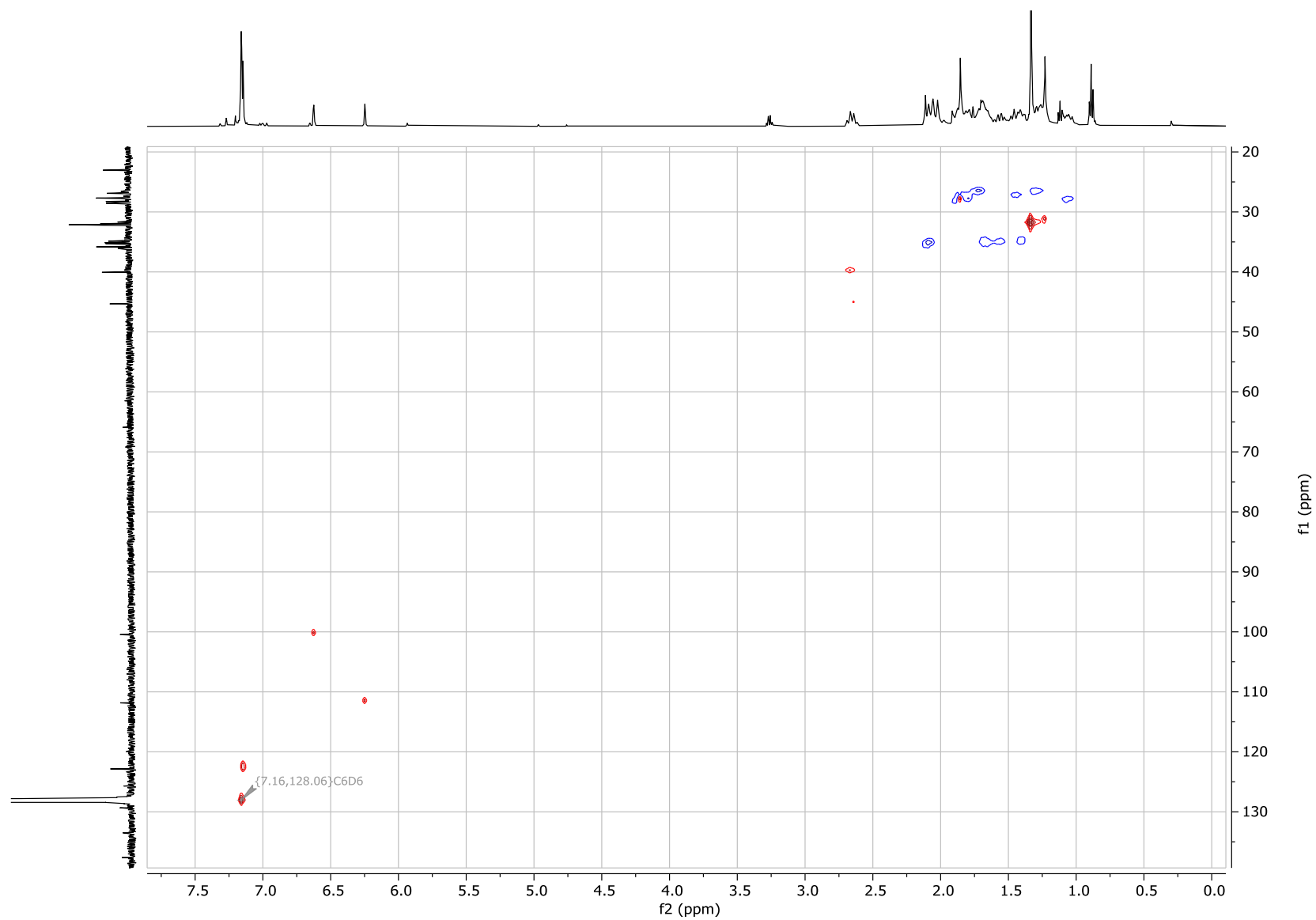
Supplementary figure 21. ¹H NMR spectrum (500 MHz, C₆D₆) of (xNON^{TCHP})InK(Cp^{4*}), (46).



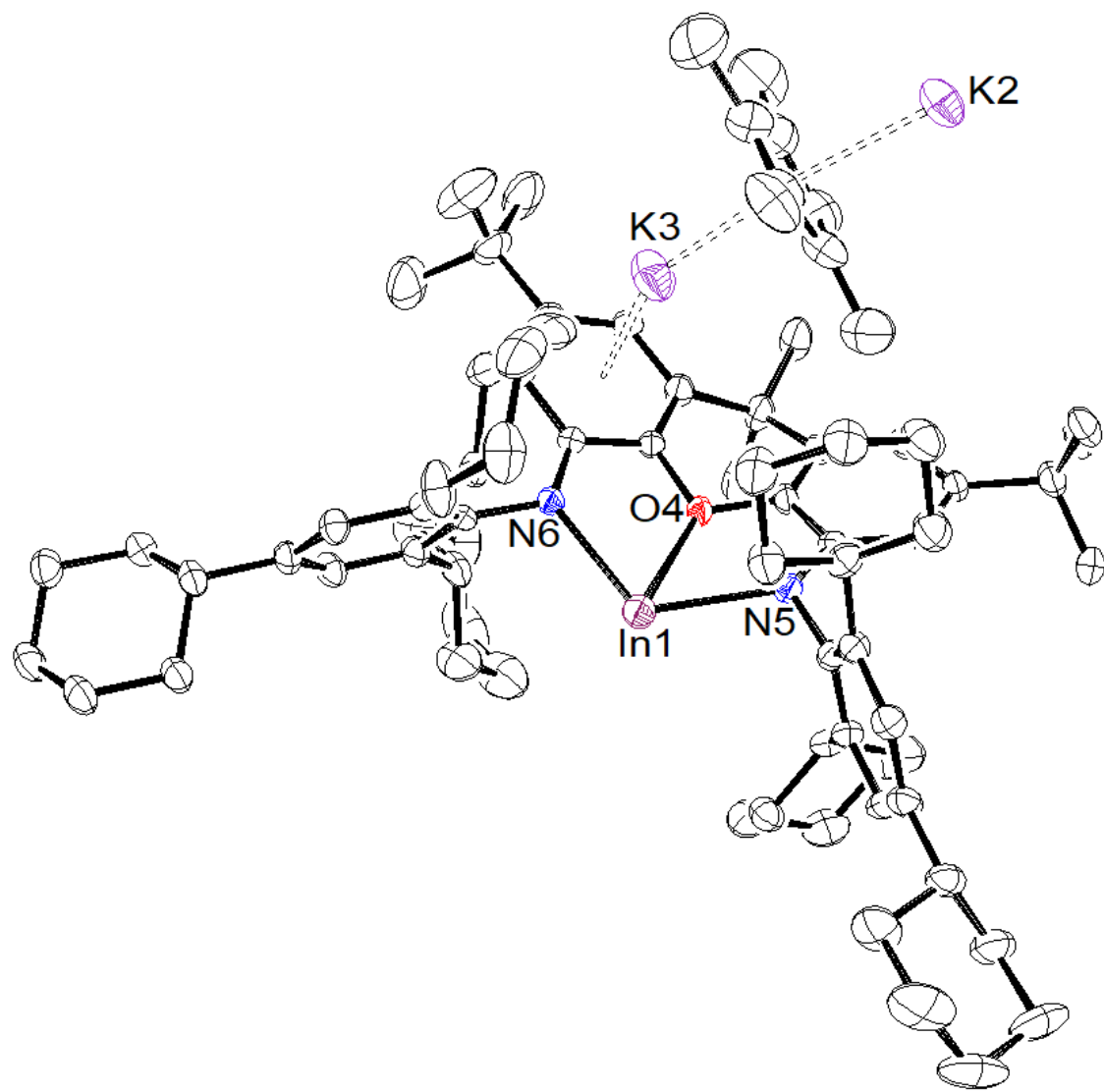
Supplementary figure 22. ¹³C NMR spectrum (126 MHz, C₆D₆) of (xNON^{TCHP})InK(Cp^{4*}), (46).



Supplementary figure 23. ^1H - ^1H COSY NMR spectrum (500 MHz, C_6D_6) of $(\text{xNON}^{\text{TCHP}})\text{InK}(\text{Cp}^{4*})$, (**46**).



Supplementary figure 24. ^1H - ^{13}C HSQC NMR spectrum (500 MHz, C_6D_6) of $(\text{xNON}^{\text{TCHP}})\text{InK}(\text{Cp}^{4*})$, (**46**).



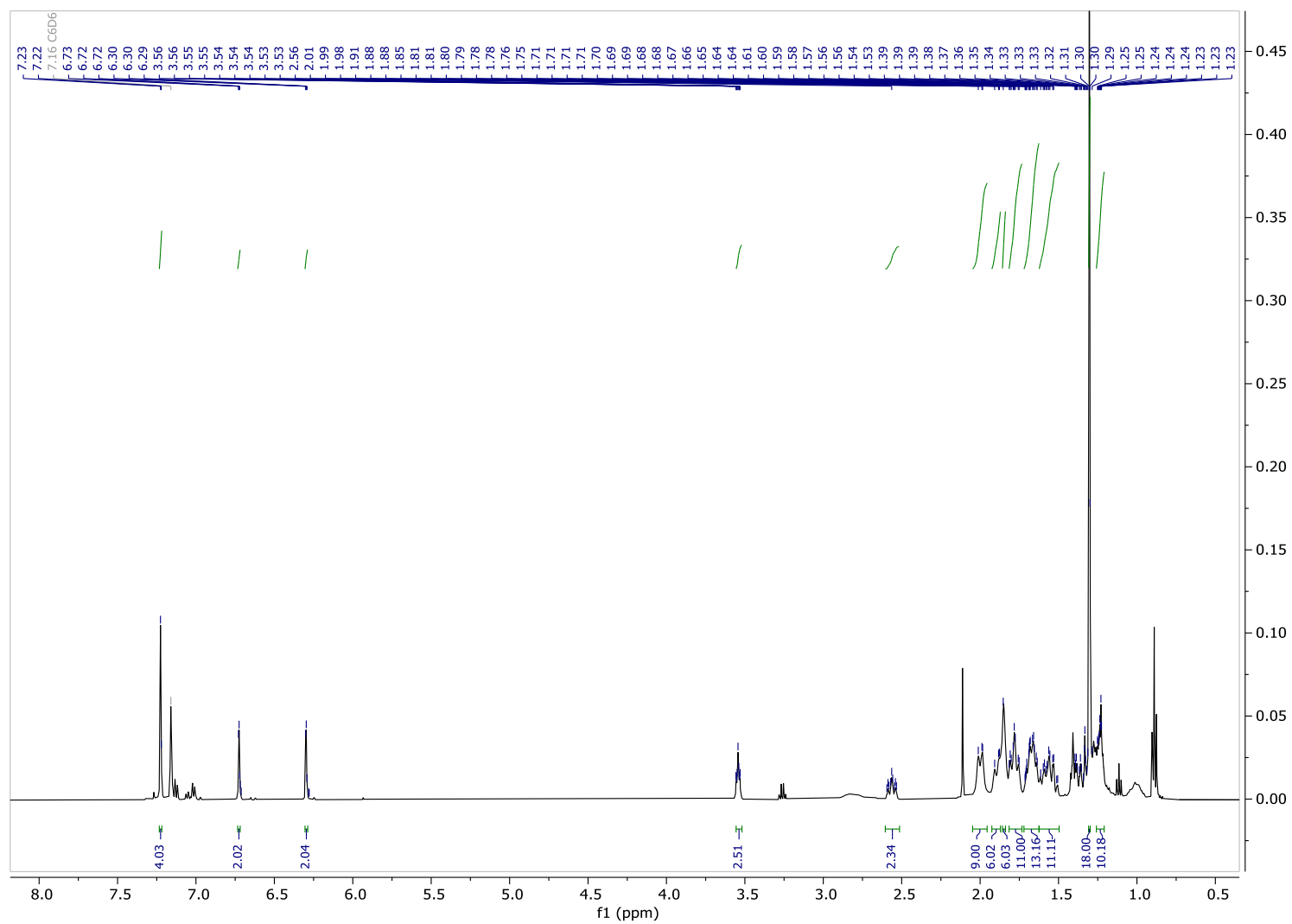
Supplementary figure 25. Ortep representation (ellipsoid 30% probability) of $(x\text{NON}^{\text{TCHP}})\text{InK}(\text{Cp}^{4+})$, (46). Hydrogen atoms have been omitted for clarity.

Preparation of (xNON^{TCHP})InK(tol), 47

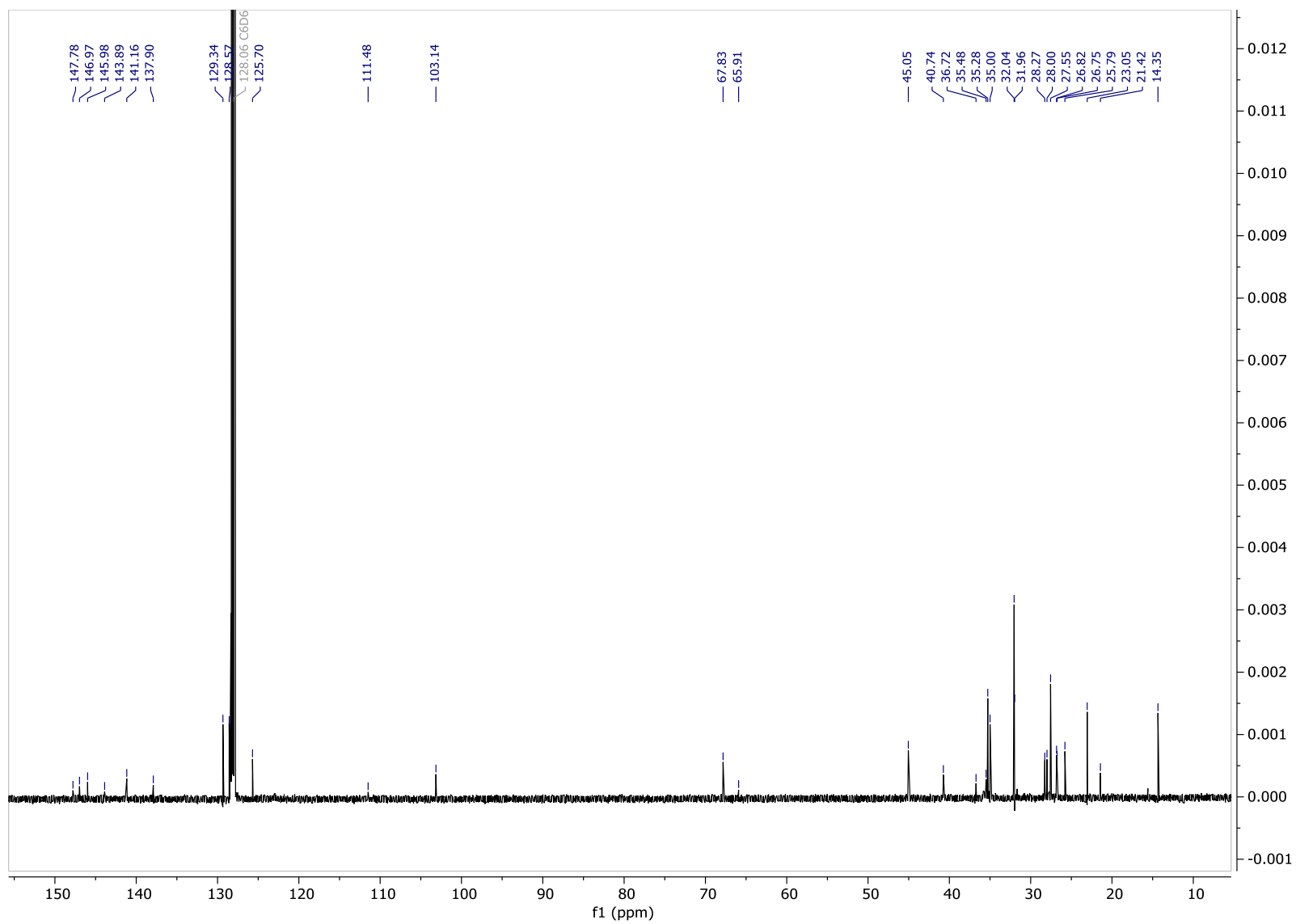
A solution of (xNON^{TCHP})K₂ (500 mg, 0.47 mmol) in THF was added to InI (114 mg, 0.47 mmol) in the dark and stirred for 48 hours under nitrogen at room temperature. The solution was filtered through celite, and the solvent was removed *in vacuo* and the residue was extracted with toluene. The solution was filtered again through celite, concentrated, and left to crystallise at room temperature, giving bright yellow crystals suitable for an X-ray diffraction experiment Yield 375.6 mg, 63.0%.

¹H NMR (500 MHz, C₆D₆) δ 7.22 (s, 4H, ArH), 6.72 (d, *J* = 2.1 Hz, 2H, XA-*p*-CH), 6.30 (d, *J* = 2.1 Hz, 2H, XA-*o*-CH), 3.56 – 3.51 (m, 3H, CyH), 2.56 (tt, *J* = 11.9, 3.3 Hz, 3H, CyH), 2.00 (d, 9H, CyH₂), 1.93 – 1.86 (m, 6H, CyH₂), 1.85 (s, 6H, C(CH₃)₂), 1.78 (td, *J* = 12.6, 6.2 Hz, 11H, CyH₂), 1.71 – 1.62 (m, 13H, CyH₂), 1.61 – 1.50 (m, 11H, CyH₂), 1.30 (s, 18H, C(CH₃)₃), 1.26 – 1.21 (m, 10H, CyH₂).

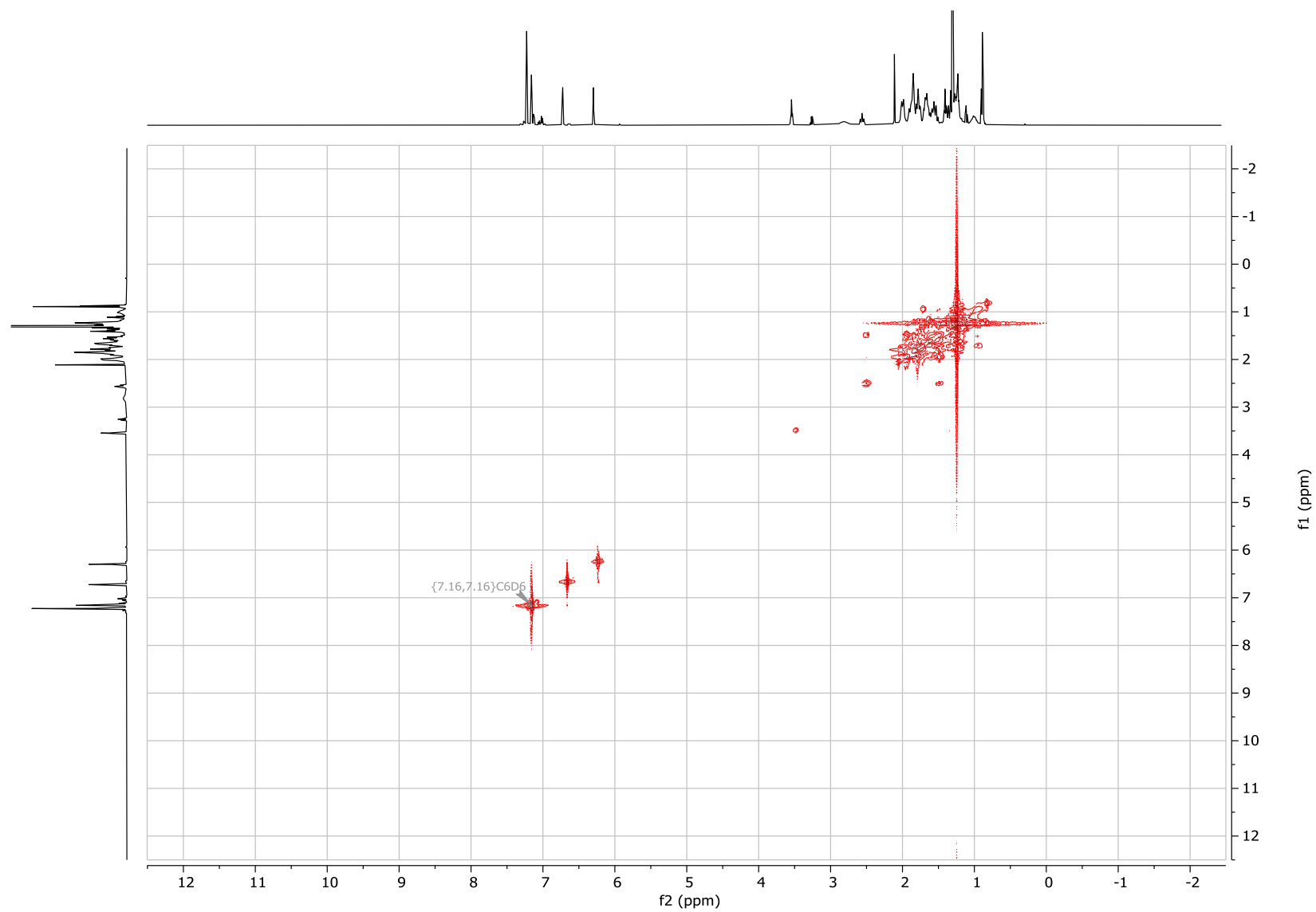
¹³C{¹H} NMR (126 MHz, C₆D₆) δ 147.8, 147.0, 146.0, 143.9, 141.2, 137.9, 129.3, 128.3, 128.6, 125.7(ArH), 111.5 (XA-*p*-CH), 103.1 (XA-*o*-CH), 67.83(*p*-CyH), 45.05 (*o*-CyH), 40.74, 36.72, 35.48, 35.28, 35.00 (CyH₂), 32.04 (C(CH₃)₃), 31.96, 28.27, 28.00 (CyH₂), 27.55 (C(CH₃)₂), 26.82, 26.75, 25.79, 23.05, 21.42, 14.35 (C(CH₃)₂, C(CH₃)₃, CyCH₂), (CyH₂).



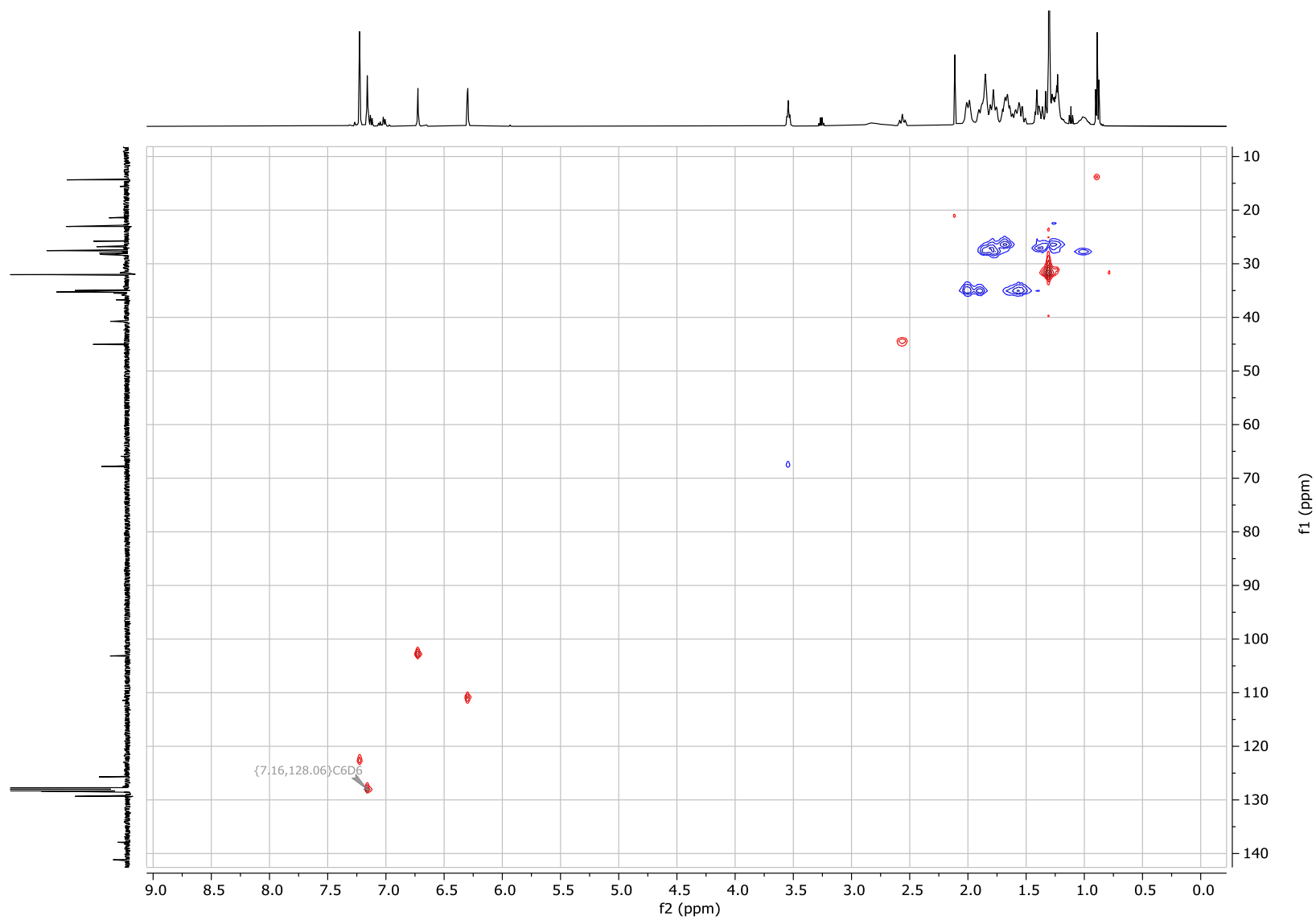
Supplementary figure 26. ^1H NMR spectrum (500 MHz, C_6D_6) of $(\text{xNON}^{\text{TCHP}})\text{InK}(\text{tol})$, (**47**).



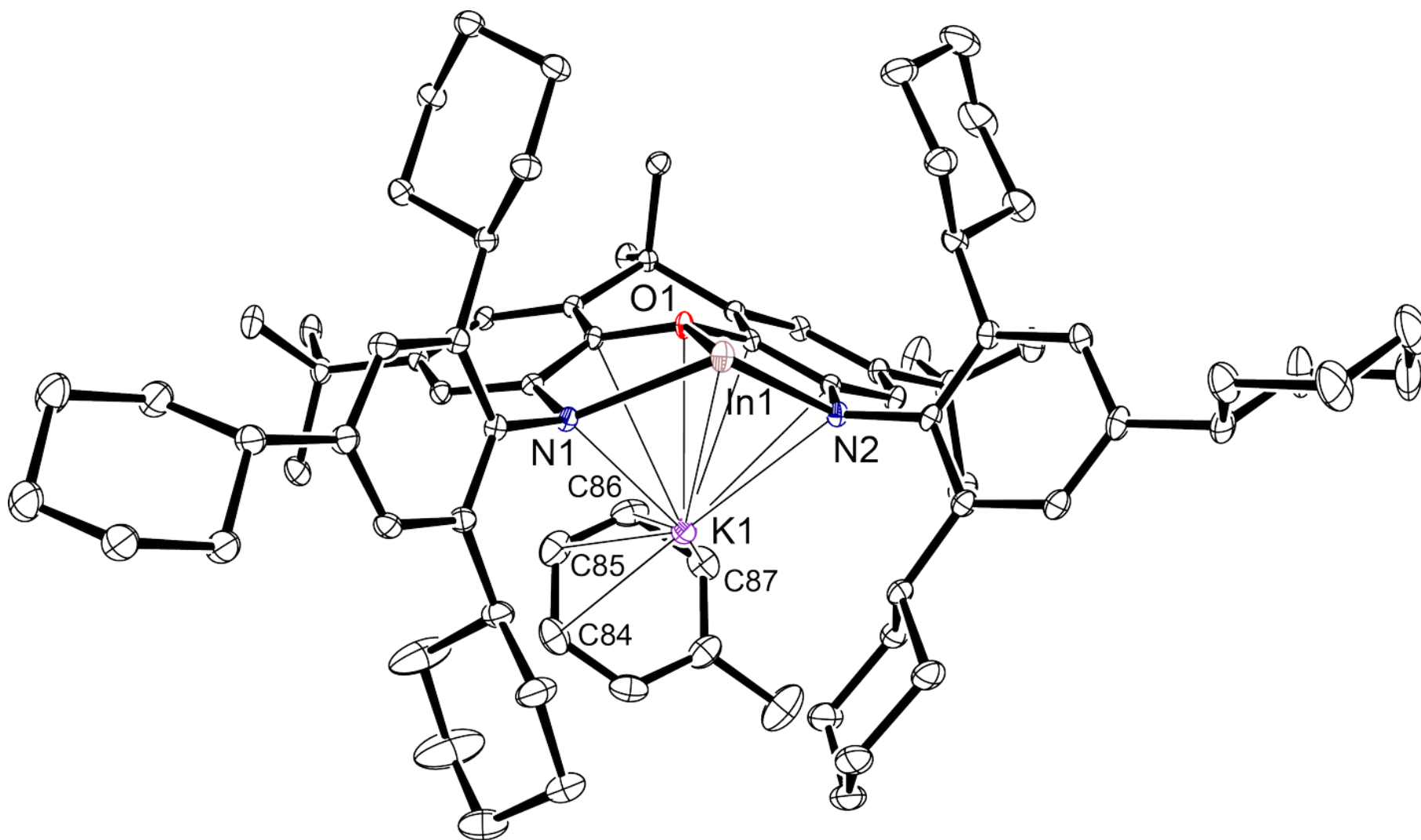
Supplementary figure 27. ¹³C NMR spectrum (126 MHz, C₆D₆) of (xNON^{TCHP})InK(tol), (47).



Supplementary figure 28. ^1H - ^1H COSY NMR spectrum (500 MHz, C_6D_6) of $(\text{xNON}^{\text{TCHP}})\text{InK}(\text{tol})$, (**47**).



Supplementary figure 29. ^1H - ^{13}C HSQC NMR spectrum (500 MHz, C_6D_6) of $(\text{xNON}^{\text{TCHP}})\text{InK}(\text{tol})$, (**47**).



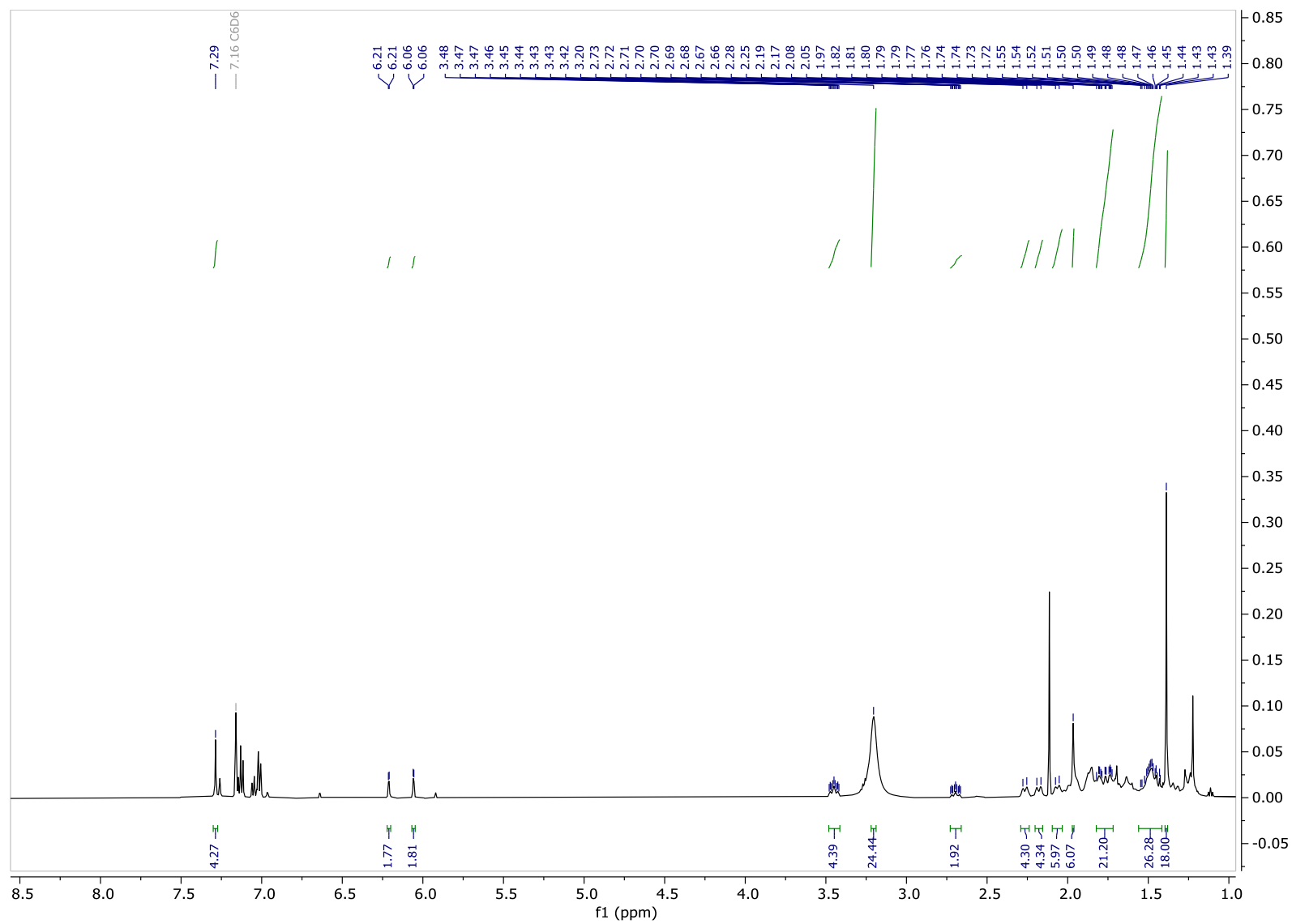
Supplementary figure 30. Ortep representation (ellipsoid 30% probability) of (xNON^{TCHP})InK(tol), (**47**). Hydrogen atoms have been omitted for clarity.

Preparation of (xNON^{TCHP})InK (18-c-6K), (48)

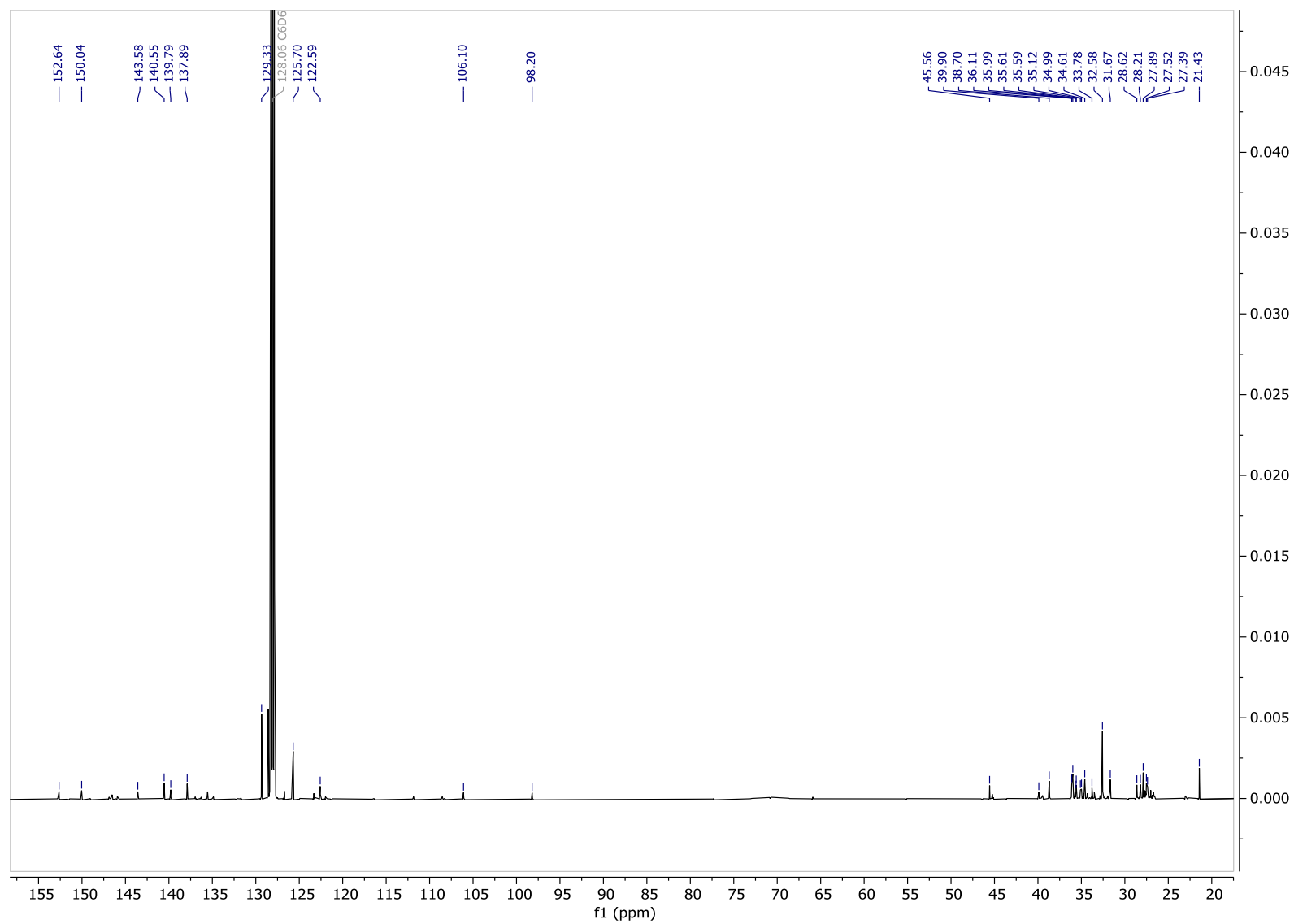
A solution of (xNON^{TCHP})InK(Et₂O)₂(THF)₂ (62.6 mg, 0.050 mmol) in C₆D₆ was added to 18-crown-6 (13.2 mg, 0.050 mmol) under nitrogen at room temperature. The solution was heated at 60°C for 5 minutes before crystallisation began in the *J Youngs* NMR tube, giving light yellow/green crystals suitable for an X-ray diffraction experiment. Yield 47.1 mg, 70.6 %.

¹H NMR (500 MHz, C₆D₆) δ 7.29 (s, 4H, ArH), 6.21 (d, J = 2.4 Hz, 2H, XA-*p*-CH), 6.06 (d, J = 2.4 Hz, 2H, XA-*o*-CH), 3.45 (tt, J = 11.9, 3.3 Hz, 4H, *p*-CyH) 3.20 (s, 24H, (18-c-6(H))), 2.70 (tt, J = 11.9, 3.4 Hz, 2H, *o*-CyH), 2.27 (d, J = 12.0 Hz, 4H, CyH₂), 2.18 (d, J = 12.8 Hz, 4H, CyH₂), 2.06 (d, J = 11.4 Hz, 6H, CyH₂), 1.97 (s, 6H, C(CH₃)₂), 1.85 – 1.69 (m, 21H, CyH₂), 1.55 – 1.43 (m, 4H, CyH₂), 1.39 (s, 18H, C(CH₃)₃).

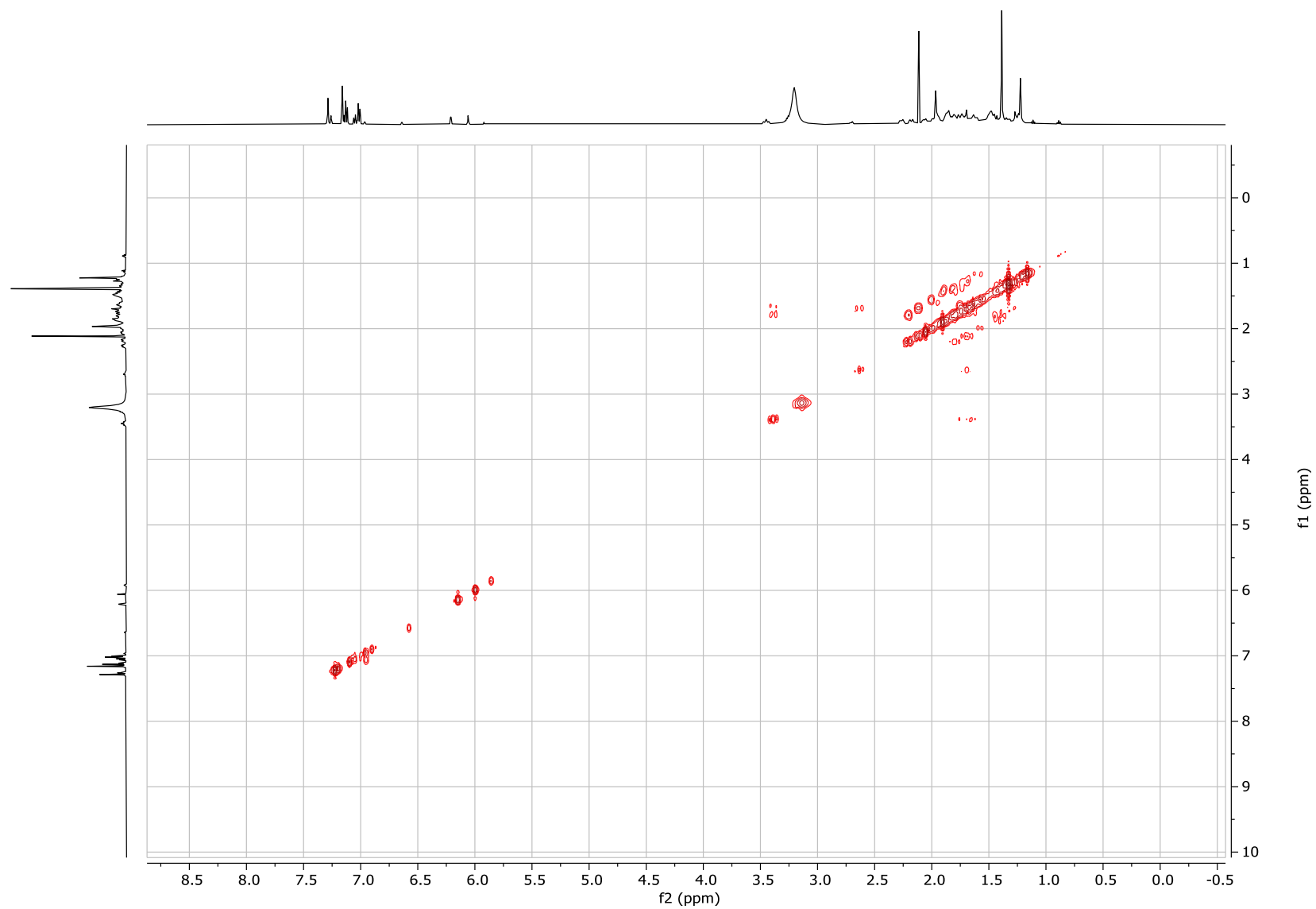
¹³C {¹H} NMR (126 MHz, , C₆D₆) δ 152.64, 150.04, 143.58, 140.55, 139.79, 137.89, 129.33, 125.70 (Ar), 122.59 (ArC), 106.10 (XA-*p*-CH), 98.20 (XA-*o*-CH), 45.56 (*o*-CyC), 39.9 (18-c-6), 38.70 (*p*-CyC), 36.11, 35.99, 35.61, 35.59, 35.12, 34.99, 34.61 (C(CH₃)₂, C(CH₃)₃, CyCH₂), 33.78 C(CH₃)₂, 32.58 (C(CH₃)₃), 31.67, 28.62, 28.21, 27.89, 27.52, 27.39, 21.43 (C(CH₃)₂, C(CH₃)₃, CyCH₂).



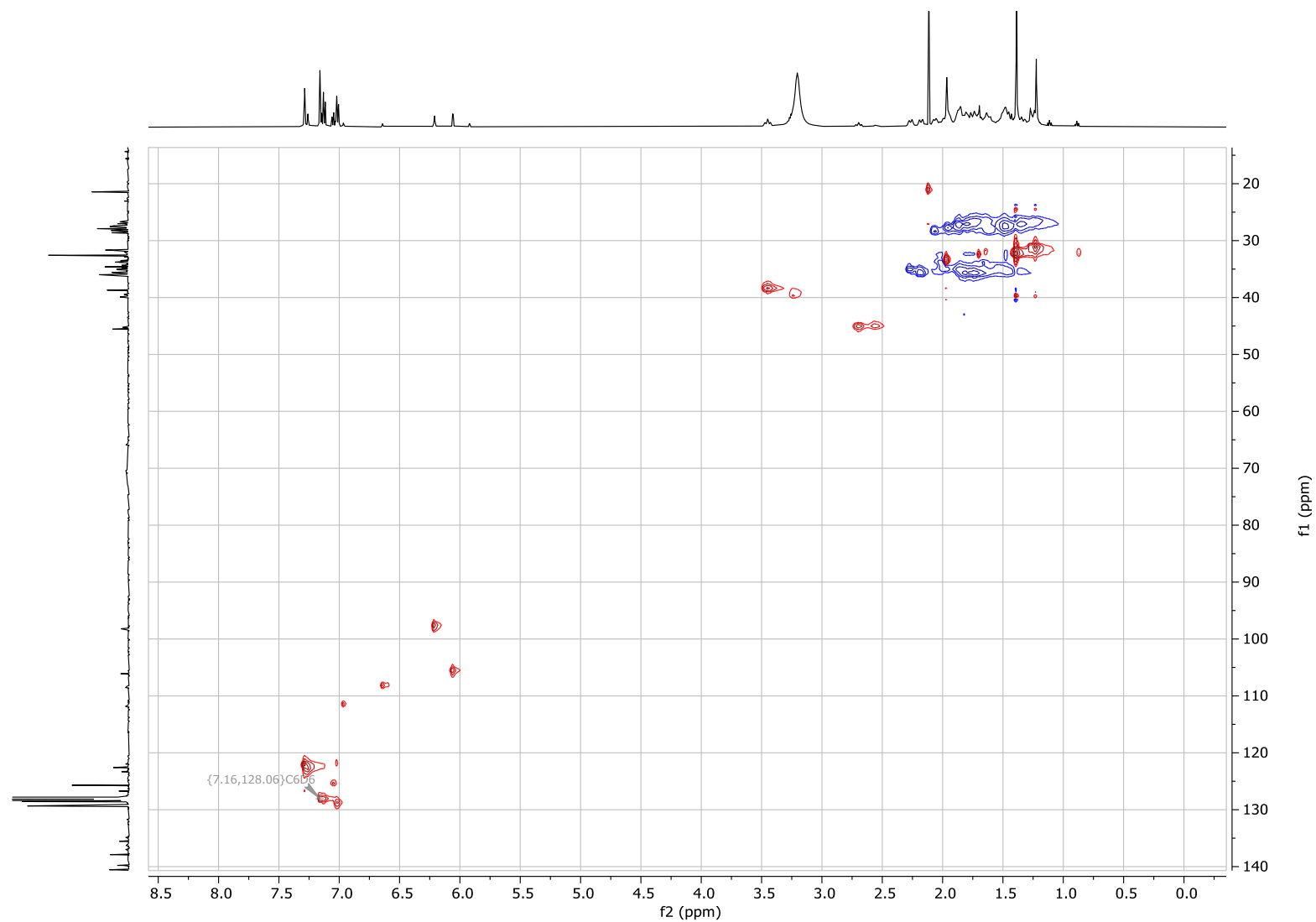
Supplementary figure 31. ¹H NMR spectrum (500 MHz, C₆D₆) of (xNON^{TCHP})InK (18-c-6K), (48).



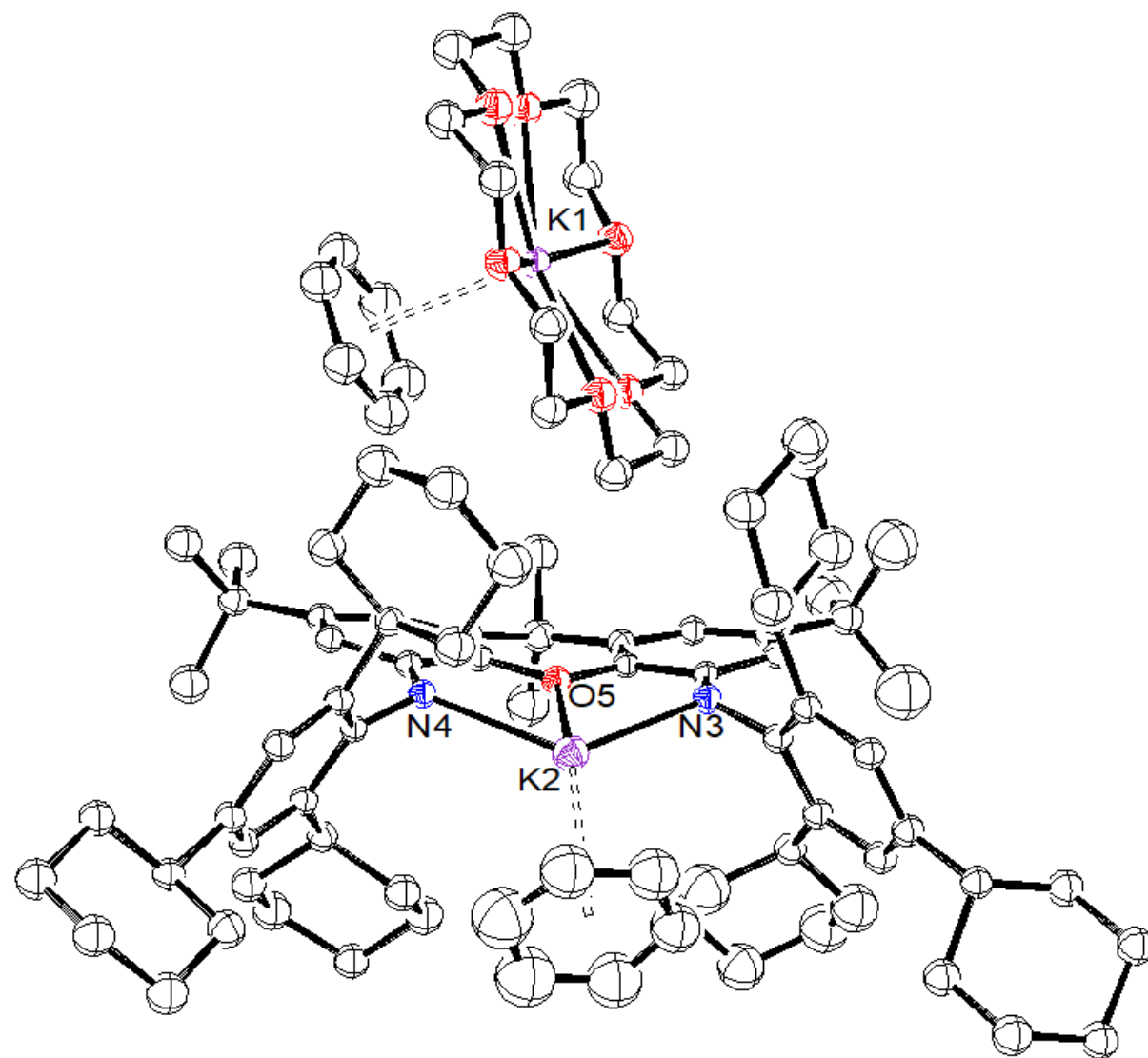
Supplementary figure 32. ¹³C NMR spectrum (126 MHz, C₆D₆) of (xNON^{TCHP})InK (18-c-6K), (**48**).



Supplementary figure 33. ^1H - ^1H COSY NMR spectrum (500 MHz, C_6D_6) of $(\text{xNON}^{\text{TCHP}})\text{InK}$ (18-c-6K), (**48**).



Supplementary figure 34. ^1H - ^{13}C HSQC NMR spectrum (500 MHz, C_6D_6) of $(\text{xNON}^{\text{TCHP}})\text{InK}$ (18-c-6K), (**48**).



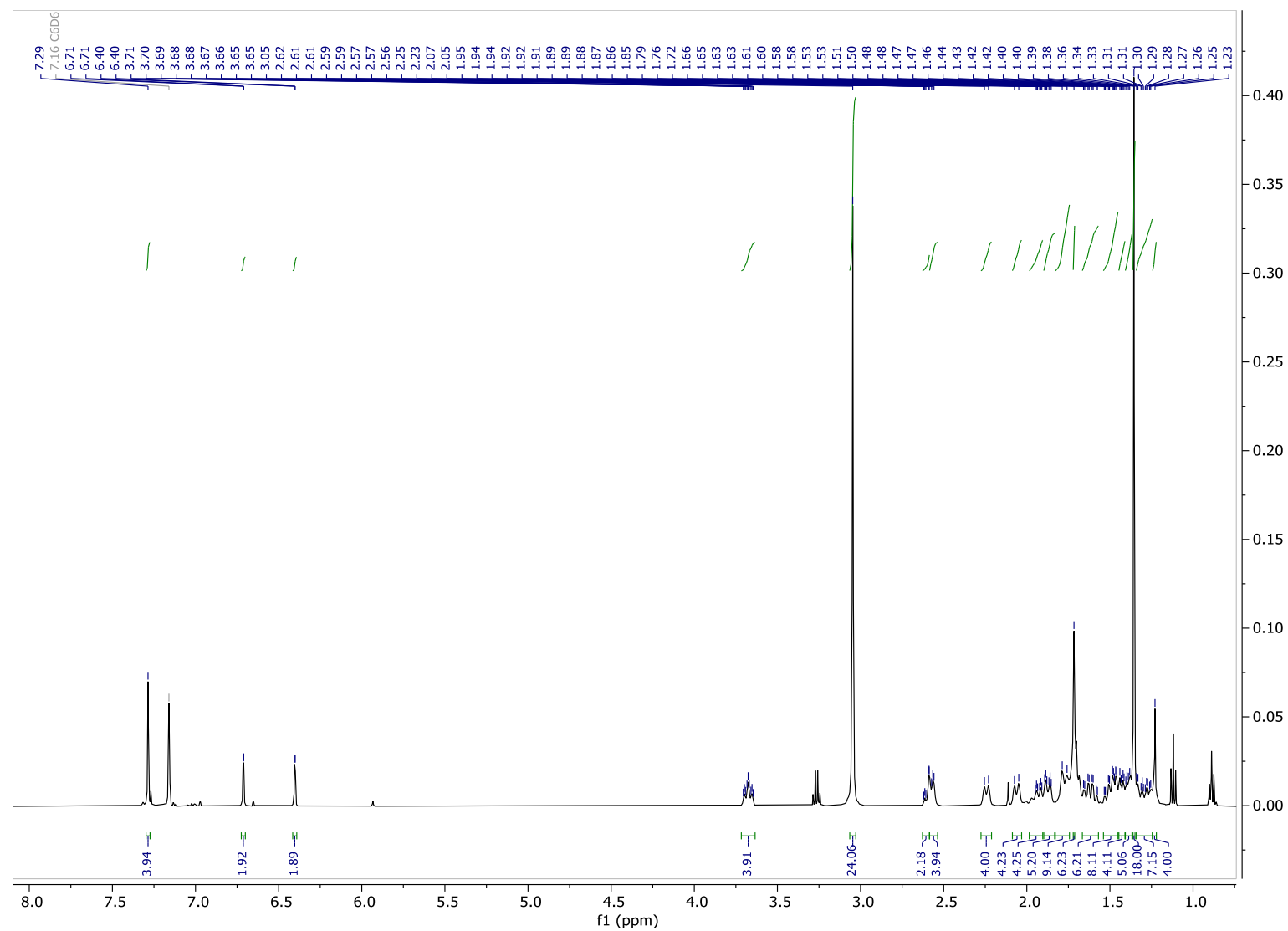
Supplementary figure 35. Ortep representation (ellipsoid 30% probability) of (xNON^{TCHP})InK (18-c-6K), (**48**). Hydrogen atoms have been omitted for clarity.

Preparation of (xNON^{TCHP})InI₂ (18-c-6)K, (49)

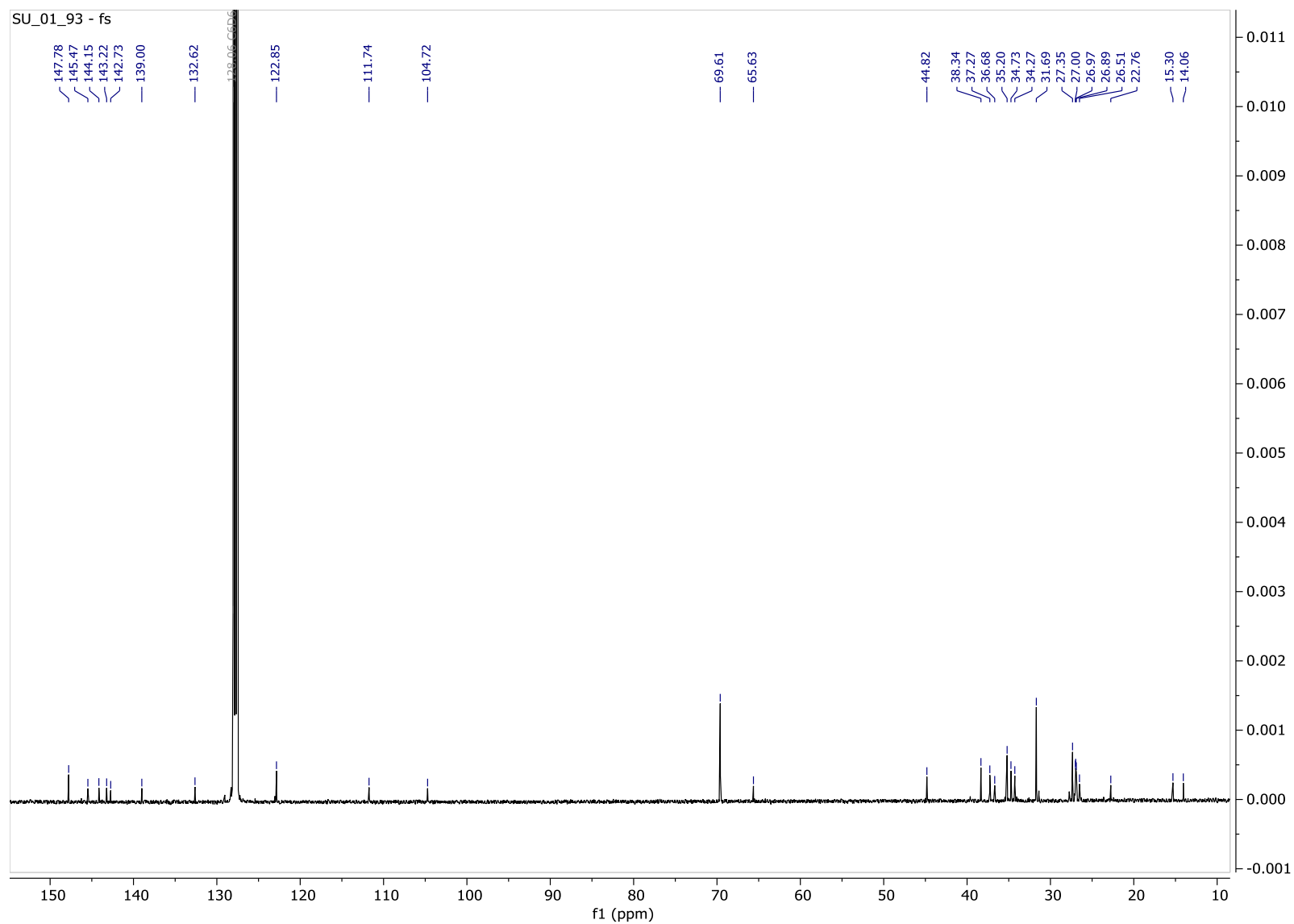
A solution of (xNON^{TCHP})InK(Et₂O)₂(THF)₂ (43.7 mg, 0.031 mmol) in C₆D₆ was added to 18-crown-6 (8.8 mg, 0.033 mmol) under nitrogen at room temperature. The solution was heated at 60°C for 5 minutes before crystallisation began in the *J Youngs* NMR tube, giving light yellow/green crystals suitable for an X-ray diffraction experiment. Yield 13.4 mg, 25.8 %.

¹H NMR (500 MHz, C₆D₆) δ 7.29 (s, 4H, Ar-*H*), 6.71 (d, *J* = 2.1 Hz, 2H, XA-*p*-CH), 6.40 (d, *J* = 2.0 Hz, 2H, XA-*o*-CH), 3.68 (tt, *J* = 11.7, 3.3 Hz, 4H, Cy-*H*), 3.05 (s, 24H, (18-c-6-*H*)), 2.62 – 2.59 (m, 2H, Cy-*H*), 2.57 (d, *J* = 3.5 Hz, 4H, Cy-*H*₂), 2.24 (d, *J* = 12.4 Hz, 4H, Cy-*H*₂), 2.06 (d, *J* = 12.9 Hz, 4H, Cy-*H*₂), 1.93 (dt, *J* = 12.9, 3.4 Hz, 4H, Cy-*H*₂), 1.87 (dt, *J* = 13.0, 3.2 Hz, 5H, Cy-*H*₂), 1.82 – 1.74 (m, 9H, Cy-*H*₂), 1.72 (s, 6H, CH₃), 1.62 (qd, *J* = 13.1, 3.3 Hz, 6H, Cy-*H*₂), 1.50 (ddd, *J* = 21.6, 11.1, 3.1 Hz, 8H, Cy-*H*₂), 1.45 – 1.41 (m, 4H, Cy-*H*₂), 1.41 – 1.37 (m, 5H, Cy-*H*₂), 1.36 (s, 18H, CH(CH₃)₃), 1.34 – 1.25 (m, 7H, Cy-*H*₂), 1.23 (s, 4H, Cy-*H*₂).

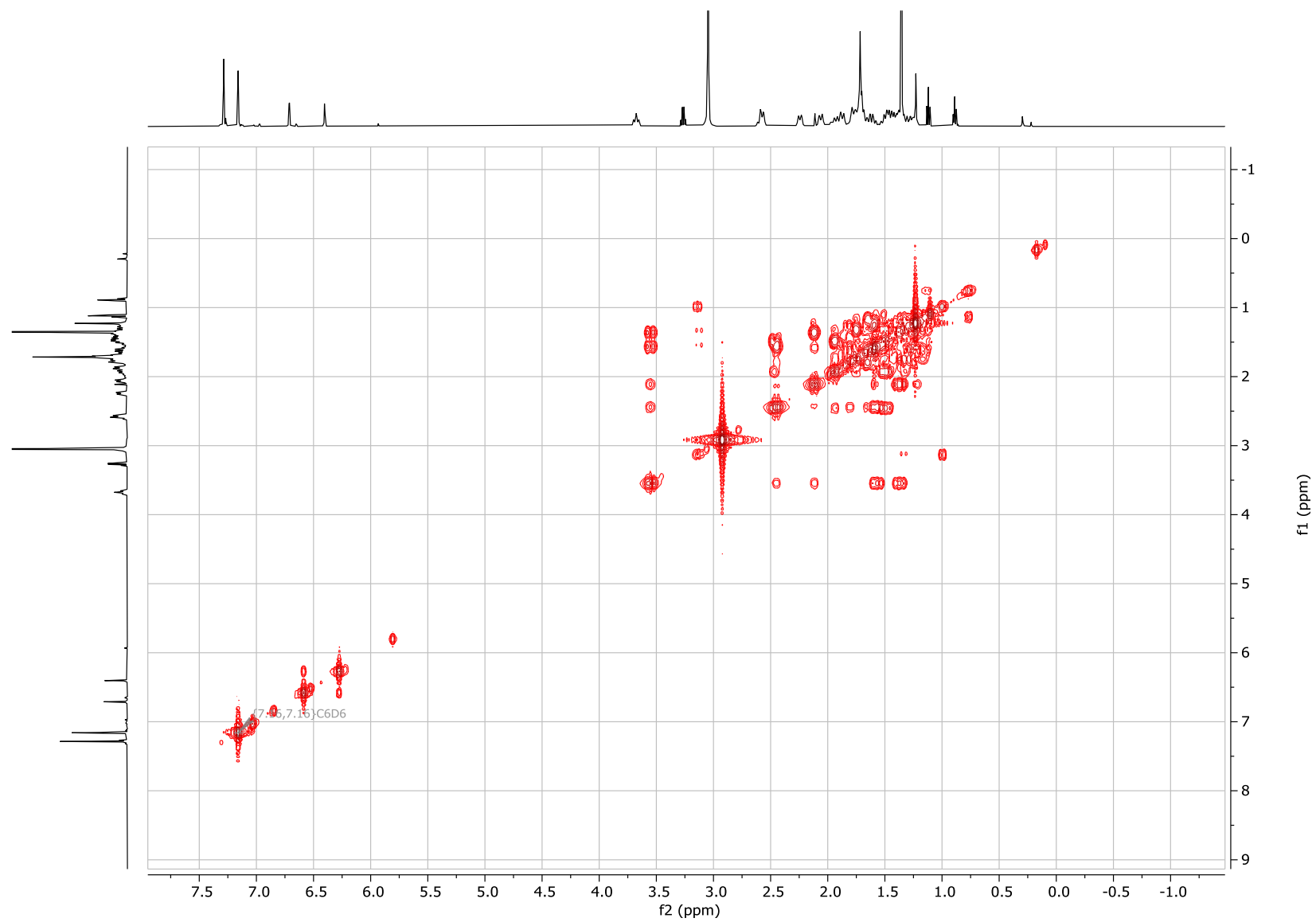
¹³C{¹H} NMR (126 MHz, C₆D₆) δ 147.8, 145.5, 144.2, 143.2, 142.7, 139.0, 132.6 (*Ar*), 122.9 (*ArC*), 111.7 (XA-*p*-CH), 104.7 (XA-*o*-CH), 69.6 (18-*c*-6), 65.6 (*Cy*), 44.8(*o*-*CyH*), 38.3 (*o*-*CyH*), 37.3, 36.7, 35.2, 34.7, 34.3 (*C*(CH₃)₂, *C*(CH₃)₃, *CyCH*₂), 31.7 (*C*(CH₃)₃), 27.4 (*C*(CH₃)₂, 27.0, 26.9, 26.9, 26.5, 22.8, 15.3, 14.1 (*C*(CH₃)₂, *C*(CH₃)₃, *CyCH*₂).



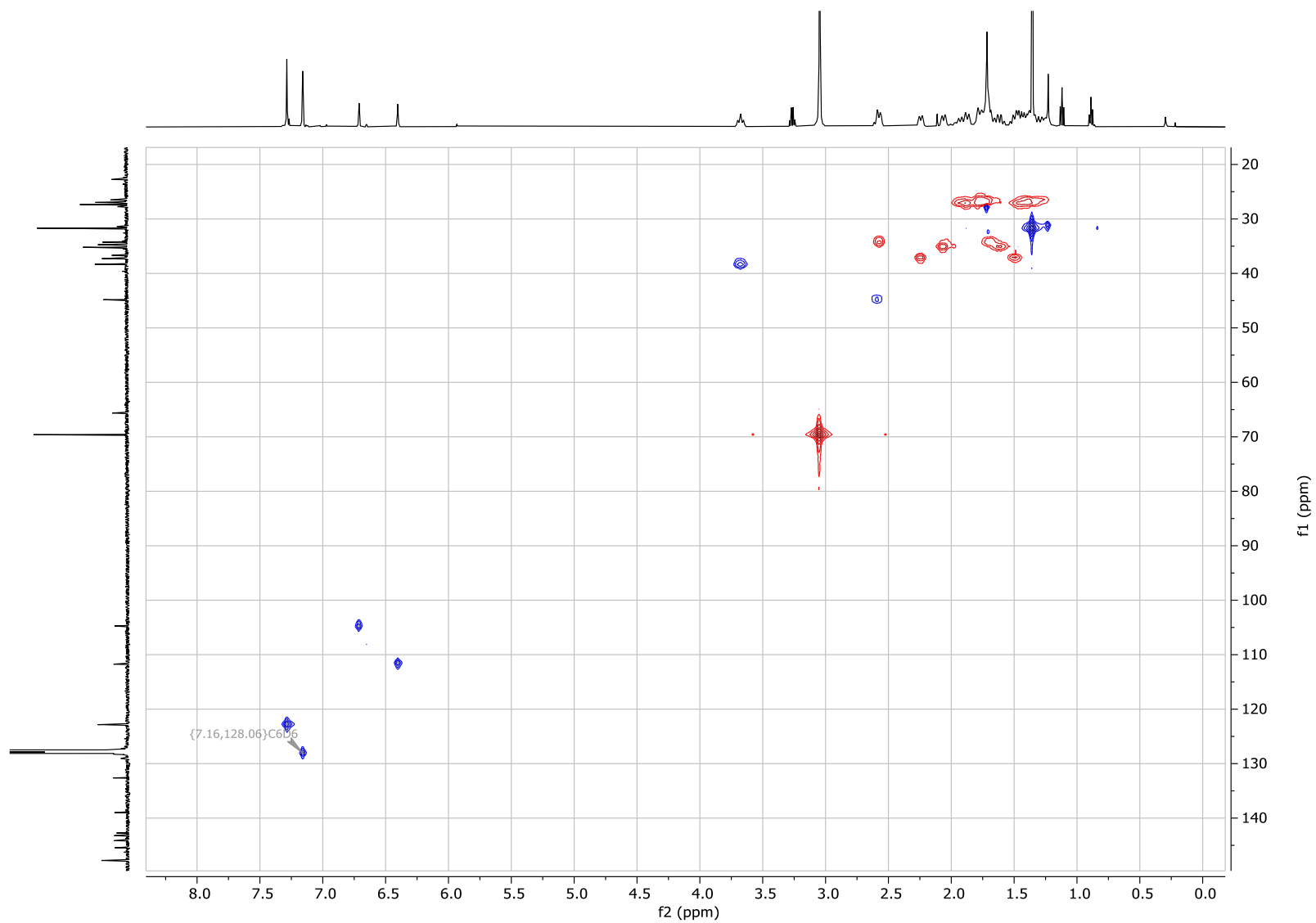
Supplementary figure 36. ¹H NMR spectrum (500 MHz, C₆D₆) of (xNON^{TCHP})InI₂ (18-c-6)K, (**49**).



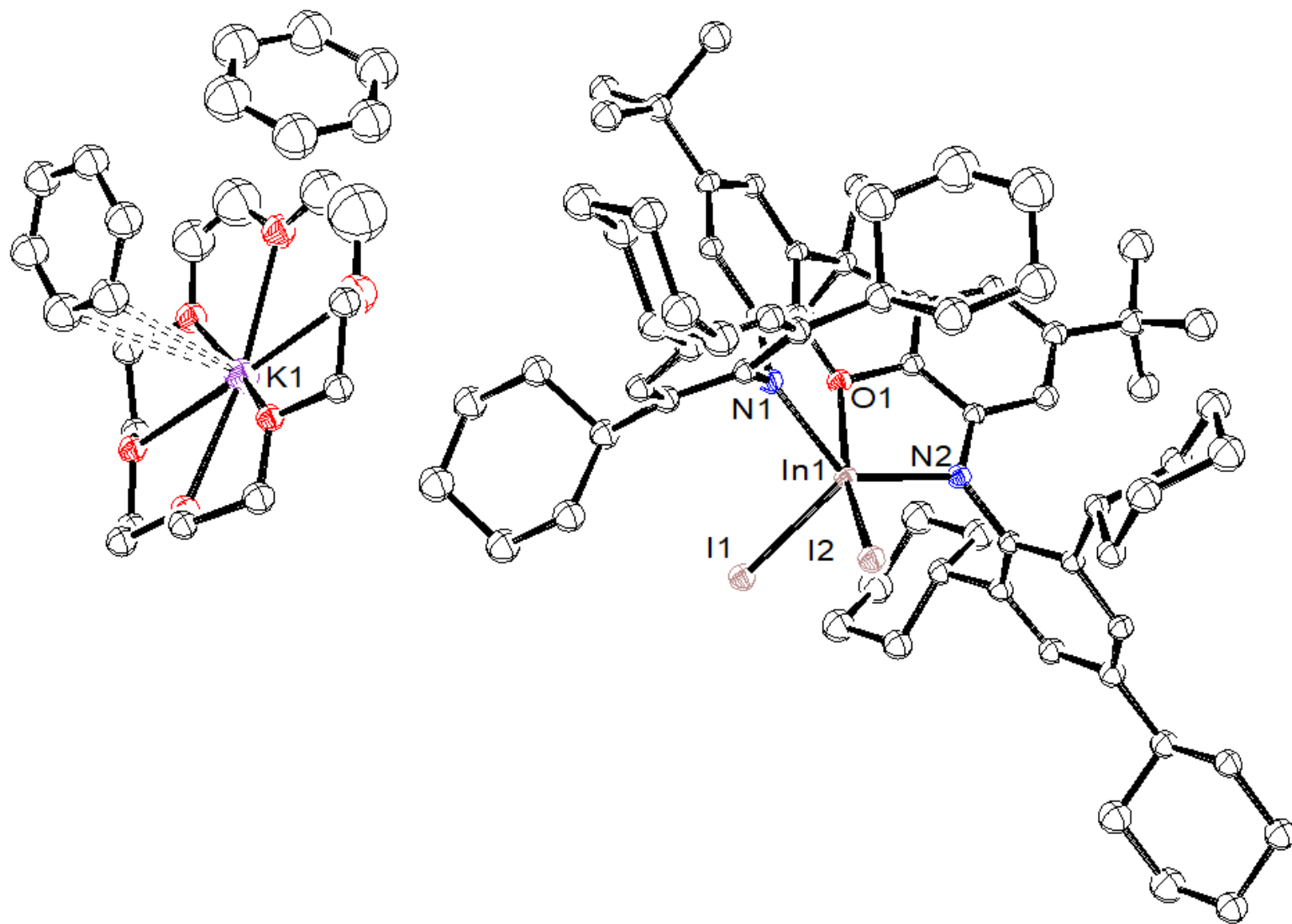
Supplementary figure 37. ^{13}C NMR spectrum (126 MHz, C_6D_6) of $(\text{xNON}^{\text{TCHP}})\text{InI}_2$ (18-c-6)K, (**49**).



Supplementary figure 38. ^1H - ^1H COSY NMR spectrum (500 MHz, C_6D_6) of $(\text{xNON}^{\text{TCHP}})\text{InI}_2$ (18-c-6)K, (**49**).



Supplementary figure 39. ^1H - ^{13}C HSQC NMR spectrum (500 MHz, C_6D_6) of $(\text{xNON}^{\text{TCHP}})\text{InI}_2$ (18-c-6)K, (**49**).



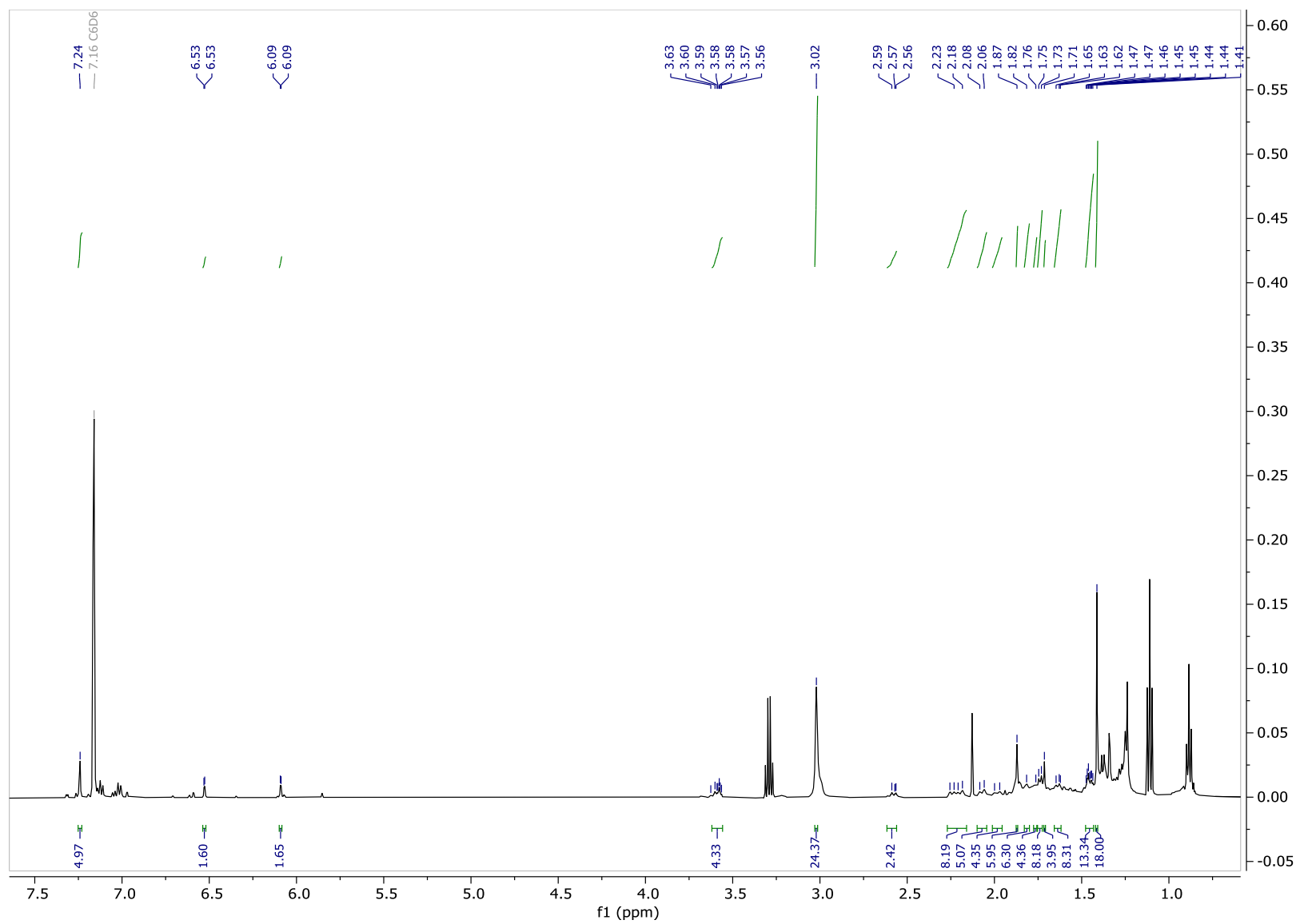
Supplementary figure 40. Ortep representation (ellipsoid 30% probability) of $(x\text{NON}^{\text{TCHP}})\text{InI}_2 (18\text{-c-6})\text{K}$, (49). Hydrogen atoms have been omitted for clarity.

Preparation of (xNON^{TCHP})In (18-c-6)K, (50)

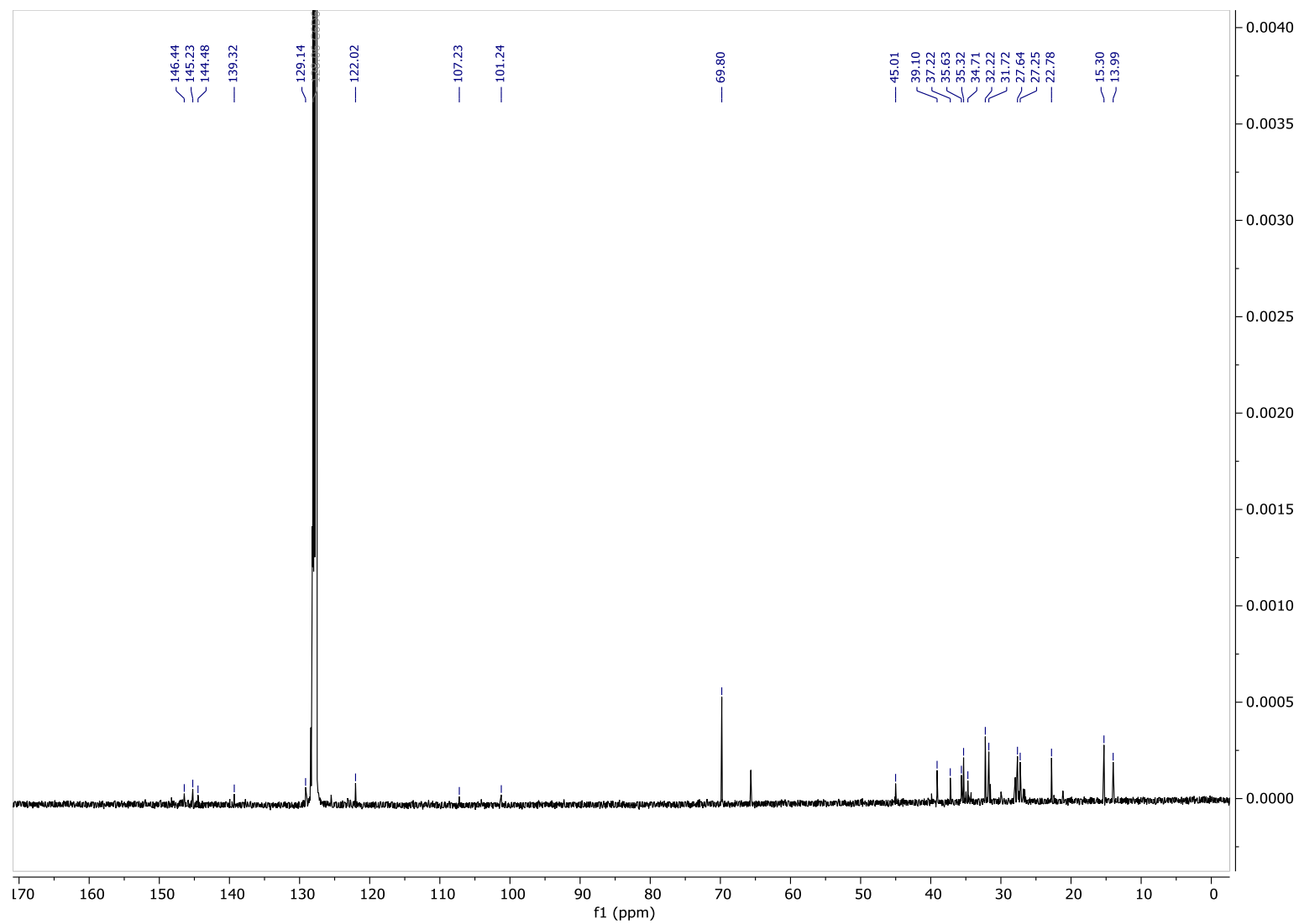
A solution of [(xNON^{TCHP})InI₂][(18-c-6)K] (13.4 mg, 0.008 mmol) in C₆D₆ was added to excess potassium metal and stirred for 48 hours under nitrogen at room temperature. The solution was filtered through celite, and washed with hexane, then reduced and left to crystallise at room temperature, giving bright yellow crystals suitable for an X-ray diffraction experiment. Yield 11.2 mg, 98.2 %.

¹H NMR (500 MHz, C₆D₆) δ 7.24 (s, 4H, ArH), 6.53 (d, J = 2.2 Hz, 2H, XA-*p*-CH), 6.09 (d, J = 2.2 Hz, 2H, XA-*o*-CH), 3.64 – 3.55 (m, 4H, *o*-CyH), 3.02 (s, 24H, (18-c-6)H), 2.62 – 2.56 (m, 2H, *p*-CyH), 2.22 (dd, J = 23.7, 12.2 Hz, 8H, CyH₂), 2.07 (d, J = 12.6 Hz, 5H, CyH₂), 1.98 (d, J = 14.8 Hz, 4H, CyH₂), 1.87 (s, 6H, C(CH₃)₂), 1.82 (s, 6H, CyH₂), 1.76 (s, 4H, CyH₂), 1.74 (d, J = 7.6 Hz, 8H, CyH₂), 1.71 (s, 4H, CyH₂), 1.66 – 1.62 (m, 8H, CyH₂), 1.45 (dt, J = 9.1, 3.5 Hz, 13H, CyH₂), 1.41 (s, 4H, C(CH₃)₃).

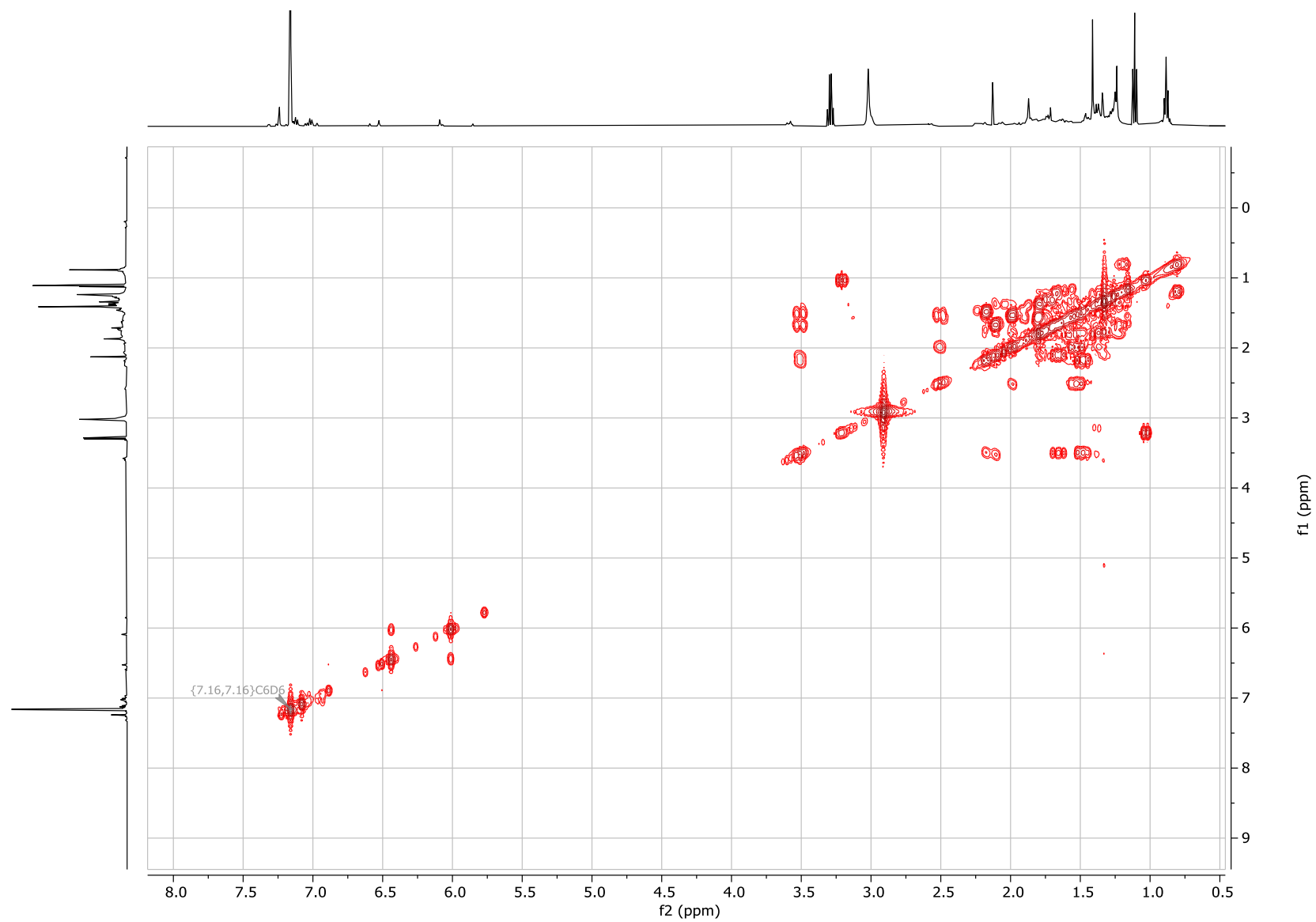
¹³C {¹H} NMR (126 MHz, C₆D₆) δ 146.44, 145.23, 144.48, 139.32, 129.14 (ArC), 122.02 (TCHP-ArC), 107.23 (XA-*o*-CH), 101.24 (XA-*p*-CH), 69.80 ((18-c-6)C), 45.01 (*p*-CyC), 39.10 (*o*-CyC), 37.22, 35.63 (C(CH₃)₂), 35.32, 34.71 CyCH₂, 32.22 (C(CH₃)₃), 31.72, 27.64, 27.25, 22.78, 15.30, 13.99 (C(CH₃)₂, C(CH₃)₃, CyCH₂).



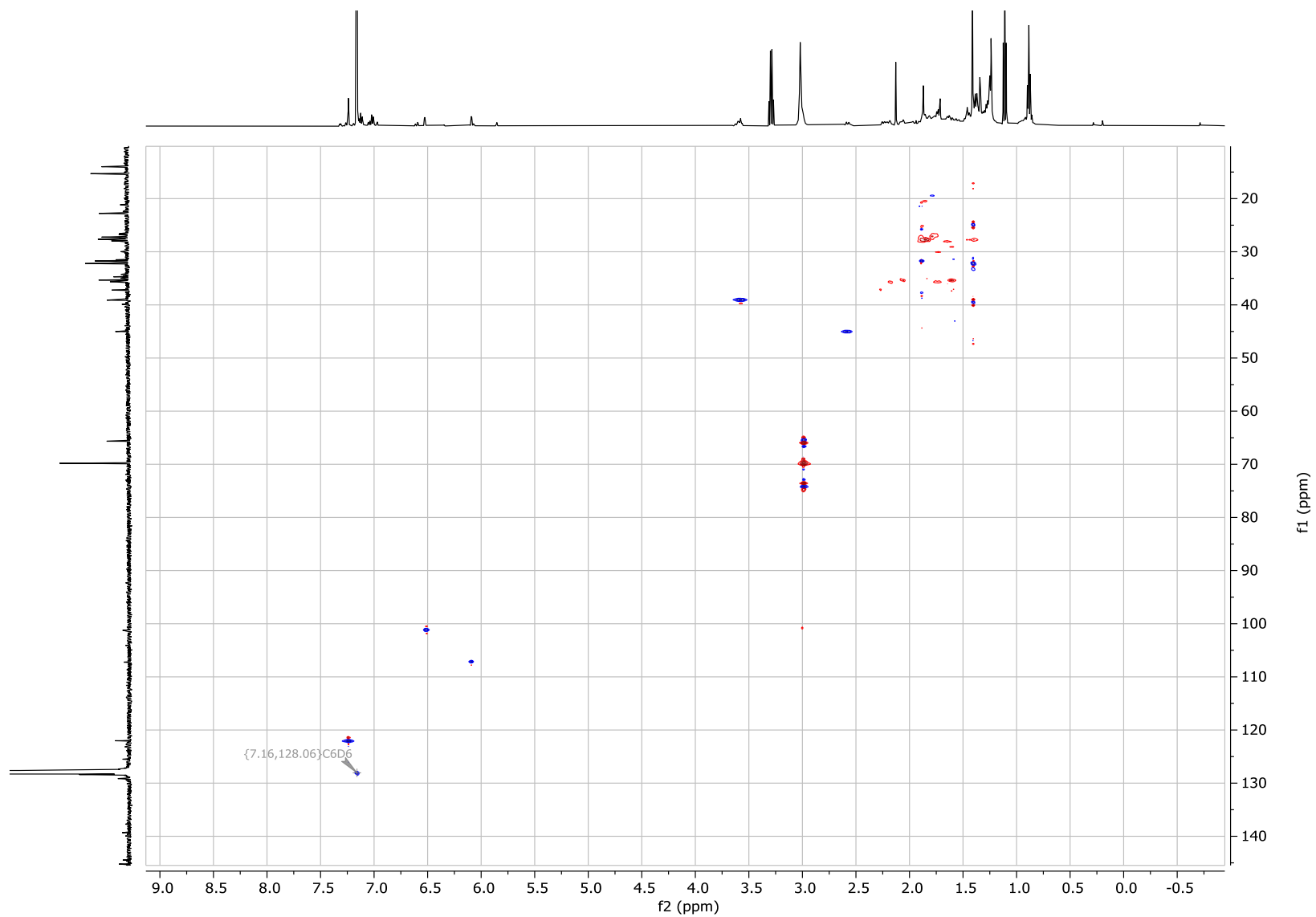
Supplementary figure 41. ^1H NMR spectrum (500 MHz, C_6D_6) of $(\text{xNON}^{\text{TCHP}})\text{In}$ (18-crown-6)K, (**50**).



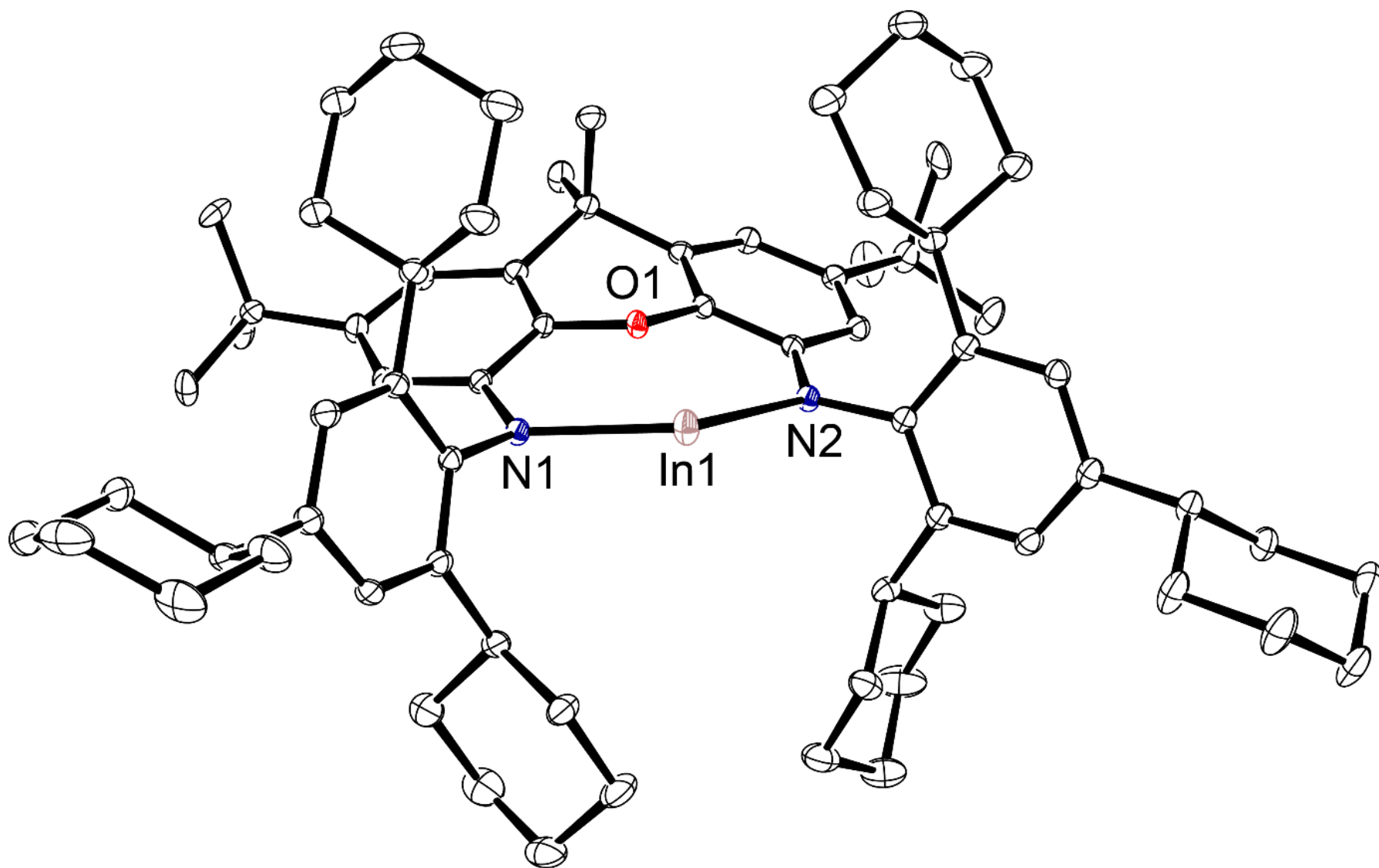
Supplementary figure 42. ¹³C NMR spectrum (126 MHz, C₆D₆) of (xNON^{TCHP})In (18-crown-6)K, (**50**).



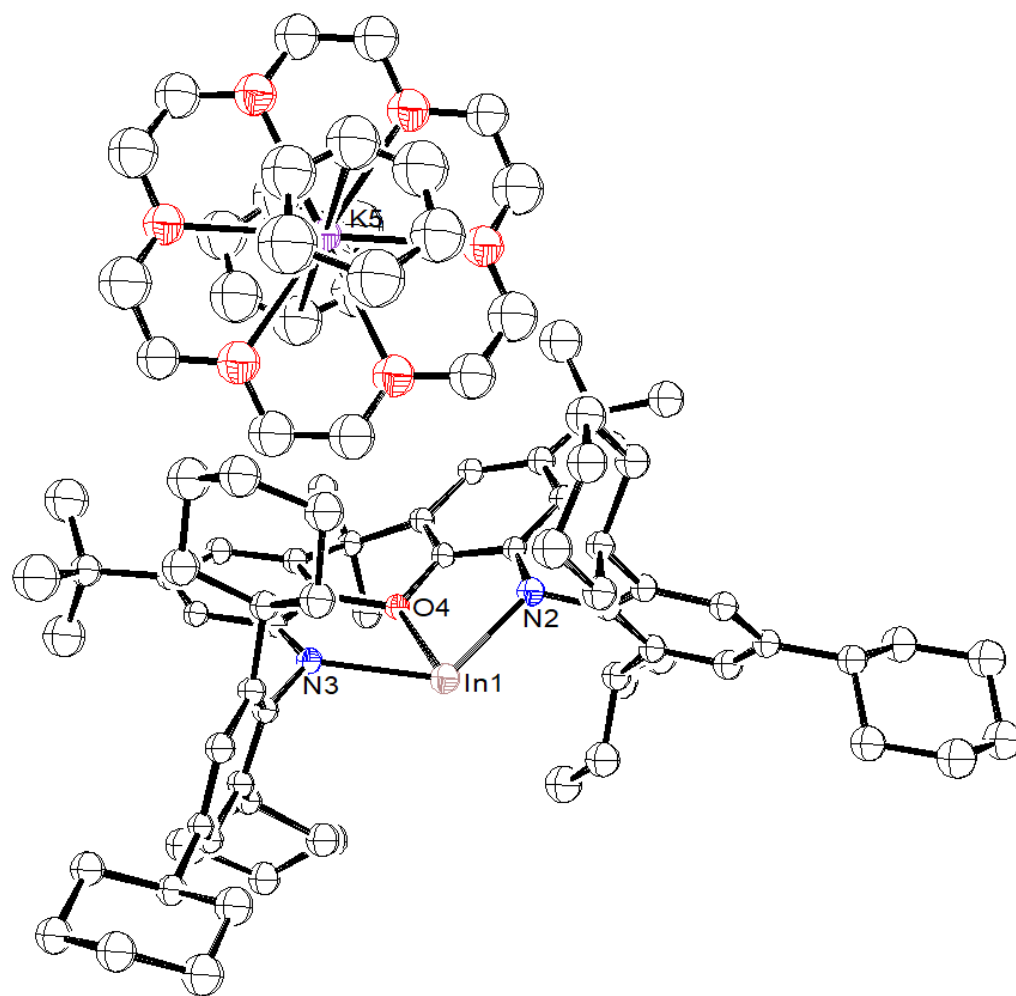
Supplementary figure 43. ^1H - ^1H COSY NMR spectrum (500 MHz, C_6D_6) of $(\text{xNON}^{\text{TCHP}})\text{In}$ (18-crown-6)K, (**50**).



Supplementary figure 44. ^1H - ^{13}C HSQC NMR spectrum (500 MHz, C_6D_6) of $(\text{xNON}^{\text{TCHP}})\text{In}$ (18-crown-6)K, (**50**).



Supplementary figure 45. Ortep representation (ellipsoid 30% probability) of (xNON^{TCHP})In, (**50**). Hydrogen atoms and [(18-crown-6)K] have been omitted for clarity.

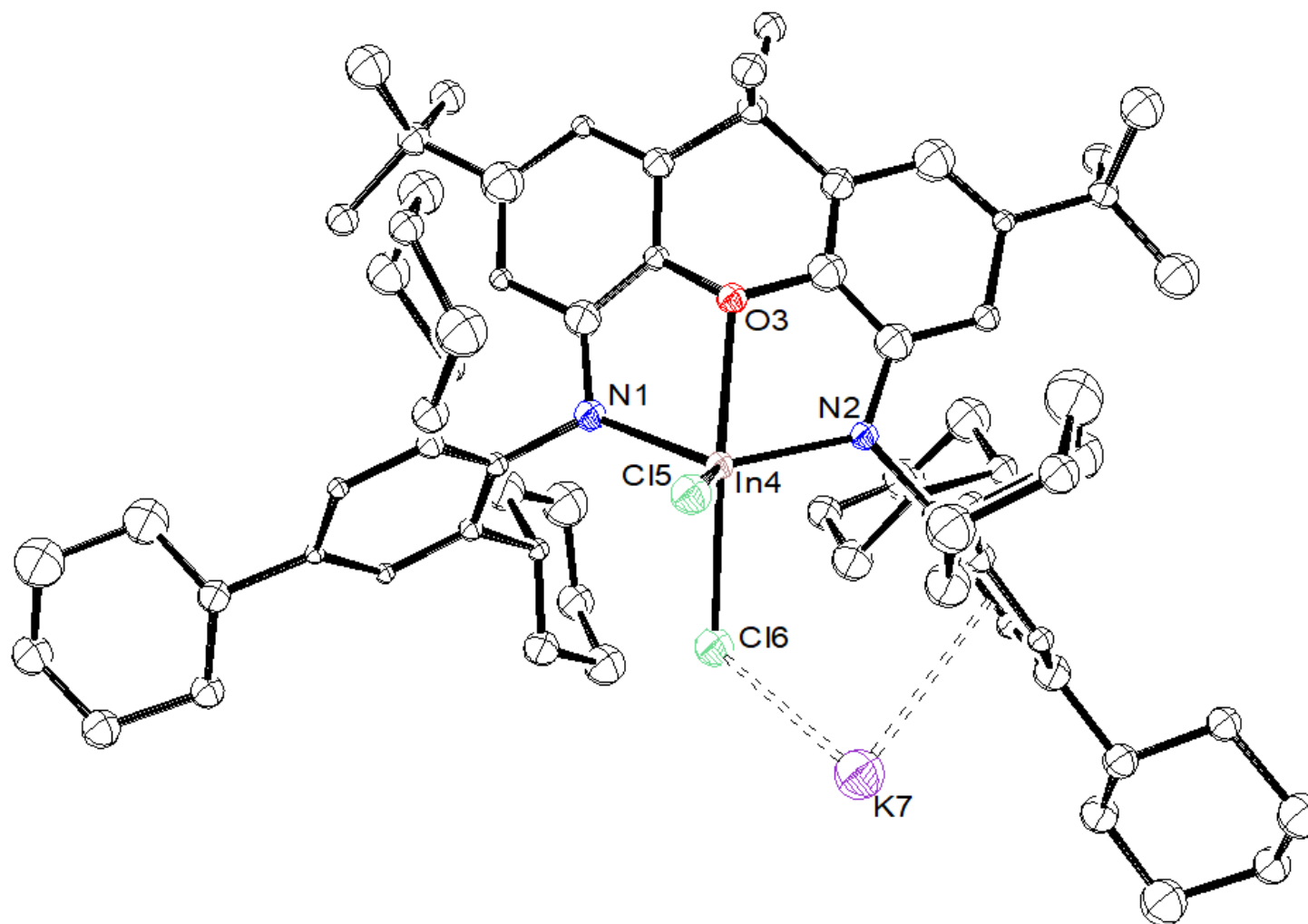


Supplementary figure 46. Ortep representation (ellipsoid 30% probability) of $[(x\text{NON}^{\text{TCHP}})\text{In}] [(18\text{-crown-6})\text{K}]$, (50). Hydrogen atoms have been omitted for clarity.

Preparation of $[(x\text{NON}^{\text{TCHP}})\text{InCl}_2\text{K}]_2$, (52)

A solution of $(x\text{NON}^{\text{TCHP}})\text{InK}(\text{Et}_2\text{O})_2(\text{THF})_2$ (114.4 mg, 0.087 mmol) in diethyl ether was added to SnCl_2 (16.6 mg, 0.087 mmol) and stirred for 24 hours under nitrogen at room temperature. The solution was filtered through celite, and reduced and left to crystallise at room temperature, giving pale orange crystals suitable for an X-ray diffraction experiment.

Due to the extreme insolubility in both coordinating and non-coordinating solvents at either room temperature or elevated temperatures we were unable to record solution state data for this compound.

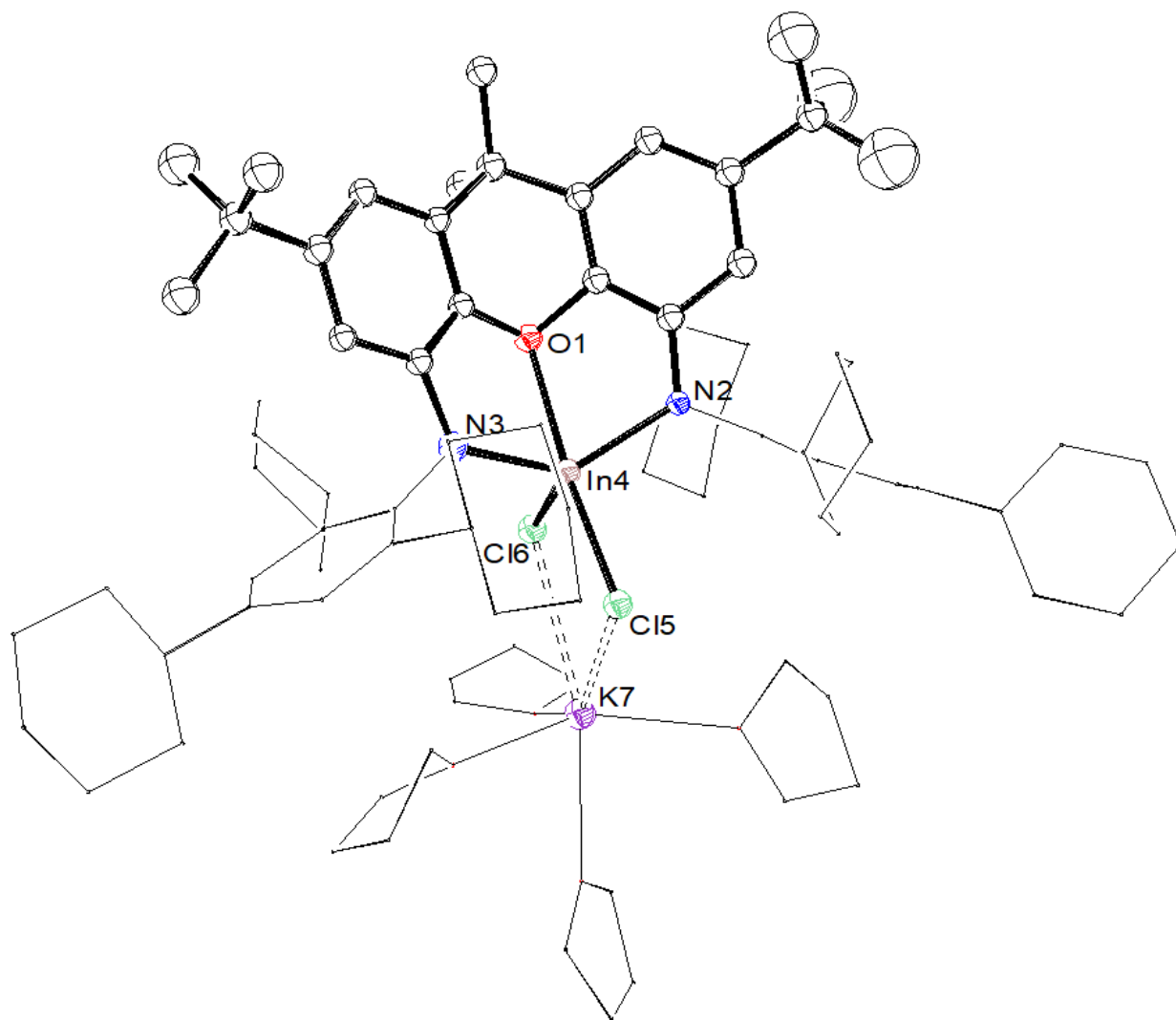


Supplementary figure 47. Ortep representation (ellipsoid 30% probability) of $[(x\text{NON}^{\text{TCHP}})\text{InCl}_2\text{K}]_2$, (**52**). Hydrogen atoms have been omitted for clarity.

Preparation of $(x\text{NON}^{\text{TCHP}})\text{InCl}_2\text{K}(\text{THF})_4$, (53)

Dried crystals of $[(x\text{NON}^{\text{TCHP}})\text{InCl}_2\text{K}]_2$ was extracted into a 1:1 ratio of THF and toluene. The solution reduced and left to crystallise at room temperature, giving colourless crystals suitable for an X-ray diffraction experiment.

Due to the extreme insolubility in both coordinating and non-coordinating solvents at either room temperature or elevated temperatures we were unable to record solution state data for this compound.



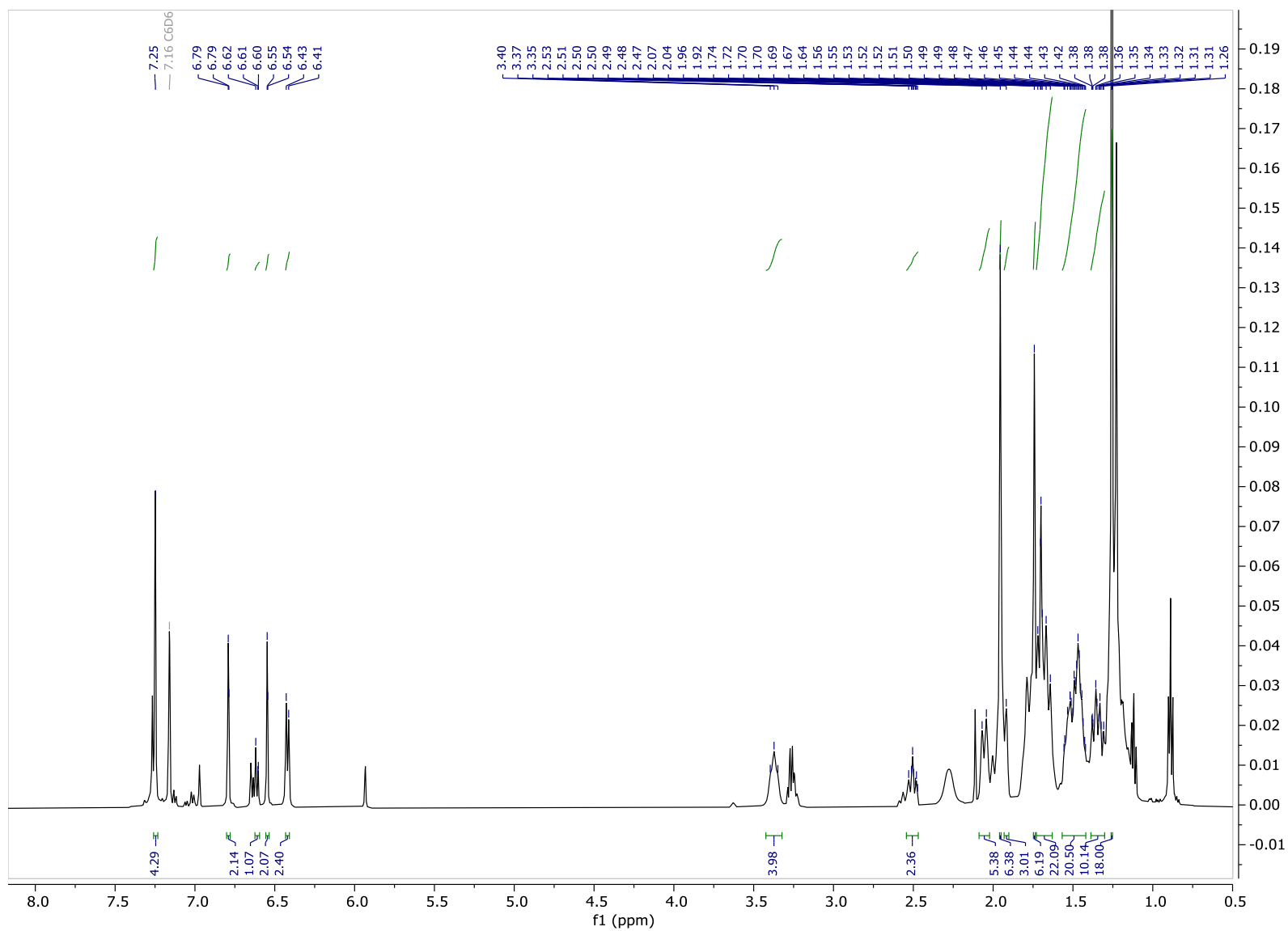
Supplementary figure 48. Ortep representation (ellipsoid 30% probability) of $(x\text{NON}^{\text{TCHP}})\text{InCl}_2\text{K}(\text{THF})_4$, (**53**). Hydrogen atoms have been omitted for clarity.

Preparation of (xNON^{TCHP})InI(DMP-NC), (55)

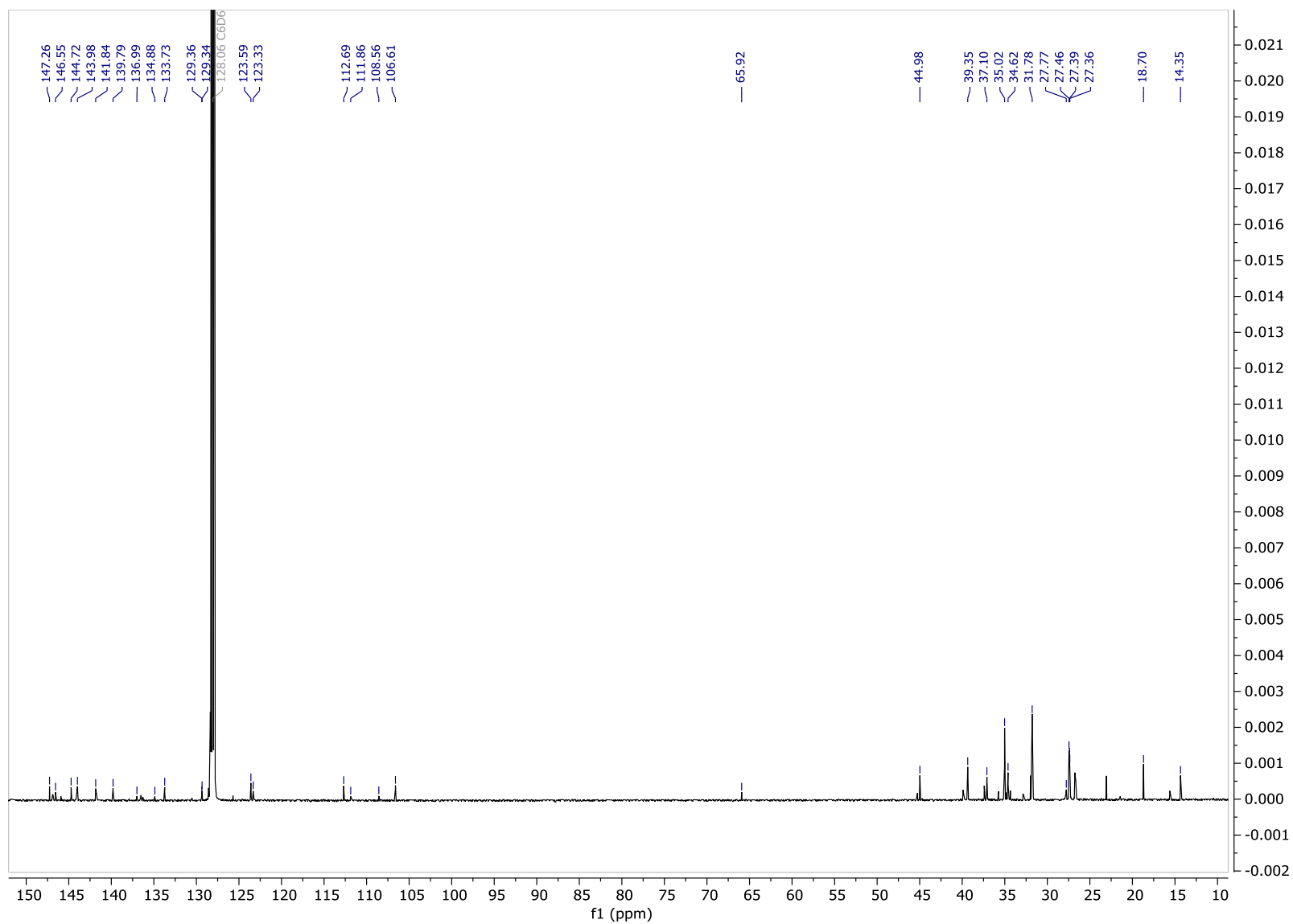
A solution of (xNON^{TCHP})InK(Et₂O)₂(THF)₂ (57.0 mg, 0.04 mmol) in C₆D₆ was added to 2,6-dimethyl phenyl isocyanide (5.8 mg, 0.04 mmol). The solution was filtered through celite, and washed with benzene, then dried and crystallised at room temperature in diethyl ether, giving light yellow crystals suitable for an X-ray diffraction experiment. Yield 25.4 mg, 40.9 %.

¹H NMR (500 MHz, C₆D₆) δ 7.25 (s, 4H, TCHP-ArH), 6.79 (d, J = 2.0 Hz, 2H, XA-*p*-CH), 6.61 (d, J = 8.2 Hz, 1H, Phen-*p*-CH), 6.55 (d, J = 2.0 Hz, 2H, XA-*o*-CH), 6.42 (d, J = 7.7 Hz, 2H, Phen-*m*-CH), 3.40 – 3.35 (m, 4H, *o*-CyH), 2.55 – 2.47 (m, 2H, *p*-CyH), 2.06 (d, J = 13.5 Hz, 5H, CyH₂), 1.96 (s, 6H, Phen-CH₃), 1.92 (s, 1H, CyH₂), 1.74 (s, 6H, C(CH₃)₂), 1.73 – 1.63 (m, 22H, CyH₂), 1.57 – 1.42 (m, 20H, CyH₂), 1.39 – 1.30 (m, 10H, CyH₂), 1.26 (s, 18H, C(CH₃)₃).

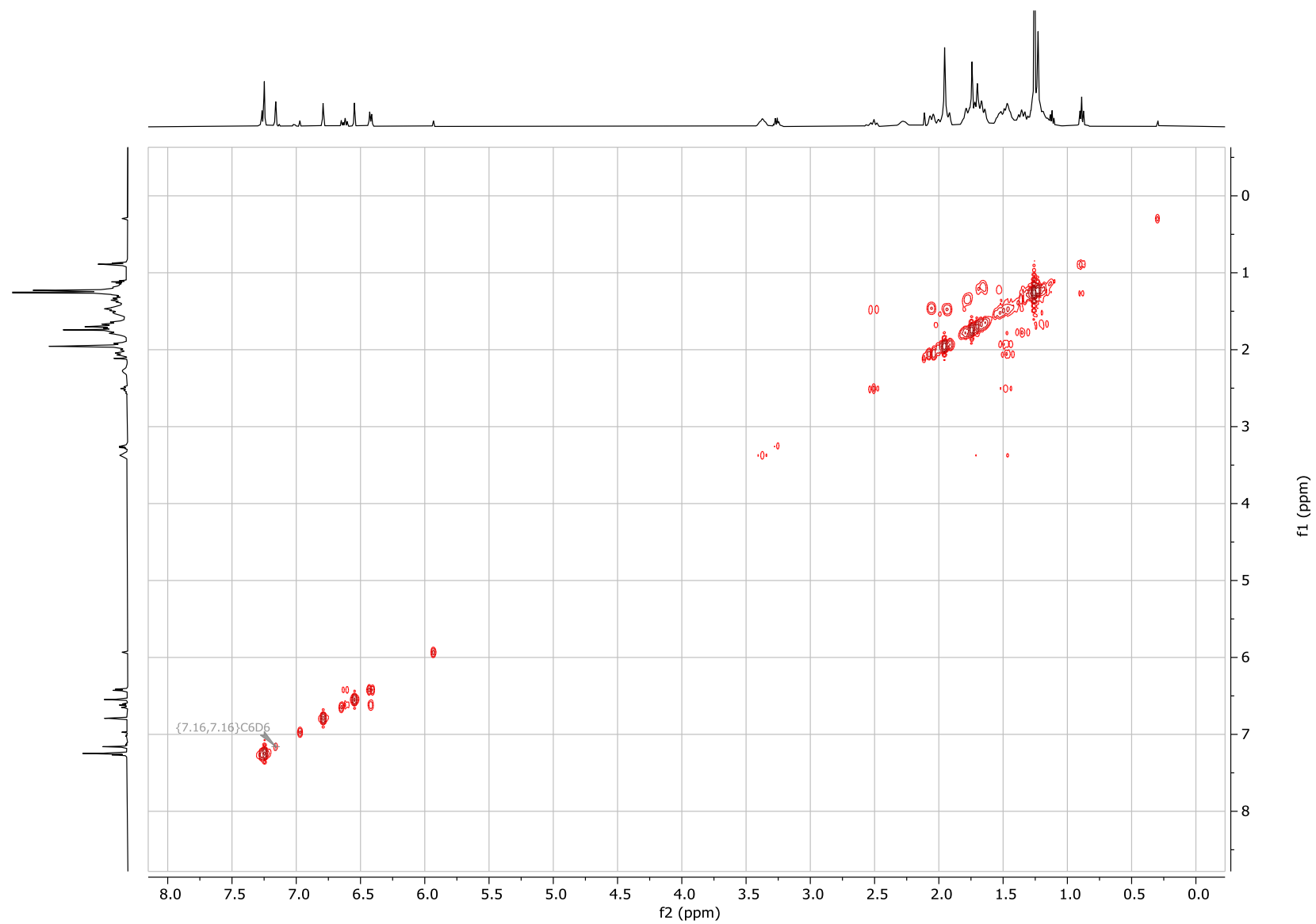
¹³C {¹H} NMR (126 MHz, C₆D₆) δ 147.26, 146.55, 144.72, 143.98, 141.84, 139.79, 136.99, 134.88, 133.73 (ArC), 130.53 (Phen-*p*-CH), 129.36 (ArC), 129.34 (Phen-*m*-CH), 123.59 (TCHP-ArH), 123.33 (ArC), 112.69 (XA-*o*-CH), 111.86, 108.56 (ArC), 106.61 (XA-*p*-CH), 65.92 (C≡N), 44.98 (*p*-CyH), 39.35 (*o*-CyH), 37.10, 35.02, 34.62 (C(CH₃)₃, CyCH₂), 31.78 C(CH₃)₃, 27.77 C(CH₃)₂, 27.46, 27.39, 27.36, (C(CH₃)₃, CyCH₂), 18.70 (CH₃), 14.35 (C(CH₃)₂).



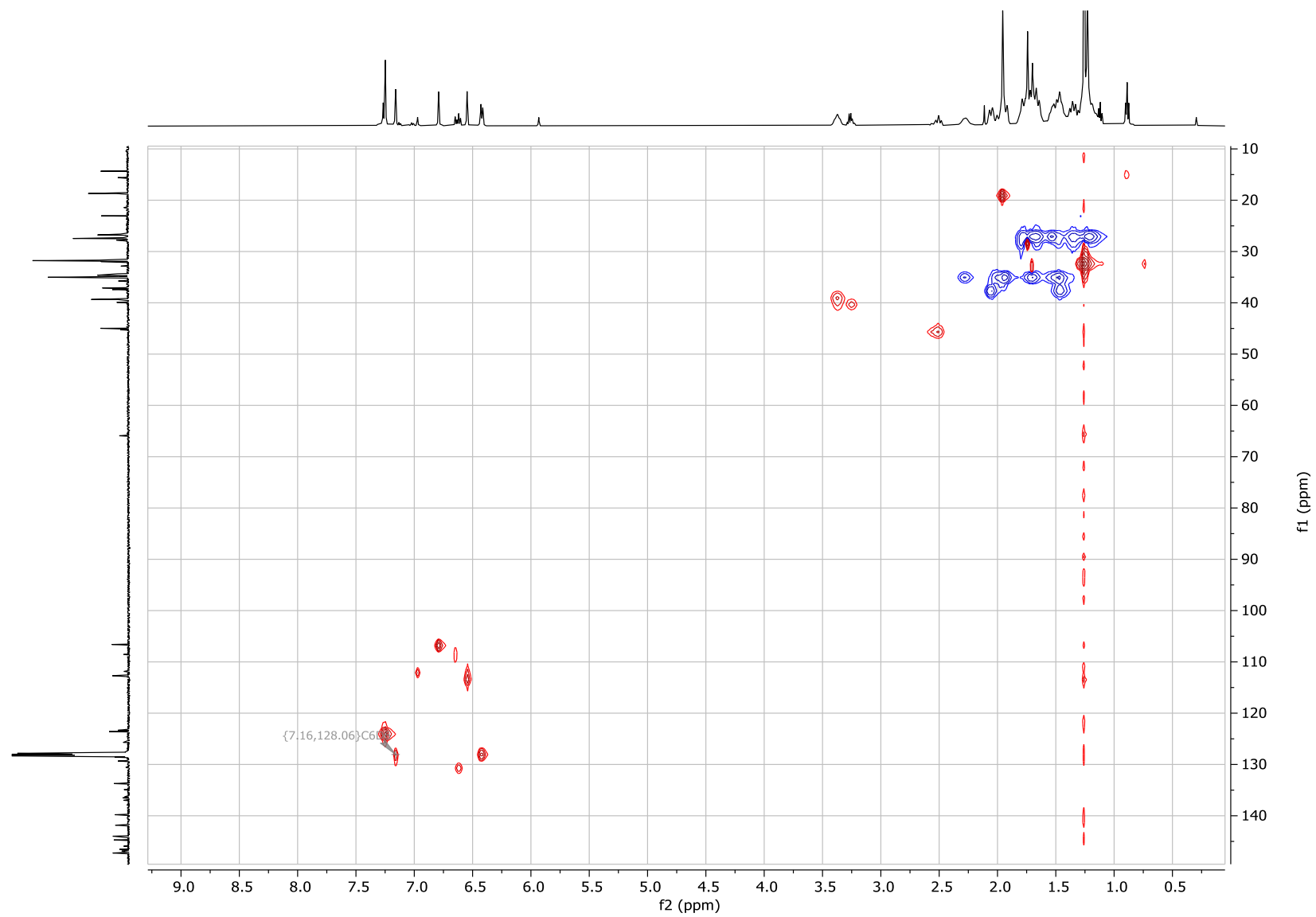
Supplementary figure 49. ^1H NMR spectrum (500 MHz, C_6D_6) of $(\text{xNON}^{\text{TCHP}})\text{InI}(\text{DMP-NC})$, (**55**).



Supplementary figure 50. ¹³C NMR spectrum (126 MHz, C₆D₆) of (xNON^{TCHP})InI(DMP-NC), (**55**).



Supplementary figure 51. ^1H - ^1H COSY NMR spectrum (500 MHz, C_6D_6) of $(\text{xNON}^{\text{TCHP}})\text{InI}(\text{DMP-NC})$, (**55**).



Supplementary figure 52. ^1H - ^{13}C HSQC NMR spectrum (500 MHz, C_6D_6) of $(\text{xNON}^{\text{TCHP}})\text{InI}(\text{DMP-NC})$, (**55**).

