Supporting Information

Functionalized Phosphonate Ester Supported Lanthanide (Ln = La, Nd, Dy, Er) Complexes.

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Contents

1 EXPERIMENTAL

All manipulations involving air- and moisture sensitive compounds were carried out under an argon atmosphere using Schlenk techniques or handled in an argon glovebox. Solvents were dried over Na or K metal or Na/K alloy and freshly distilled. Starting materials were purchased commercially and were used as received, unless stated otherwise. Filtering of moisture sensitive compounds was carried out with self-made filter-cannulas assembled from Whatman fiberglass filters (GF/B, 25 mm), which were applied with Teflon© tape to Teflon© cannulas. Flash chromatography was performed with an Interchim PuriFlash XS 520Plus device using PF-30SIHP-F0020 or -F0040 columns. CV = column volumes. For TLC analyses, pre-coated Macherey–Nagel Alugram Xtra SIL G/UV_{254} plates were used and the compounds were visualized by UV light. NMR experiments were performed with Varian 400 or 500 MHz spectrometers. ^{1}H and ^{13}C-NMR spectra are referenced relative to TMS using the residual solvent signals as internal standards.[1] IR spectra were recorded with a diamond probe Bruker ATR IR spectrometer. Excitation and emission spectra as well as luminescent quantum yield (absolute method) measurements for the range of 300−850 nm were acquired with a Hamamatsu C11347 device. Elementary analyses were performed using a HEKAtech Euro EA 3000 Elementary analyzer. For analyses, samples were prepared in tin cups with V_2O_5 as an additive to ensure complete combustion. ESI and APCI mass spectra were recorded on a Finnigan LCQ Deca (ThermoQuest) device.

1.1 Phosphonate Ligands L1-L6 & NMR Spectra:

1.1.1 *diisopropyl benzylphosphonate (**L1**) & diisopropyl 4-bromo-benzylphosphonate (**L2**)*

Freshly distilled (bromomethyl)benzene (4.75 mL, 40.0 mmol, 1.00 eq.) or 1-bromo-4-(bromomethyl)benzene (10.0 g, 40.0 mmol, 1.00 eq.) were mixed with $P(O^iPr)_3$ (10.4 mL, 42.0 mmol, 1.05 eq.) in a 50 mL round-bottom flask. The flask was attached to a distillation bridge with Vigreux column and the mixture was heated at 165 °C for 2,5 h while forming ^{i}PrBr was distilled off. The reaction mixture was cooled to r.t. and residual ^{i}PrBr and minor side products were removed in vacuo, first at r.t. then at 100 °C. Both compounds were obtained as colorless oils. **L1**: 9.26 g, 36.2 mmol, 90%. **L2**: 13,2 g, 39.5 mmol, 99%. **L1**: **^{1}H-NMR** (400 MHz, CD_2Cl_2): δ = 7.32–7.22 (m, 5H, H2–H6), 4.61–4.52 (m, 2H, C*H*$(CH_3)_2$), 3.08 (d, 2H, $^2J_{PH}$ = 21.6 Hz, CH_2), 1.27 (d, 6H, $^3J_{HH}$ = 6.2 Hz, CH(C*H*$_3)_2$), 1.17 (d, 6H, $^3J_{HH}$ = 6.2 Hz, CH(C*H*$_3)_2$) ppm; **^{31}P{^{1}H}-NMR** (202 MHz, $CDCl_3$): δ = 24.3 (s) ppm; **IR** (ATR): 1246 (P=O), 1106 (P–O^{i}Pr) cm^{-1}. **L2**: **^{1}H-NMR** (400 MHz, $CDCl_3$): δ = 7.43 (d, 2H, $^3J_{HH}$ = 8.1 Hz, H3 +H5), 7.18–7.16 (m, 2H, H2 + H6), 4.65–4.56 (m, 2H, C*H*$(CH_3)_2$), 3.05 (d, 2H, $^2J_{PH}$ = 21.7 Hz, CH_2), 1.29 (d, 6H, $^3J_{HH}$ = 6.2 Hz, CH(C*H*$_3)_2$), 1.19 (d, 6H, $^3J_{HH}$ = 6.2 Hz, CH(C*H*$_3)_2$); **^{31}P{^{1}H}-NMR** (202 MHz, $CDCl_3$): δ = 23.6 (s) ppm; **IR** (ATR): 1244 (P=O), 1106 (P–O^{i}Pr) cm^{-1}.

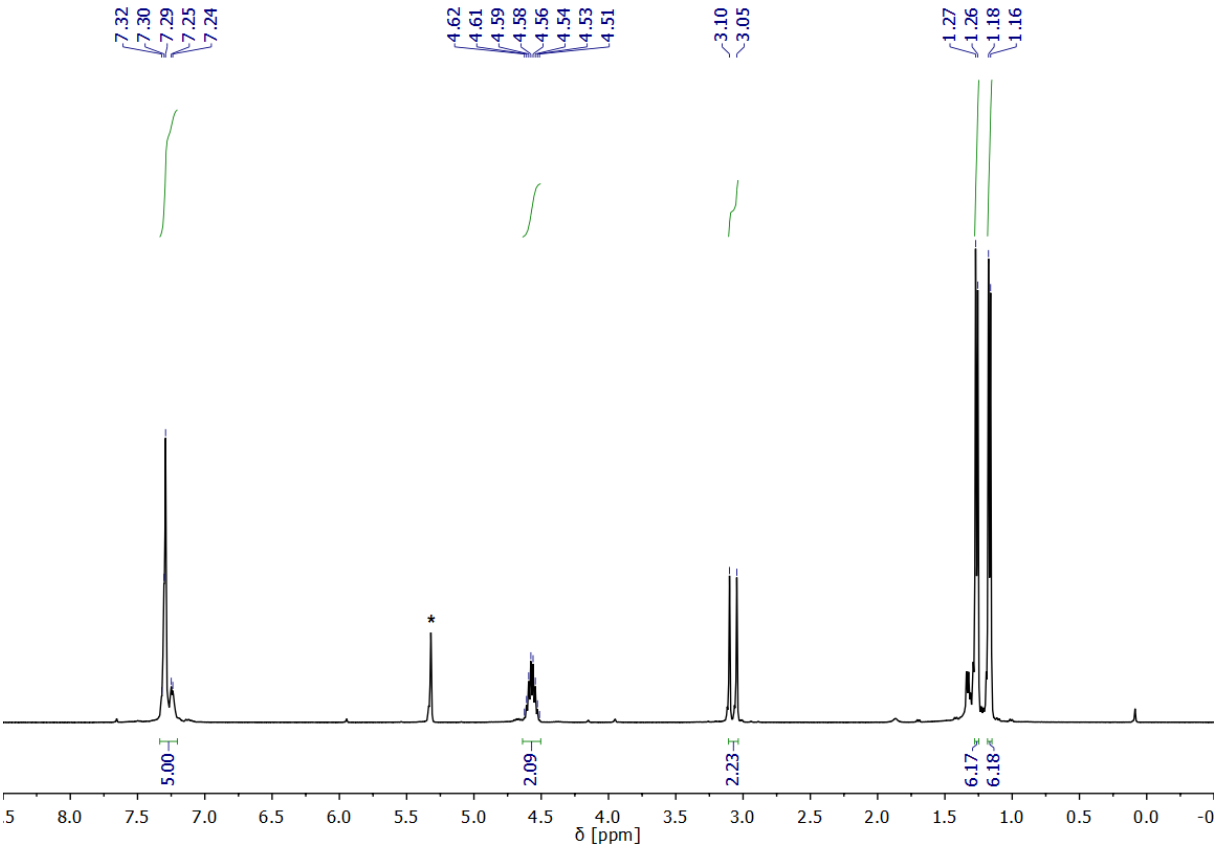

Figure 1: ^{1}H-NMR spectrum of **L1** in CD_2Cl_2 (*).

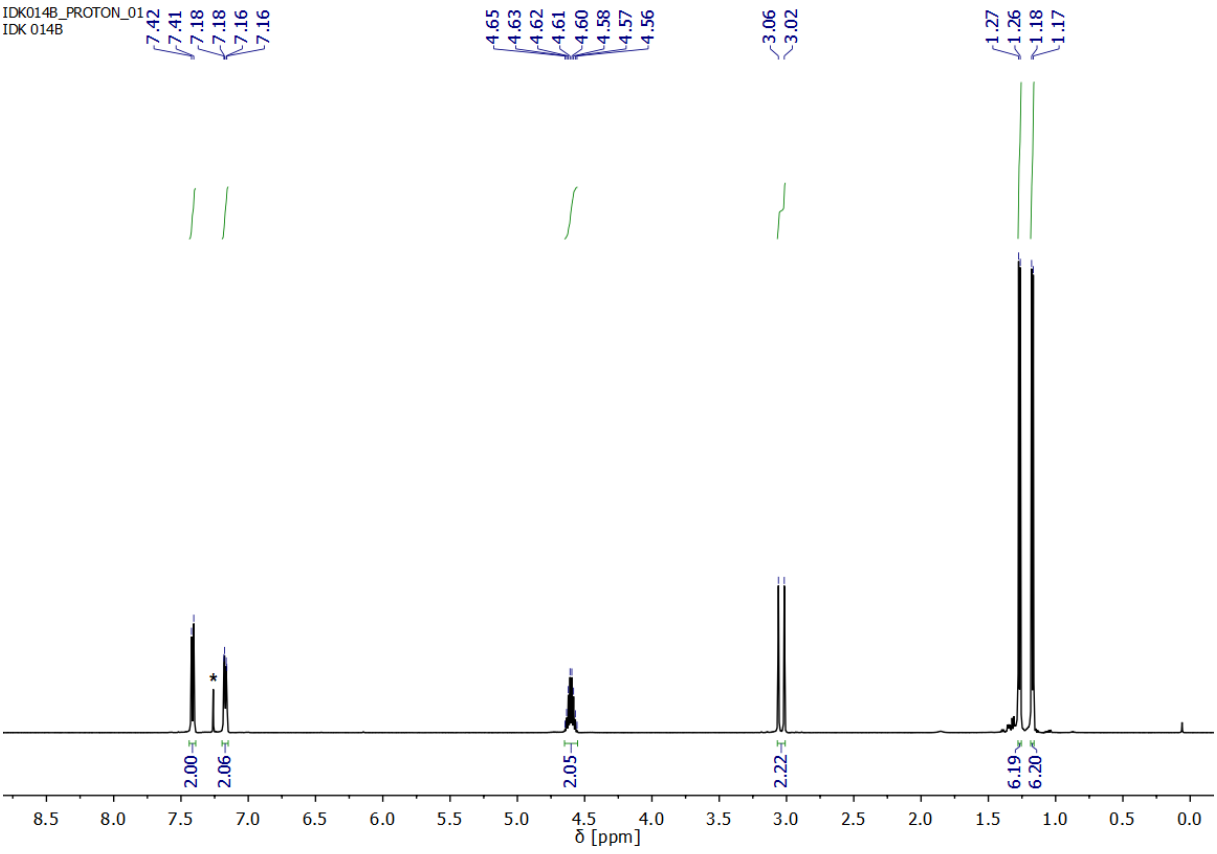

Figure 2: ^{1}H-NMR spectrum of **L2** in $CDCl_3$ (*).

1.1.2 *diisopropyl ((10-bromoanthracen-9-yl)methyl)phosphonate (L3)*

The starting material **S1** was prepared according to a modified procedure by S. E. Webber et al..[2]:

Triphenylphosphane (12.6 g, 48.0 mmol, 1.00 eq.) was dissolved under slight heating in dry MeCN (120 mL) in a 250 mL Schlenk flask capped with a rubber-septum. The solution was bubbled with argon under stirring for 15 min.. The mixture was cooled with an r.t. water-bath and Br_2 (7.62 mL, 149 mmol, 3.1 eq.) was slowly added via syringe, forming an orange solution. After cooling to r.t., 9-methanolanthracene (10.0 g, 48.0 mmol, 1.00 eq.) was slowly spatula wise added under a steady stream of argon via the open neck over a period of ca. 10 min. The neck was recapped, and the obtained suspension was vigorously stirred at r.t. for 18 h. The suspension was cooled to 0 °C, filtered, the dark yellow filter cake was washed with -20 °C MeCN (3 x 10 mL) and the obtained solid was air-dried. **S1** was obtained as a yellow powder (14.7 g, 42.0 mmol, 88%). **^{1}H-NMR** (400 MHz, $CDCl_3$): δ = 8.65–8.62 (m, 2H, H4 + H8), 8.32 (d, 2H, $^{2}J_{HH}$ = 8.4 Hz, H1 + H5), 7.70–7.61 (m, 4H, H2 + H6, H3 + H7), 5.51 (s, 2H, CH_2) ppm.

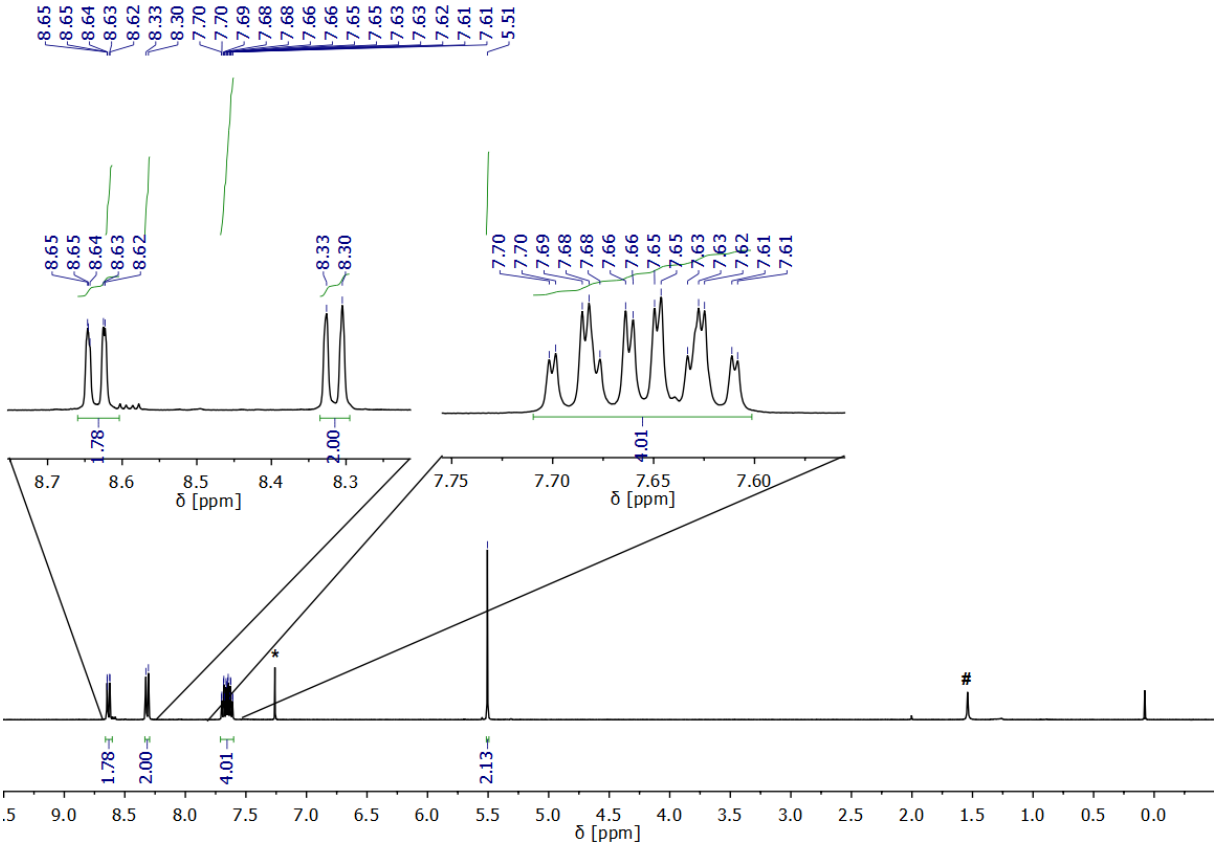

Figure 3: ^{1}H-NMR spectrum of **S1** in $CDCl_3$ (*). H_2O (#) from deuterated solvent.

9-bromo-10-(bromomethyl)anthracene (**S1**) (10.5 g, 30.0 mmol, 1.00 eq.) and $P(O^iPr)_3$ (7.77 mL, 31.5 mmol, 1.05 eq.) were mixed in a 50 mL round-bottom flask under stirring. The flask was attached to a distillation bridge with Vigreux column and the mixture was heated at 165 °C for 2.5 h. At 115-120 °C oil bath, the anthracene starting material melted completely and forming ^{i}PrBr started to distill off. The brownish-orange solution was cooled to r.t. and solidified. The solid material was ground in a mortar to obtain a yellow powder which was subsequently washed with -20 °C pentanes (3 x 50 mL), filtered (frit, P3) and dried. **L3** was obtained as a yellow powder (11.9 g, 27.3 mmol, 91%). Crystals suitable for SCXRD experiments were obtained from vapor diffusion of pentanes into a saturated solution of **L3** in THF at -20 °C. **^{1}H-NMR** (400 MHz, $CDCl_3$): δ = 8.67–8.50 (m, 2H, H4 + H8), 8.45–8.32 (m, 2H, H1 + H5), 7.61–7.56 (m, 4H, H2 + H6, H3 + H7), 4.57–4.48 (m, 2H, C*H*$(CH_3)_2$), 4.14 (d, 2H, $^{2}J_{PH}$ = 22.6 Hz, CH_2), 1.23 (d, 6H, $^{3}J_{HH}$ = 6.2 Hz, CH(C*H*$_3)_2$), 0.90 (d, 6H, $^{3}J_{HH}$ = 6.2 Hz, CH(C*H*$_3)_2$) ppm; **^{13}C{^{1}H}-NMR** (100 MHz, $CDCl_3$): δ = 131.3 (d, 2C, $^{4}J_{PC}$ = 6.8 Hz, C13 + C14), 130.5 (d, 1C, $^{5}J_{PC}$ = 4.5 Hz, C10), 128.6 (d, 2C, $^{6}J_{PC}$ = 1.7 Hz, C3 + C7), 126.9 (d, 2C, $^{5}J_{PC}$ = 1.9 Hz, C4 + C8), 126.2–126.0 (m, 4C, C1 + C5, C2 + C6), 125.5 (d, 1C, $^{2}J_{PC}$ = 11.3 Hz, C9), 123.5 (d, 2C, $^{3}J_{PC}$ = 7.9 Hz, C11 + C12), 71.1 (d, 2C, $^{2}J_{PC}$ = 7.2 Hz, *C*H$(CH_3)_2$), 28.6 (d, 1C, $^{1}J_{PC}$ = 142.1 Hz, CH_2), 24.3 (d, 2C, $^{3}J_{PC}$ = 3.6 Hz, CH(*C*H$_3)_2$), 23.8 (d, 2C, $^{3}J_{PC}$ = 5.3 Hz, CH(*C*H$_3)_2$) ppm; **^{31}P{^{1}H}-NMR** (202 MHz, $CDCl_3$): δ = 23.0 (s) ppm; **IR** (ATR) $\tilde{\nu}$ = 1246 (P=O), 1104 (P–O^{i}Pr) cm^{-1}; **MS (ESI+)** *m/z* (%): 457.06 (100) [M + Na^+]$^+$, 893.13 (95) [2M + Na^+]$^+$; **Elemental analysis** in % (calculated) $C_{21}H_{24}BrO_3P$ (435.29 g/mol): C 57.90 (57.94), H 5.55 (5.56).

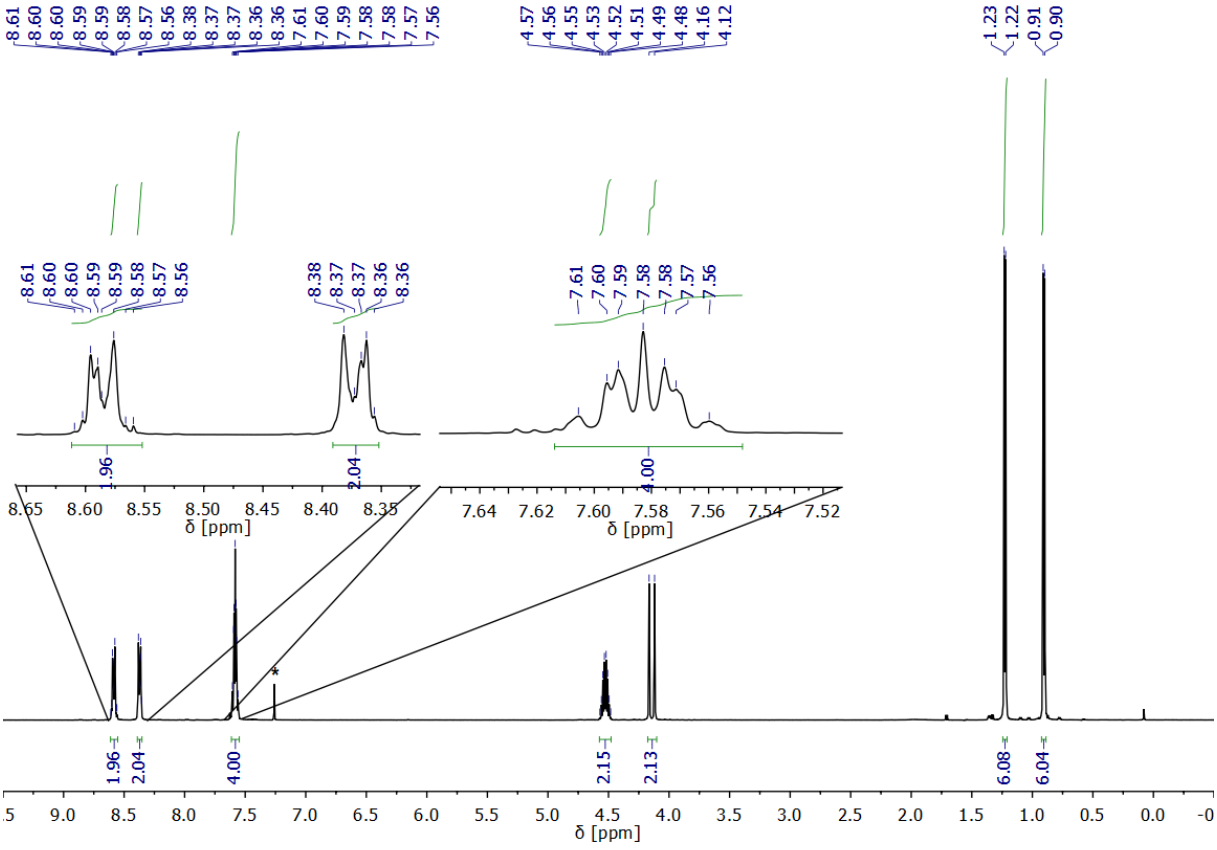

Figure 4: ^{1}H-NMR spectrum of **L3** in $CDCl_3$ (*).

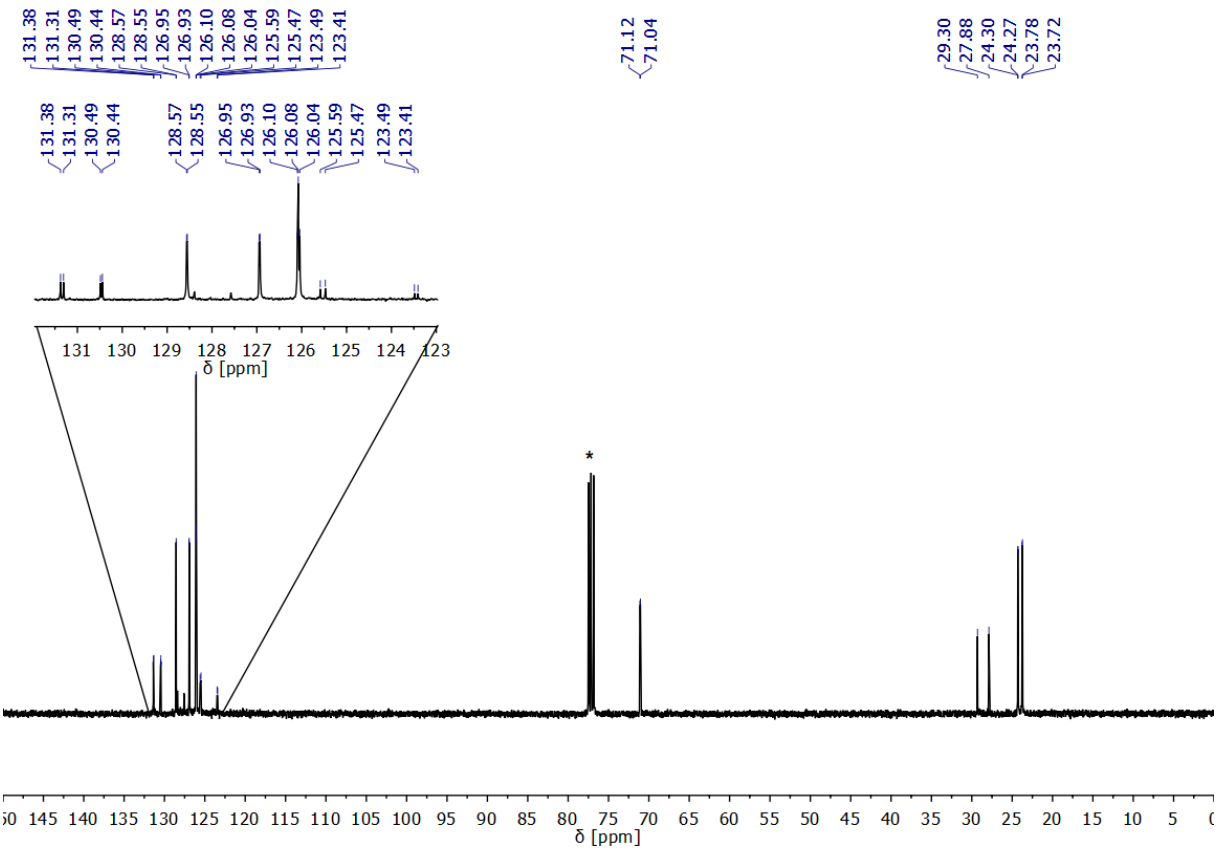

Figure 5: ^{13}C-NMR spectrum of **L3** in $CDCl_3$ (*).

1.1.3 *diisopropyl 4-(bromomethyl)benzylphosphonate (L4)*

The synthesis was performed following a modified procedure by D. Villemin et al..[3]:

In a 50 mL Schlenk flask, 1,4-bis(bromomethyl)benzene (10.0 g, 37.9 mmol, 3.00 eq.) was suspended in dry toluene (35.0 mL) and $P(O^iPr)_3$ (3.12 mL, 12.6 mmol, 1.00 eq.) was added via syringe. The flask was attached under Ar to a reflux condenser and the mixture was heated for 20 h. The mixture was cooled to r.t., the solvent was removed under reduced pressure and the residue was mixed with -20 °C MeOH (25 mL) to precipitate the excess starting material. The suspension was filtered, and the filter cake was washed with -20 °C MeOH (15 mL). The solvent from the filtrate was reduced to a minimum and the solution was stored at -20 °C to precipitate residual starting material and some of the di-phosphorylated side product. The precipitate was separated as described above, the solvent from the filtrate was removed and the crude product was purified by fractional distillation (no Vigreux-column, 2 x 10^{-2} mbar, 170 °C oil bath). **L4** was obtained as a colorless oil which becomes pale yellow over time (5.93 g, 17.0 mmol, 58%). **^{1}H-NMR** (400 MHz, $CDCl_3$): δ = 7.40–7.33 (m, 2H, H3 + H5), 7.32–7.28 (m, 2H, H2 + H6), 4.73–4.55 (m, 2H, C*H*$(CH_3)_2$), 4.50 (s, 2H, CH_2–Br), 3.12 (d, 2H, $^2J_{PH}$ = 21.8 Hz, CH_2–P), 1.30 (d, 6H, $^3J_{HH}$ = 6.2 Hz, CH(C*H*$_3)_2$), 1.19 (d, 6H, $^3J_{HH}$ = 6.2 Hz, CH(C*H*$_3)_2$) ppm; **^{13}C{^{1}H}-NMR** (100 MHz, $CDCl_3$): δ = 136.4 (d, 1C, $^5J_{PC}$ = 3.8 Hz, C4), 132.6 (d, 1C, $^2J_{PC}$ = 9.2 Hz, C1), 130.4 (d, 2C, $^3J_{PC}$ = 6.6 Hz, C2 + C6), 129.2 (d, 2C, $^4J_{PC}$ = 3.1 Hz, C3 + C5), 70.8 (d, 1C, $^2J_{PC}$ = 7.0 Hz, *C*H$(CH_3)_2$), 34.7 (d, 1C, $^1J_{PC}$ = 139.6 Hz, CH_2–P), 33.4 (d, 1C, $^6J_{PC}$ = 0.7 Hz, CH_2–Br), 24.2 (d, 2C, $^3J_{PC}$ = 3.8 Hz, CH(*C*H$_3)_2$), 23.9 (d, 2C, $^3J_{PC}$ = 5.1 Hz, CH(*C*H$_3)_2$) ppm; **^{31}P{^{1}H}-NMR** (202 MHz, $CDCl_3$): δ = 24.0 (s) ppm; **IR** (ATR) $\tilde{\nu}$ = 1243 (P=O), 1105 (P–O^{i}Pr) cm^{-1}; **MS (ESI+)** *m/z* (%): 371.04 (100) $[M + Na^+]^+$; **Elemental analysis** in % (calculated) $C_{14}H_{22}BrO_3P$ (349.20 g/mol): C 48.20 (48.15), H 6.48 (6.35).

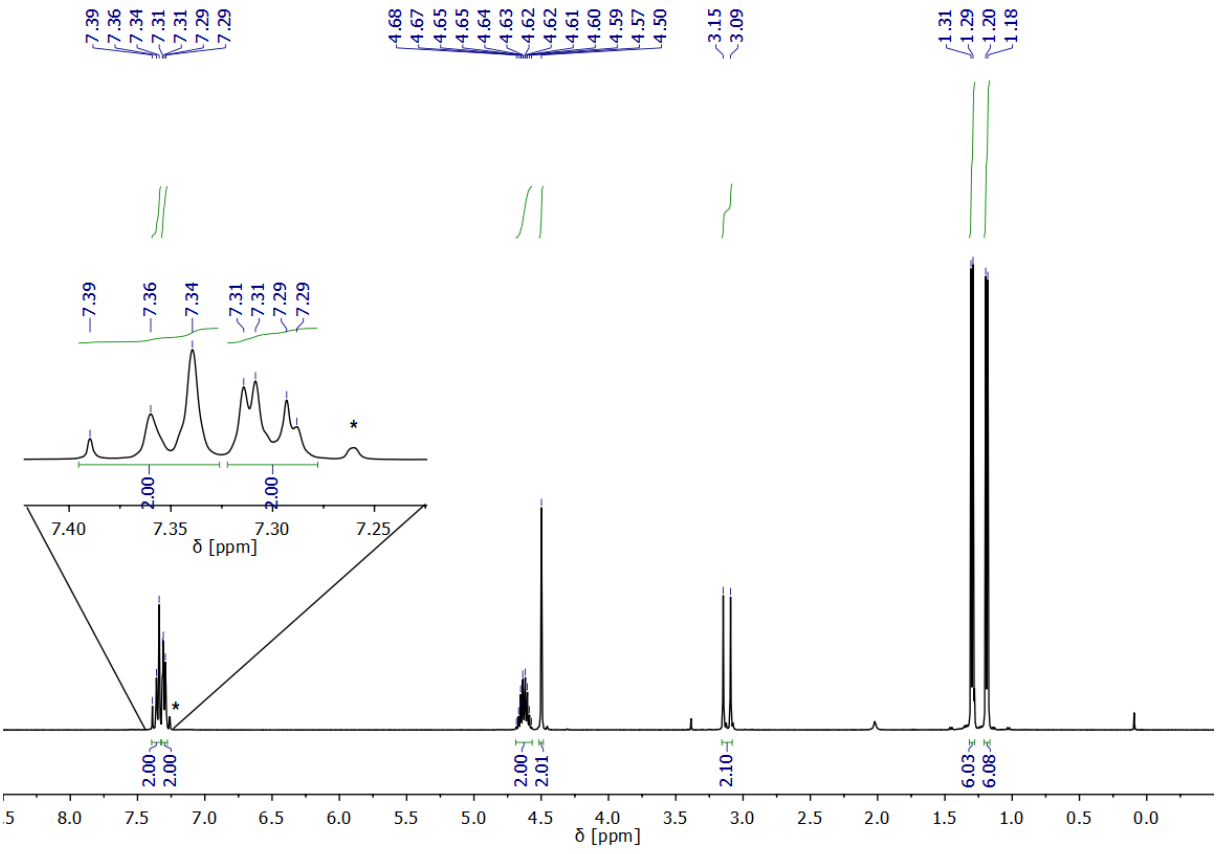

Figure 6: ^{1}H-NMR spectrum of **L4** in $CDCl_3$ (*).

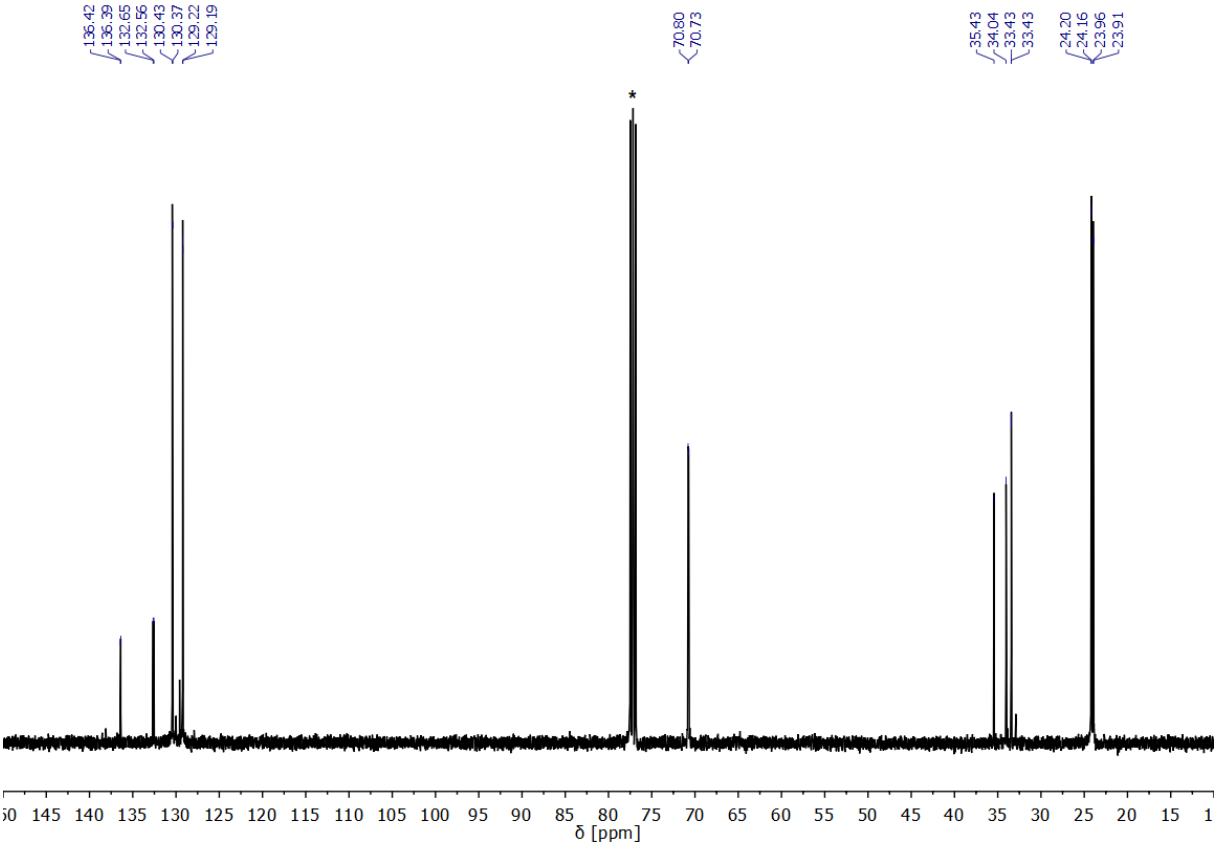

Figure 7: ^{13}C-NMR spectrum of **L4** in $CDCl_3$ (*).

1.1.4 *diisopropyl ((4'-bromo-[1,1'-biphenyl]-4-yl)methyl) phosphonate (L5)*

The first three synthesis steps (**S2**, **S3**, **S4**) were performed according to modified procedures by F. Leroux et al. and M. J. Krische et al..[4, 5]:

4,4'-dibromo-1,1'-biphenyl (31.2 g, 100 mmol, 1.00 eq.) was dissolved in dry THF (400 mL) in a 500 mL Schlenk flaks with big stirrer and cooled to -100 °C. Under vigorous stirring, *n*BuLi in hexanes (2.50 M, 40.0 mL, 100 mmol, 1.00 eq) was added dropwise via syringe over a period of ca. 15 min. Then, dry DMF (7.69 mL, 100 mmol, 1.00 eq.) was added in one portion, the cooling-bath was removed, and the mixture was stirred ad r.t. for 4 h. H_2O (50 mL) were added to the pale yellow solution and the phases were separated. The organic phase was additionally extracted with brine (50 mL) and the combined aq. phases were extracted with Et_2O (3 x 50 mL). The combined org. phases were dried over $MgSO_4$, filtered and the solvent was removed under reduced pressure yielding a 4:1 mixture of the mono- and dialdehyde. Recrystallization from neat $CHCl_3$ and subsequently from a mixture of EtOAc/hexanes (1.5:1) afforded **S2** as a white solid (18.3 g, 70.0 mmol, 70%). **^{1}H-NMR** (400 MHz, $CDCl_3$): δ = 10.05 (s, 1H, CHO), 7.95 (d, 2H, $^3J_{HH}$ = 8.2 Hz, H2 + H6), 7.70 (d, 2H, $^3J_{HH}$ = 8.2 Hz, H3 + H5), 7.60 (d, 2H, $^3J_{HH}$ = 8.5 Hz, H3' + H5'), 7.49 (d, 2H, $^3J_{HH}$ = 8.5 Hz, H2' + H6') ppm.

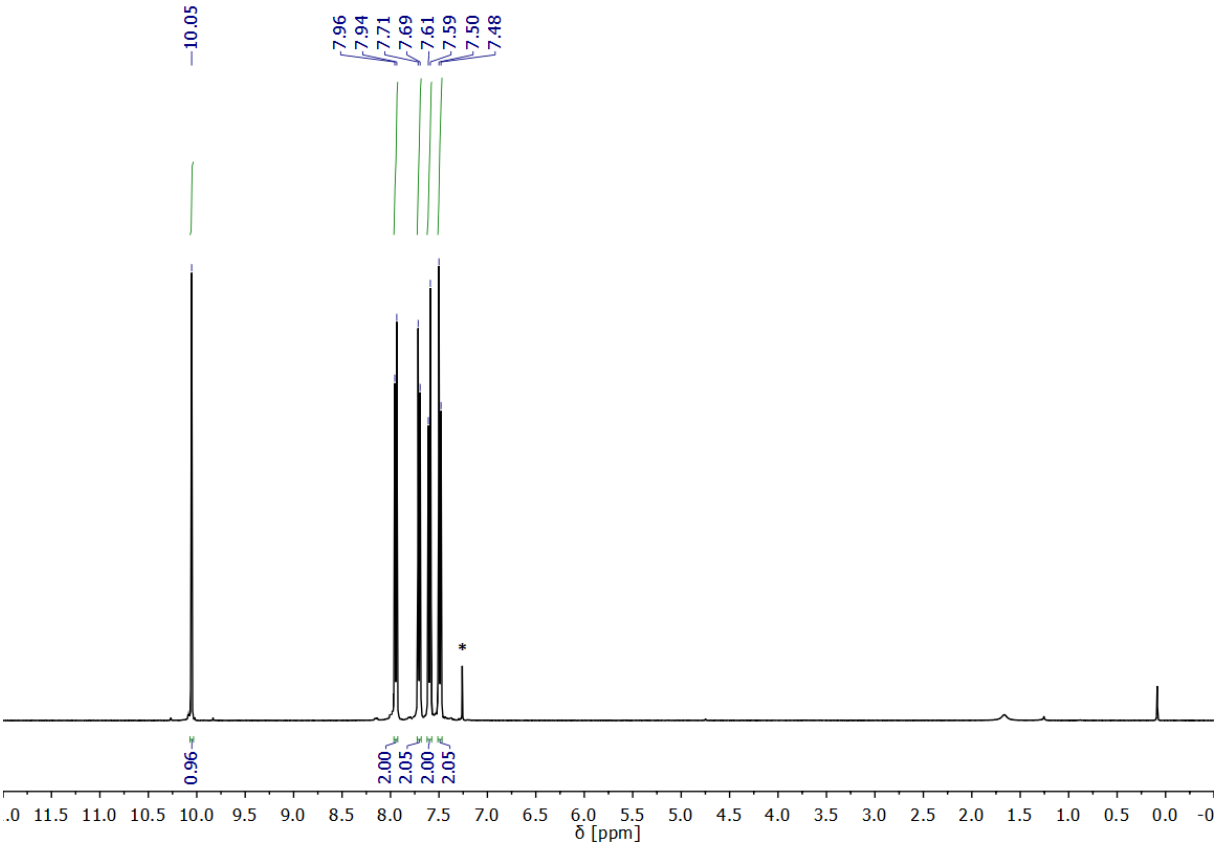

Figure 8: ^{1}H-NMR spectrum of **S2** in $CDCl_3$ (*).

4'-bromo-[1,1'-biphenyl]-4-carbaldehyde (**S2**) (15.0 g, 57.5 mmol, 1.00 eq.) was dissolved in a mixture of dry THF (100 mL) and dry MeOH (60 mL) in a 250 mL round-bottom flask and cooled with a r.t. water-bath. $NaBH_4$ tablets (3.26 g, 86.3 mmol, 1.50 eq.) were added over a period of ca. 20 min. and the mixture was stirred at r.t. overnight. An aq. solution of HCl (ca. 2 M, 60 mL) was slowly added and the formed precipitate was filtered off and washed several times with Et_2O. The filtrate was transferred to a separation-funnel, additional H_2O (50 mL) was added, and the phases were separated. The aq. phase was extracted with Et_2O (3 x 50 mL). The combined org. phases were extracted with brine (2 x 50 mL), dried over $MgSO_4$, filtered, and evaporated. After recrystallization from EtOAc/hexanes (1:1), **S3** was obtained as a white solid (10.5 g, 39.7 mmol, 69%). **^{1}H-NMR** (400 MHz, $CDCl_3$): δ = 7.61–7.52 (m, 4H, H2' + H6', H3' + H5'), 7.48–7.38 (m, 4H, H2 + H6, H3 + H5), 4.74 (s, 2H, CH_2), 1.77 (s, 1H, OH) ppm.

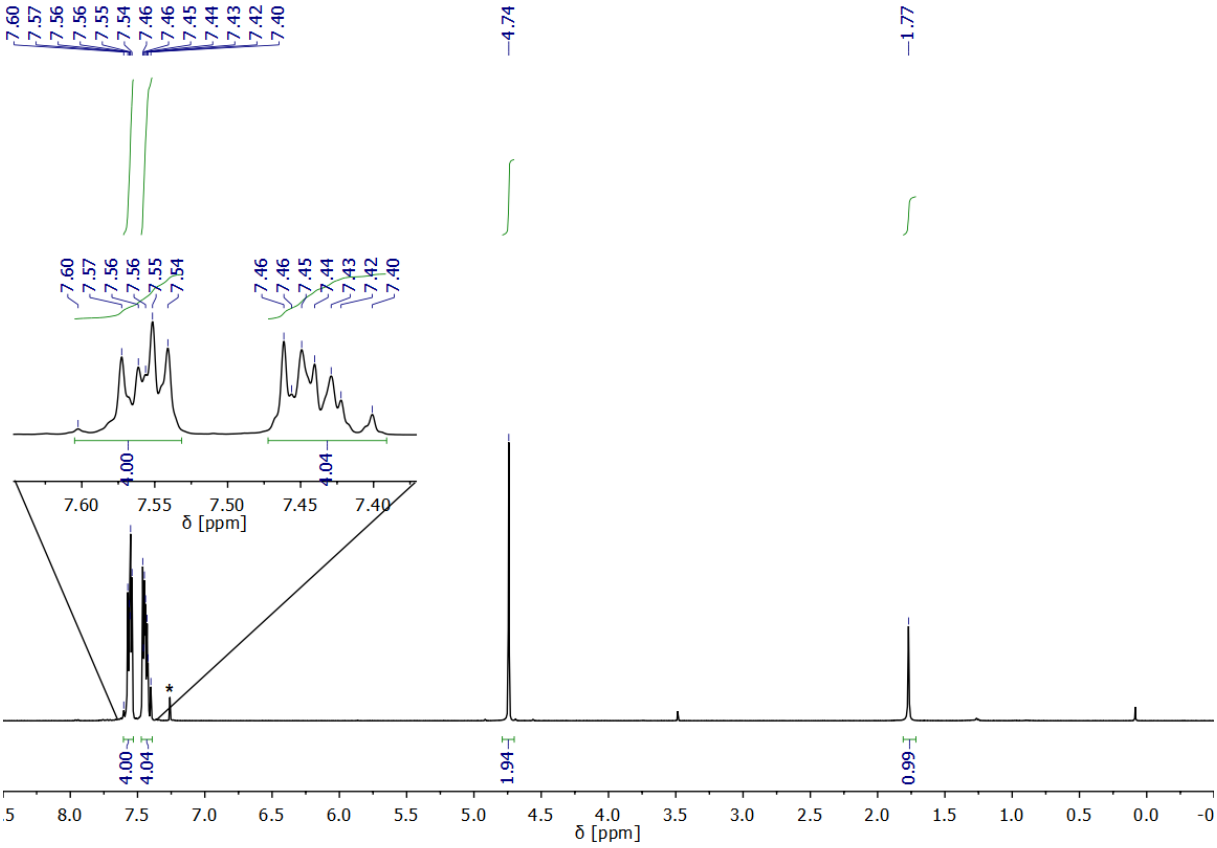

Figure 9: ^{1}H-NMR spectrum of **S3** in $CDCl_3$ (*).

(4'-bromo-[1,1'-biphenyl]-4-yl)methanol (**S3**) (11.3 g, 42.9 mmol, 1.00 eq.) was dissolved in dry THF (120 mL) in a 250 mL Schlenk flask. The flask was attached to a dropping-funnel under argon and cooled to 0 °C. The dropping-funnel was charged with dry THF (40 mL) and PBr_3 (2.85 mL, 30.0 mmol, 0.7 eq.) and the mixture was slowly dropwise added over a period of ca. 45 min.. The cooling-bath was removed, and the solution was stirred at r.t. overnight. The mixture was poured onto 100 g of ice and was transferred to a separation-funnel. The aq. phase was extracted with Et_2O (3 x 50 mL) and the combined org. phases were extracted with brine (3 x 50 mL). The org. phase was dried over Na_2SO_4, filtered, and evaporated. Washing of the obtained off-white solid in a Büchner-funnel with -20 °C MeOH and subsequent drying afforded **S4** as a white solid (11.0 g, 33.7 mmol, 79%). **^{1}H-NMR** (400 MHz, $CDCl_3$): δ = 7.59–7.51 (m, 4H, H2' + H6', H3' + H5'), 7.49–7.42 (m, 4H, H2 + H6, H3 + H5), 4.54 (s, 2H, CH_2) ppm.

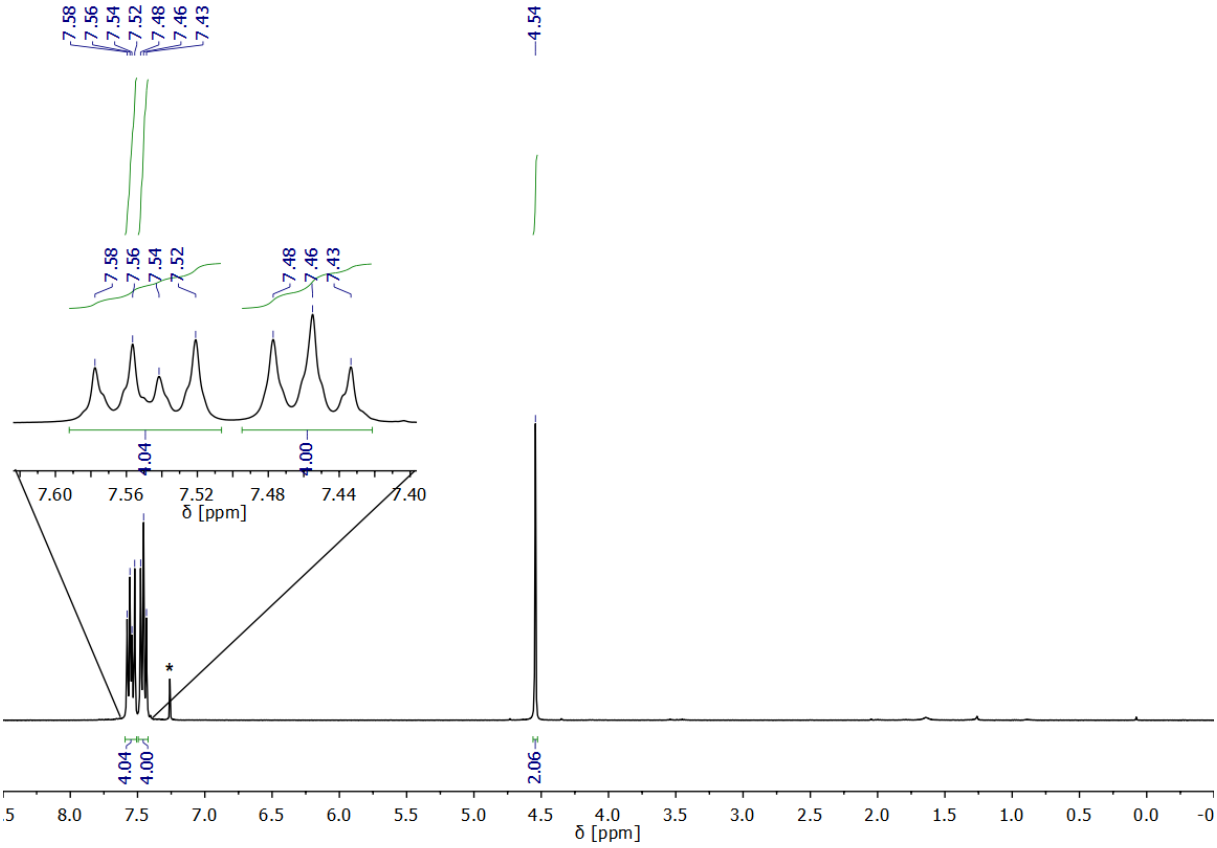

Figure 10: ^{1}H-NMR spectrum of **S4** in $CDCl_3$ (*).

4-bromo-4'-(bromomethyl)-1,1'-biphenyl (**S4**) (13.5 g 41.4 mmol, 1.00 eq.) and $P(O^{i}Pr)_3$ (10.7 mL, 43.5 mmol, 1.05 eq.) were mixed in a 50 mL round-bottom flask under stirring. The flask was attached to a distillation bridge with Vigreux column and the mixture was heated at 165 °C for 2.5 h. The biphenyl starting material melted at 95-100 °C oil bath. At 135 °C oil bath, the formed ^{i}PrBr started to distill off. The mixture was cooled to r.t. forming an opaque oil. The opaque oil was diluted with MeOH and filtered to obtain a clear and colorless to pale yellow oil. First, residual ^{i}PrBr was removed under reduced pressure (2.3 x 10^{-2} mbar) at r.t., and then the temp. was successively raised to 50 °C and 100 °C. Compound **L5** was obtained as an oil which solidified to an off-white solid upon standing (15.9 g, 38.6 mmol, 93%). **^{1}H-NMR** (400 MHz, $CDCl_3$): δ = 7.56–7.51 (m, 2H, H3' + H5'), 7.48 (d, 2H, $^{3}J_{HH}$ = 8.0 Hz, H2' + H6'), 7.43 (d, 2H, $^{3}J_{HH}$ = 8.5 Hz, H3 + H5), 7.37 (dd, 2H, $^{3}J_{HH}$ = 8.2, $^{4}J_{HH}$ = 2.3 Hz, H2 + H6), 4.68–4.56 (m, 2H, C*H*$(CH_3)_2$), 3.13 (d, 2H, $^{2}J_{PH}$ = 21.8 Hz, CH_2), 1.29 (d, 6H, $^{3}J_{HH}$ = 6.2 Hz, CH(C*H*$_3)_2$), 1.19 (d, 6H, $^{3}J_{HH}$ = 6.2 Hz, CH(C*H*$_3)_2$) ppm; **^{13}C{^{1}H}-NMR** (100 MHz, $CDCl_3$): δ = 139.8 (d, 1C, $^{6}J_{PC}$ = 1.4 Hz, C1'), 138.5 (d, 1C, $^{5}J_{PC}$ = 3.8 Hz, C1), 131.9 (s, 2C, C3' + C5'), 131.7 (d, 1C, $^{2}J_{PC}$ = 9.2 Hz, C4), 130.5 (d, 2C, $^{3}J_{PC}$ = 6.7 Hz, C3 + C5), 128.7 (d, 2C, $^{7}J_{PC}$ = 0.7 Hz, C2' + C6'), 127.0 (d, 2C, $^{4}J_{PC}$ = 3.1 Hz, C2 + C6), 121.6 (s, 1C, C4'), 70.7 (d, 2C, $^{2}J_{PC}$ = 7.0 Hz, *C*H$(CH_3)_2$), 34.6 (d, 1C, $^{1}J_{PC}$ = 139.5 Hz, CH_2), 24.2 (d, 2C, $^{3}J_{PC}$ = 3.8 Hz, CH(*C*H$_3)_2$), 24.0 (d, 2C, $^{3}J_{PC}$ = 5.1 Hz, CH(*C*H$_3)_2$) ppm; **^{31}P{^{1}H}-NMR** (202 MHz, $CDCl_3$): δ = 24.4 (s) ppm; **IR** (ATR) $\tilde{\nu}$ = 1236 (P=O), 1108 (P–O^{i}Pr) cm^{-1}; **MS (ESI+)** *m/z* (%): 411.17 (90) $[M + H^+]^+$, 823.32 (100) $[2M + H^+]^+$; **Elemental analysis** in % (calculated) $C_{19}H_{24}BrO_3P$ (411.27 g/mol): C 55.74 (55.49), H 5.95 (5.88).

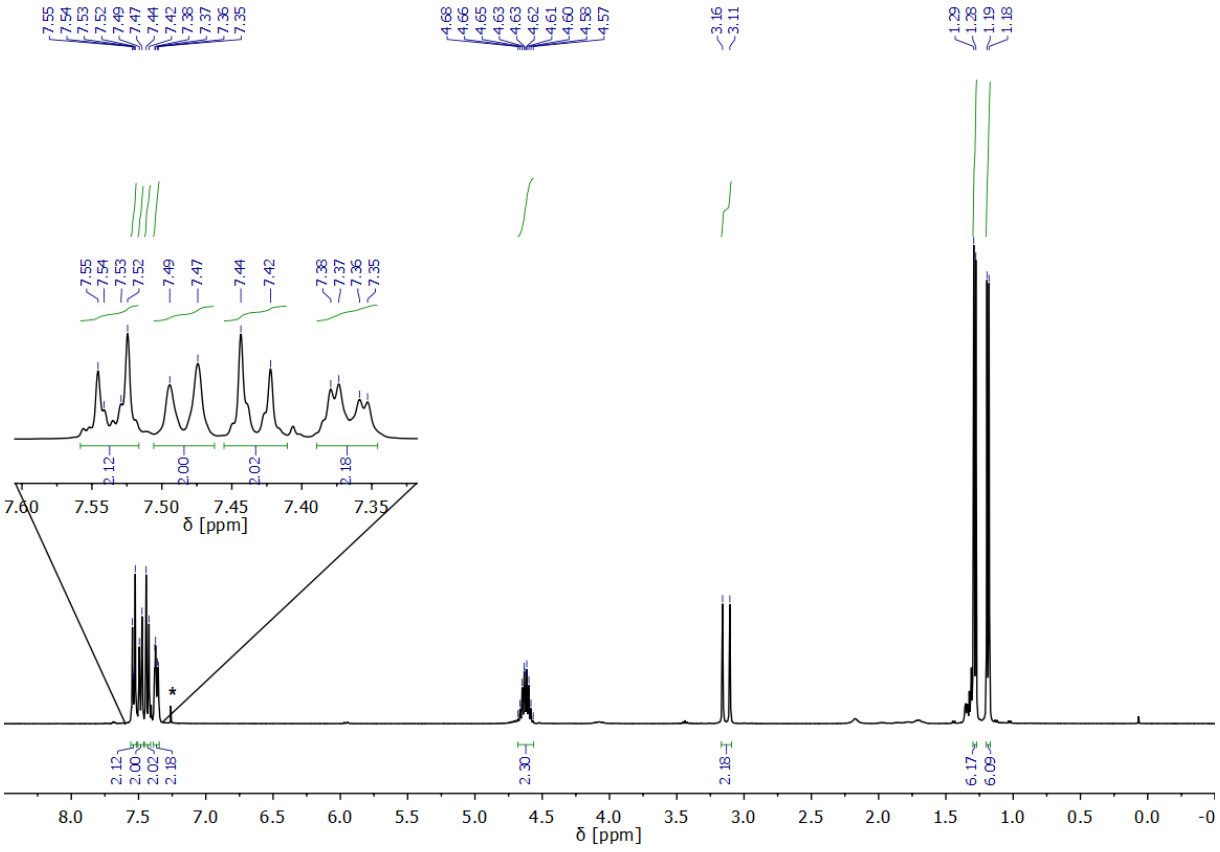

Figure 11: ^{1}H-NMR spectrum of **L5** in $CDCl_3$ (*).

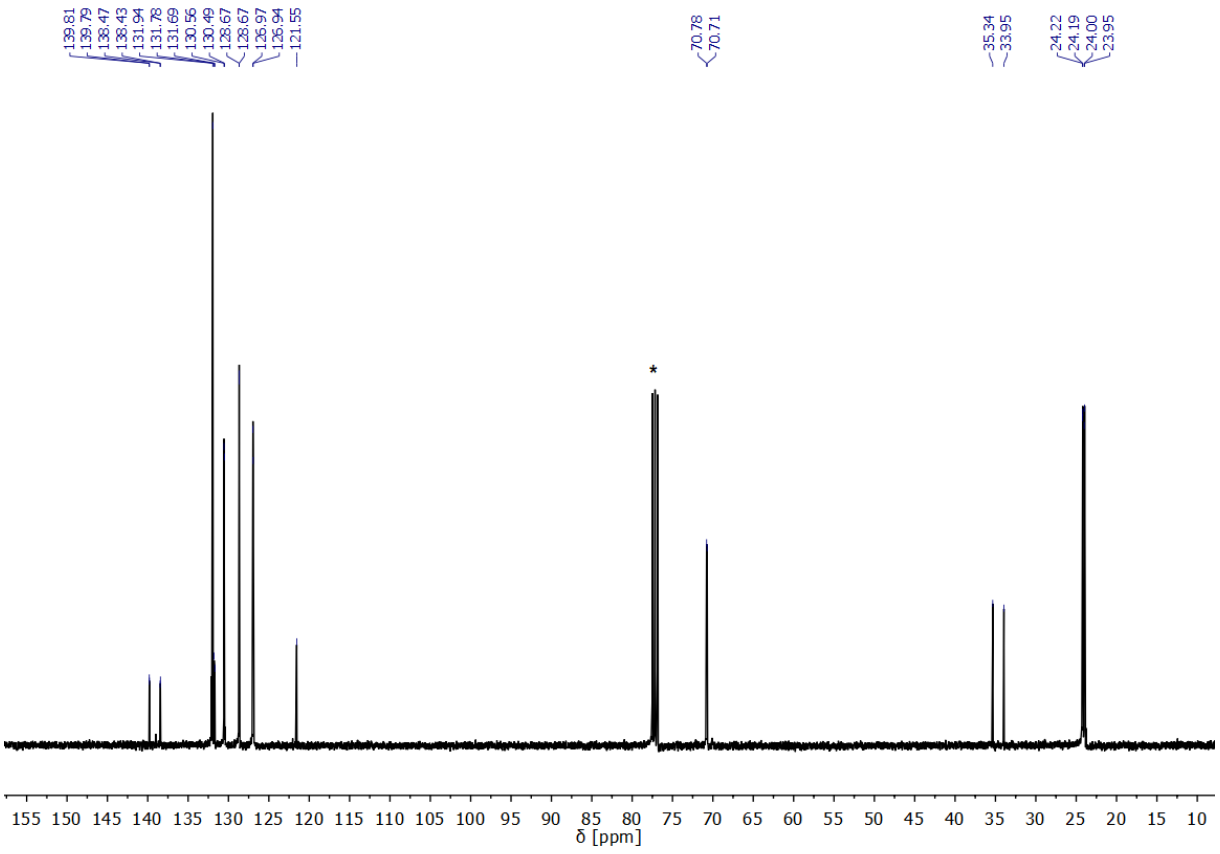

Figure 12: ^{13}C-NMR spectrum of **L5** in $CDCl_3$ (*).

1.1.5 *diisopropyl 4-bromo-2,3,5,6-tetrafluorobenzylphosphonate (L6)*

The first two synthesis steps (**S5**, **S6**) were performed according to modified procedures by A. Pfaltz et al..[6]:

In a 100 mL pressure proof Schlenk tube with screw cap and Young valve, water free LiBr (8.86 g, 102 mmol, 1.13 eq.) from the glovebox was suspended in dry NMP (50 mL). 2,3,4,5,6-pentafluorobenzaldehyde (11.2 mL, 90.3 mmol, 1.00 eq.) was molten in a 45 °C water-bath, added via syringe and the yellow solution was heated at 165 °C for 3.5 h. The formed brown suspension was cooled to r.t., filtered over celite at ambient atmosphere (frit P3), and the filter cake was washed with NMP (5 x 10 mL). H_2O (200 mL) was added under stirring to the filtrate and the formed beige precipitate was recovered via filtration (frit P3), washed with H_2O (3 x 20 mL), air-dried on a filter paper and additionally dried in a desiccator over $CaCl_2$. The filtrate was extracted with Et_2O (3 x50 mL) and the solvent was removed. The orange oily residue was diluted with H_2O (10 mL) and the formed precipitate was recovered and dried as above. The combined crude products were recrystallized from Et_2O, decanted hot, cooled to r.t. and stored at -20 °C. The formed crystals were recovered by decanting the supernatant solution, washing the solid material with -20 °C Et_2O (2 x 15 mL) and drying. Successive reduction of the mother liquor volume yielded **S5** in several crops as beige to amber crystals (17.7 g, 69.0 mmol, 76%). **^{1}H-NMR** (400 MHz, $CDCl_3$): δ = 10.3 (s, 1H, CHO) ppm; **^{19}F-NMR** (375 MHz, $CDCl_3$): δ = -131.3–-131.4 (m, 2F, F3 + F5), -141.0– -141.1 (m, 2F, F2 + F6) ppm.

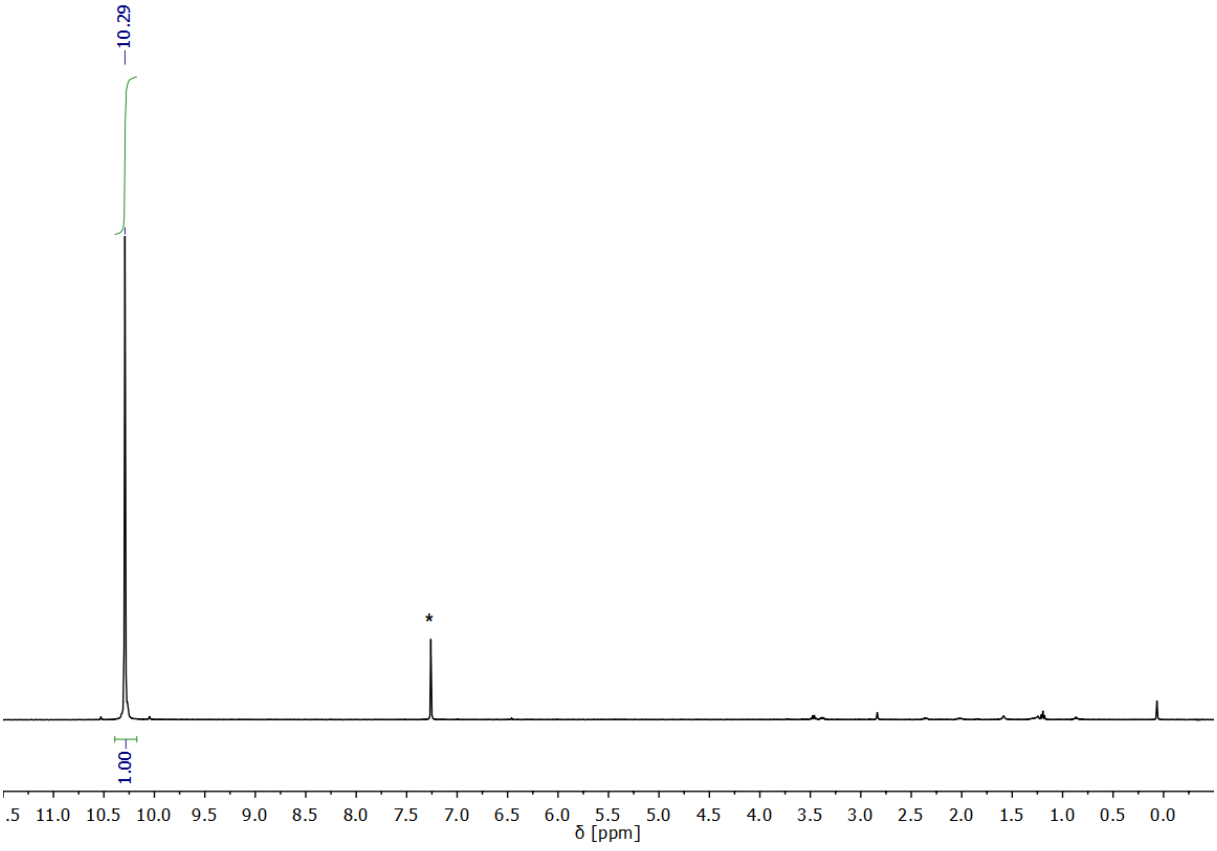

Figure 13: ^{1}H-NMR spectrum of **S5** in $CDCl_3$ (*).

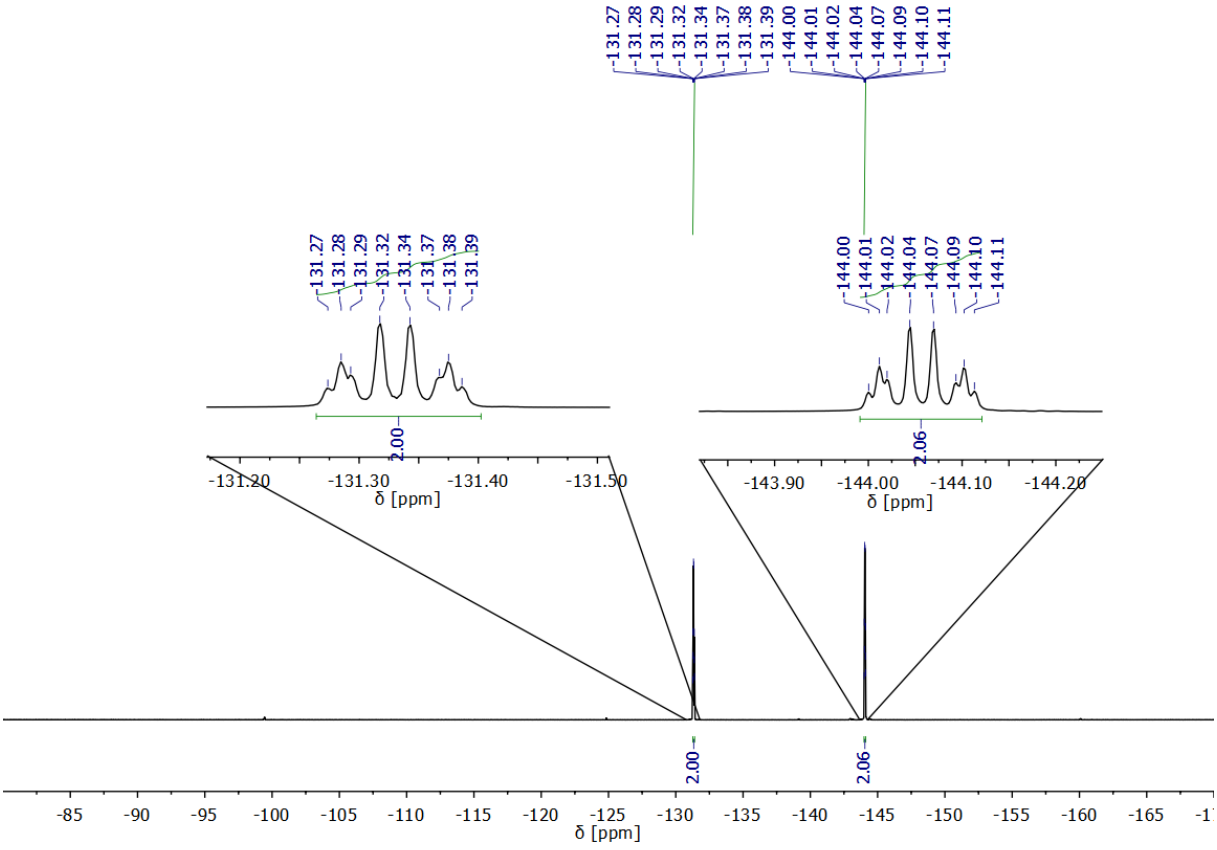

Figure 14: ^{19}F-NMR spectrum of **S5** in $CDCl_3$.

In a 250 mL round-bottom flask, 4-bromo-2,3,5,6-tetrafluorobenzaldehyde (**S5**) (18.4 g, 71.6 mmol, 1.00 eq.) was dissolved in dry MeOH (150 mL) and cooled to 0 °C. $NaBH_4$ (4.06 g, 107 mmol, 1.50 eq.) was portion wise added over a period of ca. 30 min., the mixture was stirred at 0 °C for additional 1 h, then at r.t. for 3 h. The formed pale-yellow solution was poured into a 0 °C aq. HCl solution (ca. 3.6%, 600 mL) and was stirred for 5 min.. The formed pale-yellow precipitate was recovered by filtration (frit P3), washed thoroughly with H_2O (4 x 30 mL), air-dried on a filter paper and additionally dried in a desiccator over $CaCl_2$. The filtrate was evaporated in a big bowl to a volume of ca. 100 mL within several days, the formed precipitate was recovered via filtration, washed with H_2O (4 x 20 mL) and dried as above. **S6** was obtained as an off-white solid (13.3 g, 51.4 mmol, 72%). **^{1}H-NMR** (400 MHz, $CDCl_3$): δ = 4.79 (s, 2H, CH_2), 2.37 (s, 1H, OH) ppm; **^{19}F-NMR** (375 MHz, $CDCl_3$): δ = -133.1– -133.2 (m, 2F, F3 + F5), -143.3– -143.4 (m, 2F, F2 + F6) ppm.

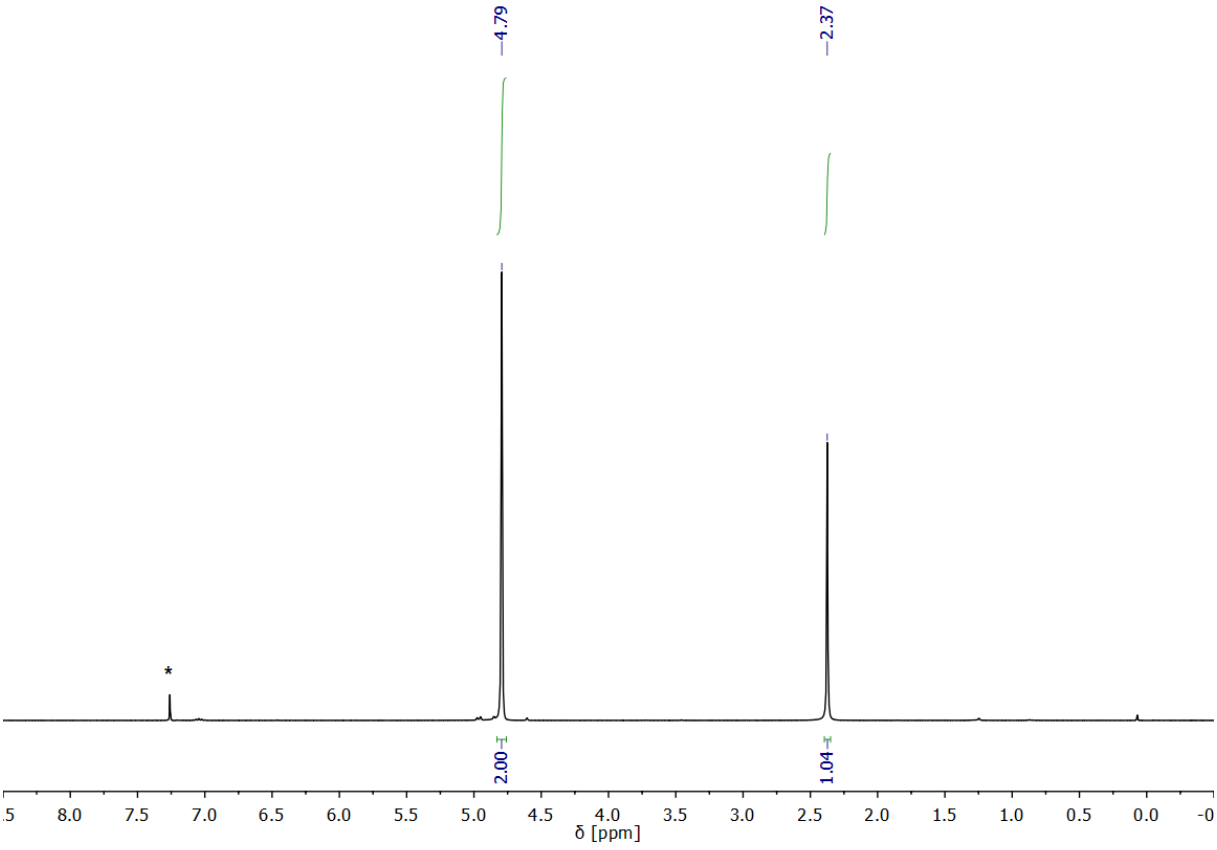

Figure 15: ^{1}H-NMR spectrum of **S6** in $CDCl_3$ (*).

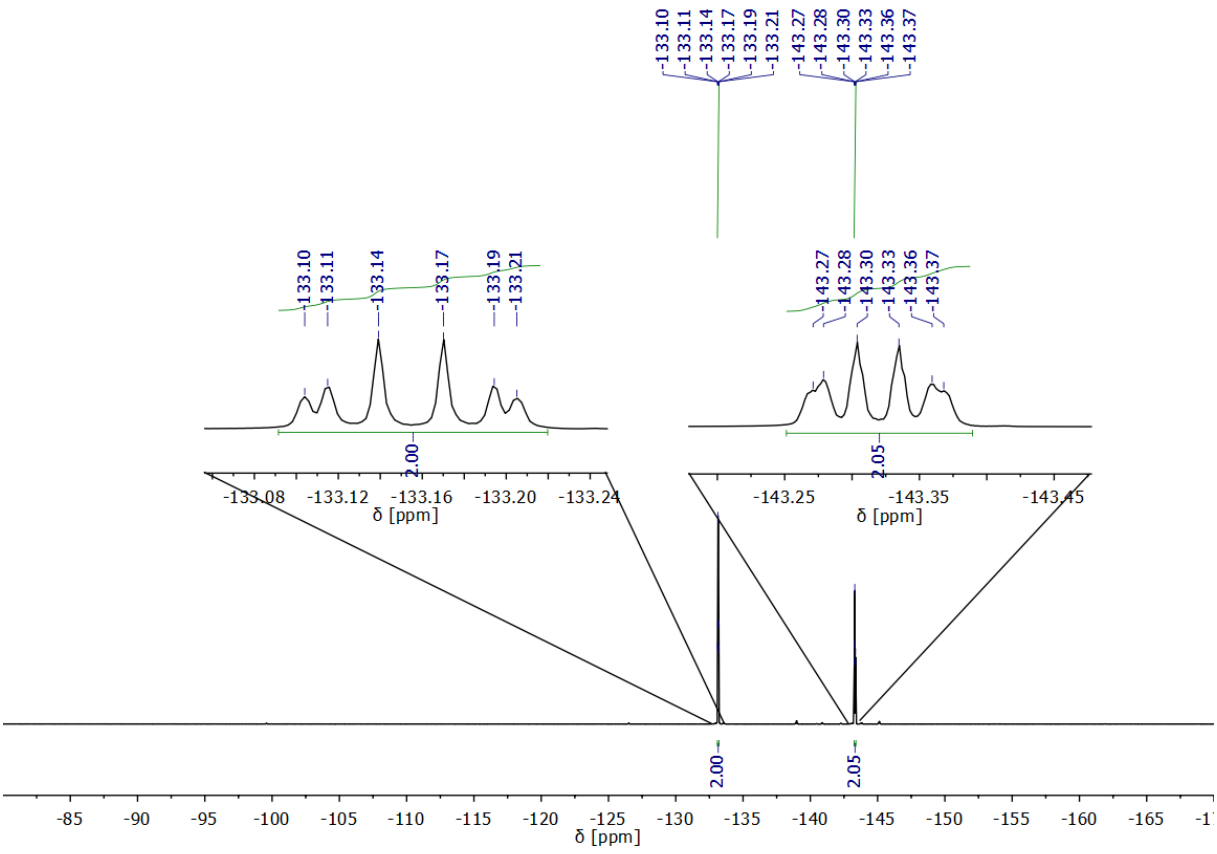

Figure 16: ^{19}F-NMR spectrum of **S6** in $CDCl_3$.

(4-bromo-2,3,5,6-tetrafluorophenyl)methanol (**S6**) (15.0 g, 57.9 mmol, 1.00 eq.) was dissolved in dry THF (150 mL) in a 250 mL Schlenk flask. The flask was attached to a dropping-funnel under argon and cooled to 0 °C. The dropping-funnel was charged with dry THF (40 mL) and PBr_3 (3.85 mL, 40.5 mmol, 0.7 eq.) and the mixture was slowly dropwise added over a period of ca. 45 min.. The cooling-bath was removed, and the solution was stirred at r.t. overnight. The yellow solution was poured under stirring into H_2O (300 mL) and was transferred to a separation-funnel. The aq. phase was extracted with Et_2O (3 x 50 mL) and the combined org. phases were extracted with brine (3 x 50 mL). The org. phase was dried over Na_2SO_4, filtered, and evaporated. The obtained orange oil was purified by fractional distillation (2.5 x 10^{-2} mbar, 105 °C oil bath, Vigreux column coated with aluminum foil) yielding **S7** (bp. 45-47 °C) as a colorless oil (17.1 g, 53.3 mmol, 92%). **^{1}H-NMR** (400 MHz, $CDCl_3$): δ = 4.50 (s, 2H, CH_2) ppm; **^{13}C{^{1}H}-NMR** (100 MHz, $CDCl_3$): δ = 146.4–146.0 (m, 2C, C3 + C5) 143.9–143.5 (m, 2C, C2 + C6), 116.7 (t, 1C, $^2J_{FC}$ = 17.0 Hz, C1), 101.0 (tt, 1C, $^2J_{FC}$ = 22.5, $^3J_{FC}$ = 2.0 Hz, C4), 16.4–16.3 (m, 1C, CH_2) ppm; **^{19}F-NMR** (375 MHz, $CDCl_3$): δ = -132.4– -132.6 (m, 2F, F3 + F5), -141.2– -141.4 (m, 2F, F2 + F6) ppm; **IR** (ATR) $\tilde{\nu}$ = 2124, 2095, 1638, 1490, 1463, 1408, 1274, 1218, 1067, 1123, 1076, 1042, 1004, 974, 860, 827, 646, 611 cm^{-1}; **GC-MS (EI, 70 eV)** *m/z* (%): 321.85 (5) $[M + H^+]^+$, 240.92 (100) $[M - Br]^{\cdot+}$, 162.00 (75) $[M - 2Br]^{\cdot+}$, 143.00 (40) $[M - 2Br - F]^{\cdot+}$; **Elemental analysis** in % (calculated) $C_7H_2Br_2F_4$ (321.89 g/mol): C 26.52 (26.12), H 0.65 (0.63).

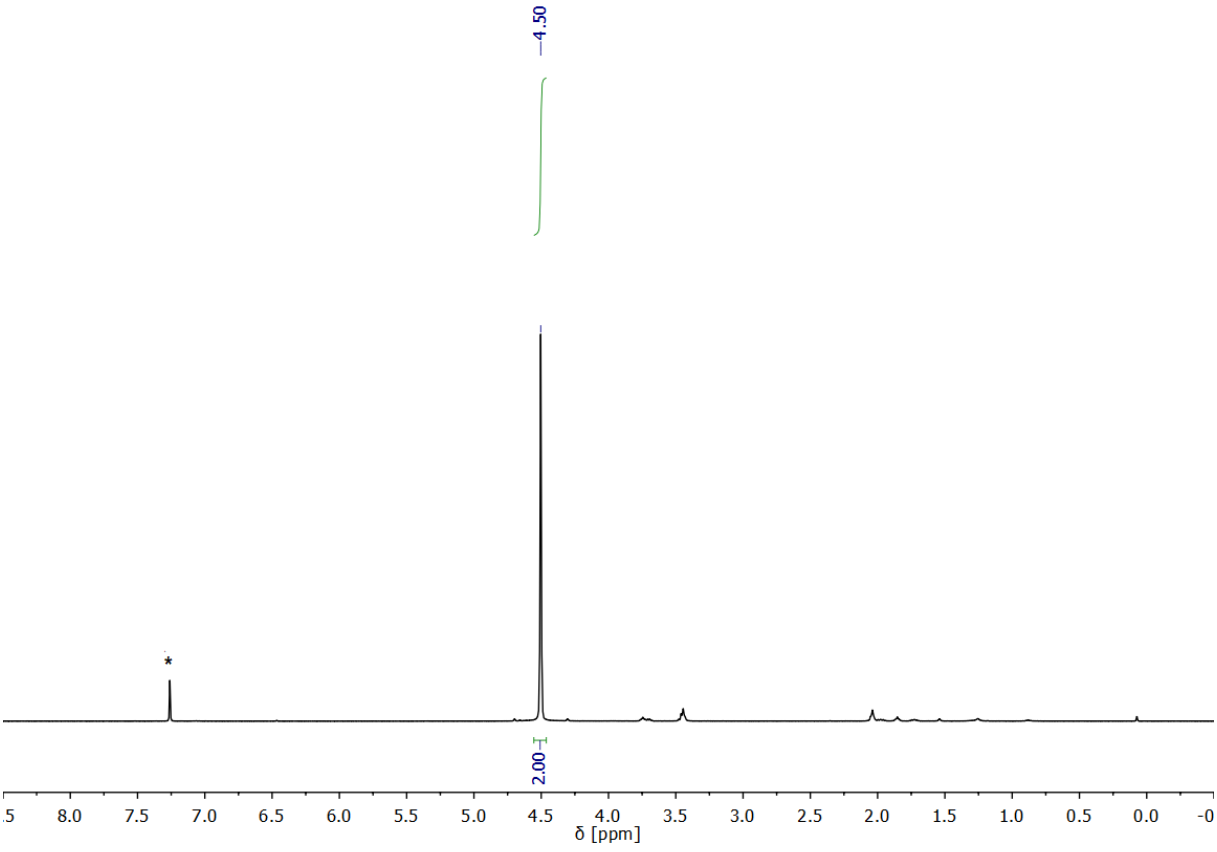

Figure 17: ^{1}H-NMR spectrum of **S7** in $CDCl_3$ (*) + a minor impurity.

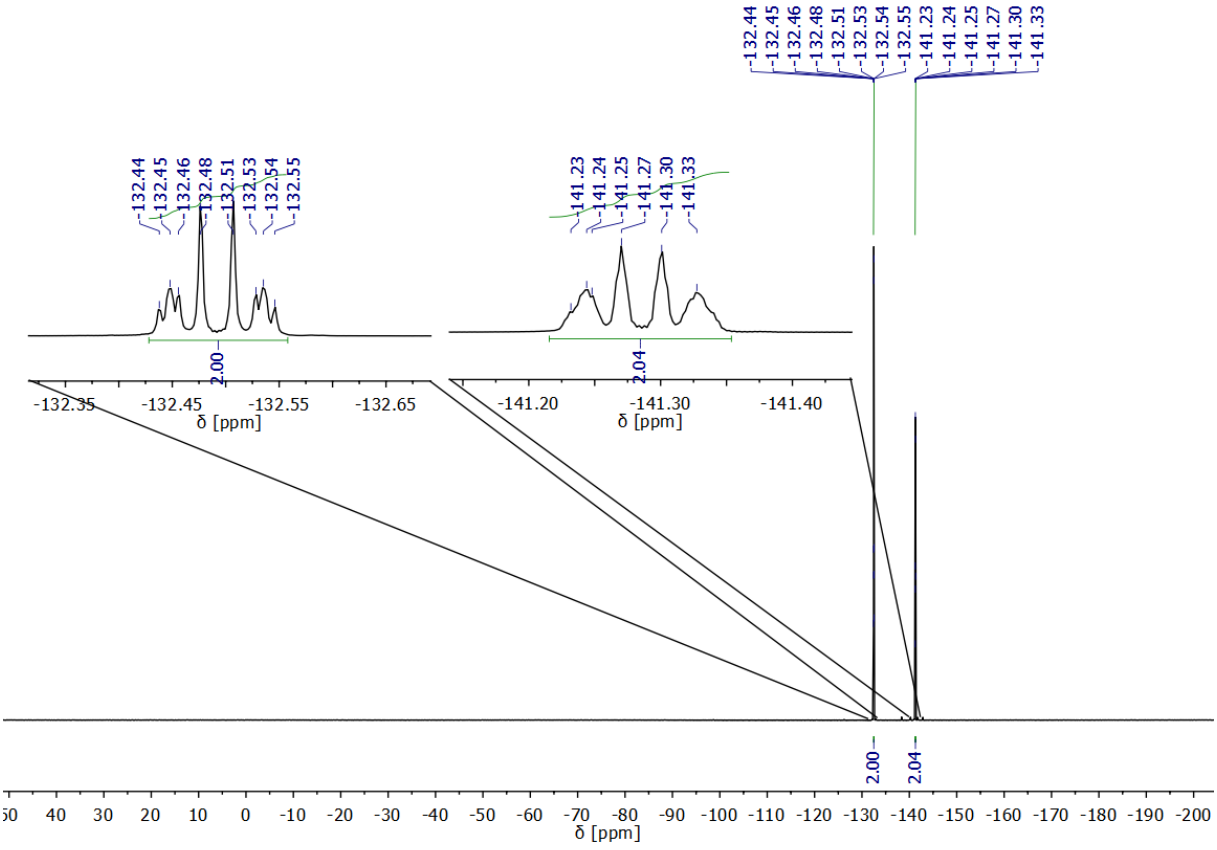

Figure 18: ^{19}F-NMR spectrum of **S7** in $CDCl_3$.

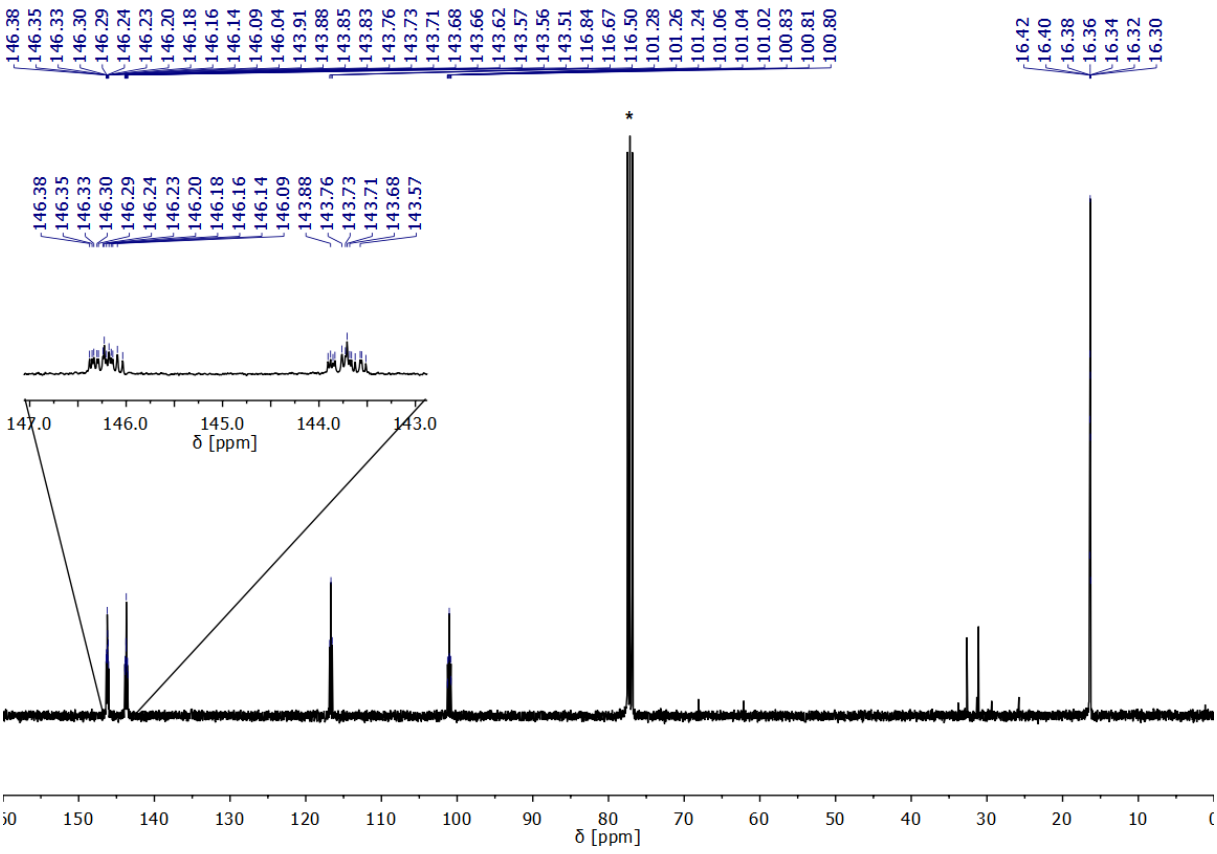

Figure 19: ^{13}C-NMR spectrum of **S7** in $CDCl_3$ (*) + a minor impurity.

1-bromo-4-(bromomethyl)-2,3,5,6-tetrafluorobenzene (**S7**) (17.1 g 53.1 mmol, 1.00 eq.) and $P(O^iPr)_3$ (13.8 mL, 55.8 mmol, 1.05 eq.) were mixed in a 50 mL round-bottom flask under stirring. The flask was attached to a distillation bridge with Vigreux column and the mixture was heated at 165 °C for 2.5 h. At 130 °C, the formed ^{i}PrBr started to distill off. The colorless oil was cooled to r.t.. First, residual ^{i}PrBr and most side products/impurities were distilled off under reduced pressure (5.3×10^{-3} mbar) at r.t., then the temp. was successively raised to 160 °C. Further workup via flash chromatography (PF-30SIHP-F0040 column; Gradient: 3 CV neat DCM => 1 CV to reach DCM/EtOAc (9:1) => 10 CV DCM/EtOAc (9:1)) yielded **L6** as a colorless oil which solidifies to a white solid upon standing (19.5 g, 47.8 mmol, 90%). **^{1}H-NMR** (400 MHz, $CDCl_3$): δ = 4.72–4.61 (m, 2H, C*H*$(CH_3)_2$), 3.16 (d, 2H, $^2J_{PH}$ = 21.6 Hz, CH_2), 1.25 (d, 12H, $^3J_{HH}$ = 6.2 Hz, CH(C*H*$_3)_2$) ppm; **^{13}C{^{1}H}-NMR** (100 MHz, $CDCl_3$): δ = 146.6–145.9 (m, 2C, C3 + C5), 144.1–143.5 (m, 2C, C2 + C6), 112.2 (td, 1C, $^2J_{FC}$ = 18.2, $^2J_{PC}$ = 10.5 Hz, C1), 98.4 (tdd, 1C, $^2J_{FC}$ = 23.0, $^5J_{PC}$ = 4.5, $^3J_{FC}$ = 2.1 Hz, C4), 71.7 (d, 2C, $^2J_{PC}$ = 6.9 Hz, *C*H$(CH_3)_2$), 24.1 (d, 2C, $^3J_{PC}$ = 4.0 Hz, CH(*C*$H_3)_2$), 23.9 (d, 2C, $^3J_{PC}$ = 5.1 Hz, CH(*C*$H_3)_2$), 23.4 (d (partially obscured by doublet at 24.1 ppm), 1C, $^1J_{PC}$ = 141.9 Hz, CH_2) ppm; **^{19}F-NMR** (375 MHz, $CDCl_3$): δ = -134.0– -134.1 (m, 2F, F3 + F5), -139.6– -139.7 (m, 2F, F2 + F6) ppm; **^{31}P{^{1}H}-NMR** (202 MHz, $CDCl_3$): δ = 19.0 (s) ppm; **IR** (ATR) $\tilde{\nu}$ = 1256 (P=O), 1105 (P–O^{i}Pr) cm^{-1}; **MS (ESI)** *m/z* (%): 428.91 (100) $[M + Na^+]^+$; **Elemental analysis** in % (calculated) $C_{13}H_{16}BrF_4O_3P$ (407.14 g/mol): C 38.06 (38.35), H 4.05 (3.96).

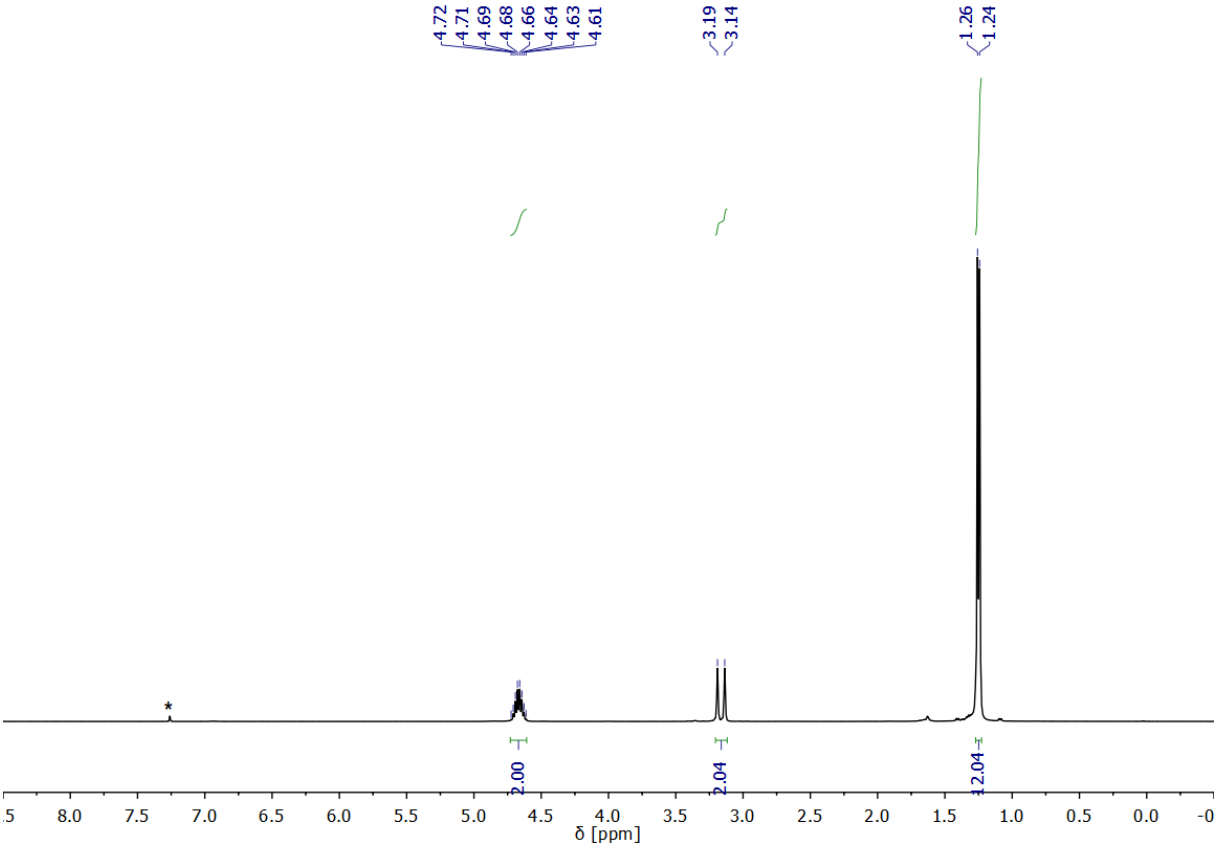

Figure 20: ^{1}H-NMR spectrum of **L6** in $CDCl_3$ (*).

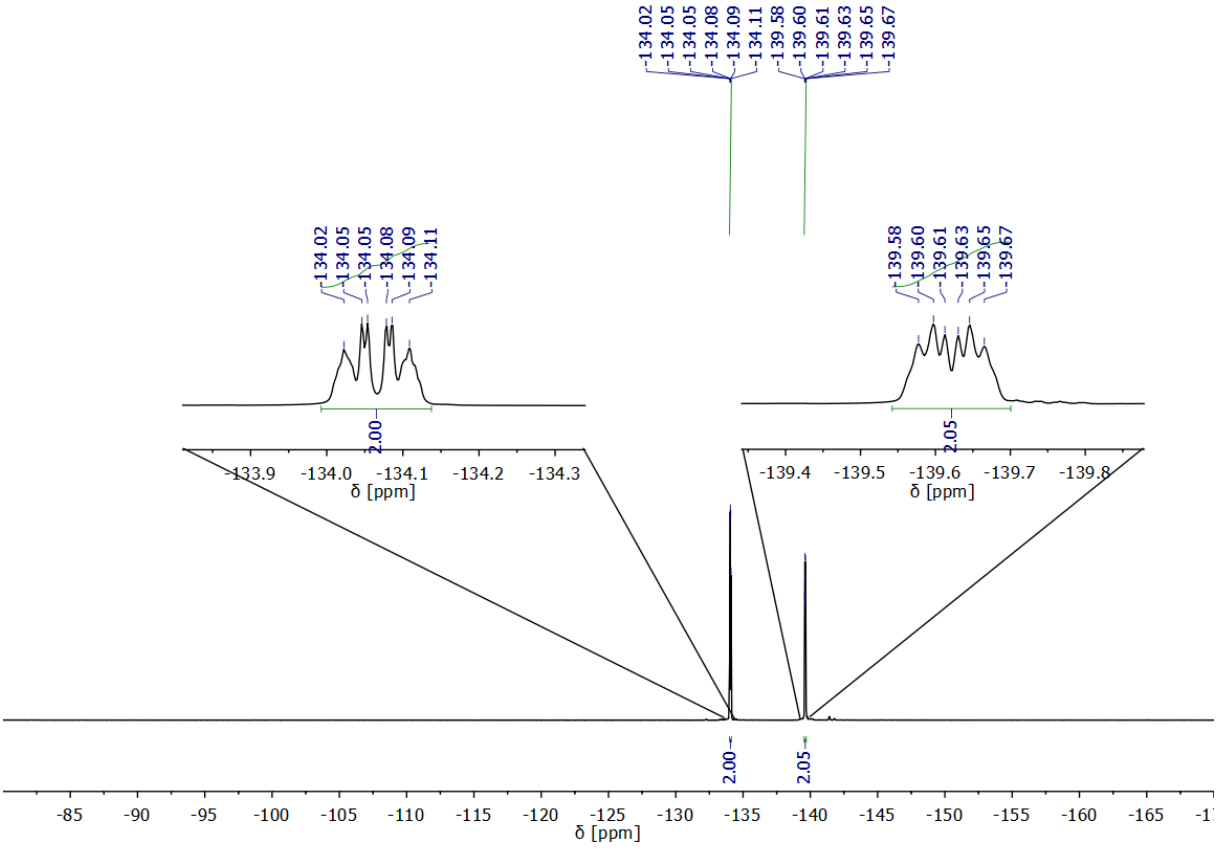

Figure 21: ^{19}F-NMR spectrum of **L6** in $CDCl_3$.

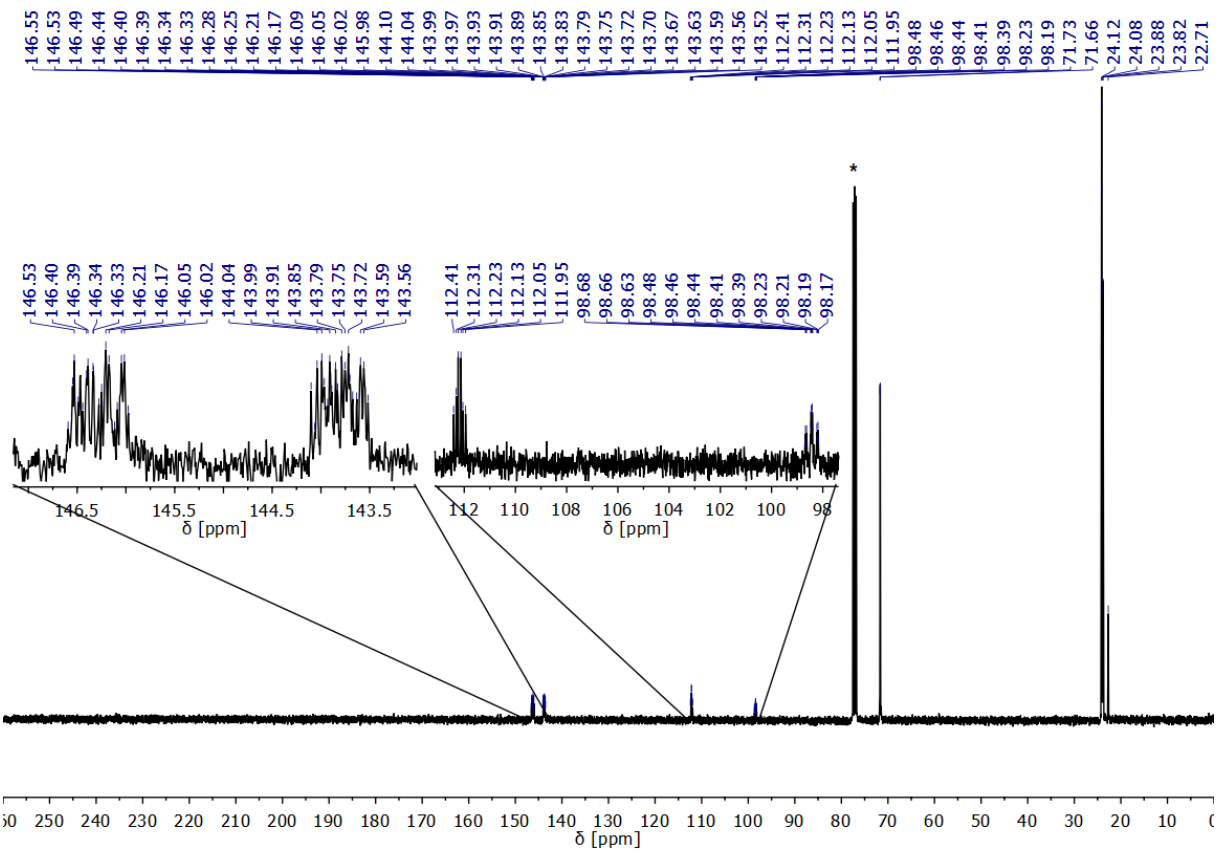

Figure 22: ^{13}C-NMR spectrum of **L6** in $CDCl_3$ (*).

1.2 Lanthanide Complexes [1-32] of L1-L6 & NMR Spectra

The corresponding lanthanide salt [$LnX_3(H_2O)_6$] (1.00 mmol, 1.00 eq.) (Ln = La, Nd, Dy, Er; X = NO_3, Cl) was dissolved in EtOH (15 mL). The respective phosphonate ligand (4.00 mmol, 4.00 eq., X = Cl, **L1**, **L2**, **L4**, **L5**, **L6**; 3.00 mmol, 3.00 eq., X = NO_3; **L1**, **L2**) was dissolved in EtOH (10 mL) (Exception: Ligand **L3** (3.00 mmol, 3.00 eq., X = Cl) was dissolved in EtOH (50 mL)). The lanthanide salt solution was added to the ligand solution and the mixture was stirred at r.t. overnight. In case of phosphonate **L3**, the yellow reaction mixtures were filtered prior to EtOH removal and were used without further treatment. For the other complexes, the solvent was evaporated, the obtained residue was dissolved in DCM and the DCM was again evaporated to remove residual EtOH. In case of X = Cl, the excess phosphonates were removed by thoroughly extraction with pentanes. **NOTE**: In case of X = Cl, 4.00 eq. of phosphonates **L1**, **L2**, **L4**, **L5**, **L6** are needed to successfully replace all water ligands in the lanthanide salts.

1.2.1 *L1, X = NO_3*

1: Yield: quantitative. **^{1}H-NMR** (400 MHz, CD_2Cl_2): δ = 7.32–7.22 (m, 15H, H2–H6), 4.52–4.61 (m, 6H, C*H*(CH_3)$_2$), 3.08 (d, 6H, $^2J_{PH}$ = 21.6 Hz, CH_2), 1.27 (d, 18H, $^3J_{HH}$ = 6.2 Hz, CH(C*H*$_3$)$_2$), 1.17 (d, 18H, $^3J_{HH}$ = 6.2 Hz, CH(C*H*$_3$)$_2$) ppm; **^{13}C{^{1}H}-NMR** (100 MHz, CD_2Cl_2): δ = 130.9 (d, 3C, $^2J_{PC}$ = 10.1 Hz, C1), 130.0 (d, 6C, $^3J_{PC}$ = 6.8 Hz, C2 + C6) 128.6 (d, 6C, $^4J_{PC}$ = 3.2 Hz, C3 + C5), 127.2 (d, 3C, $^5J_{PC}$ = 3.7 Hz, C4), 73.4 (d, 6C, $^2J_{PC}$ = 7.6 Hz, *C*H(CH_3)$_2$), 34.1 (d, 3C, $^1J_{PC}$ = 140.4 Hz, CH_2), 23.9 (m, 12C, CH(*C*H$_3$)$_2$) ppm; **^{31}P{^{1}H}-NMR** (202 MHz, CD_2Cl_2): δ = 23.3 (s) ppm; **IR** (ATR) $\tilde{\nu}$ = 1164 (P=O), 1098 (P–O^{i}Pr) cm^{-1}; **MS (MALDI)** *m/z*: 1031.3 [$La(NO_3)_2(L1)_3]^+$, 775.1 [$La(NO_3)_2(L1)_2]^+$; **Elemental analysis** in % (calculated) $C_{39}H_{63}LaN_3O_{18}P_3$ (1093.75 g/mol): C 65.67 (42.83), H 5.81 (5.81), N 3.78 (3.84).

2: Yield: quantitative. **IR** (ATR) $\tilde{\nu}$ = 1165 (P=O), 1099 (P–O^{i}Pr) cm^{-1}; **MS (MALDI)** *m/z*: 778.1 [$Nd(NO_3)_2(L1)_2]^+$, 523.3 [$Nd(NO_3)_2(L1)]^+$; **Elemental analysis** in % (calculated) $C_{39}H_{63}N_3NdO_{18}P_3$ (1099.09 g/mol): C 42.67 (42.62), H 5.73 (5.78), N 3.86 (3.82).

3: Yield: quantitative. **IR** (ATR) $\tilde{\nu}$ = 1169 (P=O), 1099 (P–O^{i}Pr) cm^{-1}; **MS (MALDI)** *m/z*: 1056.3 [$Dy(NO_3)_2(L1)_3]^+$, 798.2 [$Dy(NO_3)_2(L1)_2]^+$; **Elemental analysis** in % (calculated) $C_{39}H_{63}N_3DyO_{18}P_3$ (1117.35 g/mol): C 41.97 (41.92), H 5.66 (5.68), N 3.73 (3.76).

4: Yield: quantitative. **IR** (ATR) $\tilde{\nu}$ = 1170 (P=O), 1099 (P–O^{i}Pr) cm^{-1}; **MS (MALDI)** *m/z*: 1060.4 [$Er(NO_3)_2(L1)_3]^+$, 802.3 [$Er(NO_3)_2(L1)_2]^+$; **Elemental analysis** in % (calculated) $C_{39}H_{63}N_3ErO_{18}P_3$ (1122.11 g/mol): C 40.51 (41.74), H 5.86 (5.66), N 3.87 (3.74).

1.2.2 *L1, X = Cl*

5: Yield: quantitative. **^{1}H-NMR** (500 MHz, acetone-d_6): δ = 7.44–7.41 (m, 6H, H3 + H5), 7.33–7.28 (m, 6H, H2 + H6), 7.26–7.21 (m, 3H, H4), 5.00–4.89 (m, 6H, C*H*(CH_3)$_2$), 3.48 (d, 6H, $^2J_{PH}$ = 22.5 Hz, CH_2), 1.27 (d, 18H, $^3J_{HH}$ = 6.2 Hz, CH(C*H*$_3$)$_2$), 1.23 (d, 18H, $^3J_{HH}$ = 6.2 Hz, CH(C*H*$_3$)$_2$) ppm; **^{13}C{^{1}H}-NMR** (100 MHz, acetone-d_6): δ = 133.1 (d, 3C, $^2J_{PC}$ = 9.9 Hz, C1), 131.2 (d, 6C, $^3J_{PC}$ = 6.9 Hz, C2 + C6), 129.3 (d, 6C, $^4J_{PC}$ = 2.9 Hz, C3 + C5), 127.6 (d, 3C, $^5J_{PC}$ = 3.4 Hz, C4), 72.9 (d, 6C, $^2J_{PC}$ = 7.0 Hz, *C*H(CH_3)$_2$), 34.96 (d, 3C, $^1J_{PC}$ = 139.3 Hz, CH_2), 24.5 (d, 6C, $^3J_{PC}$ = 4.3 Hz, CH(C*H*$_3$)$_2$), 24.4 (d, 6C, $^3J_{PC}$ = 4.7 Hz, CH(*C*H$_3$)$_2$) ppm; **^{31}P{^{1}H}-NMR** (202 MHz, acetone-d_6): δ = 24.2 (s) ppm; **IR** (ATR) $\tilde{\nu}$ = 1165 (P=O), 1103 (P–O^{i}Pr)

cm^{-1}; **MS (MALDI)** *m/z*: 1467.0 $[LaCl_2(L1)_3 + 2\ LaCl_3]^+$, 1223.2 $[LaCl_2(L1)_3 + LaCl_3]^+$, 721.2 $[LaCl_2(L1)_2]^+$; **Elemental analysis** in % (calculated) $C_{39}H_{63}Cl_3LaO_9P_3$ (1014.10 g/mol): C 46.20 (46.19), H 6.14 (6.26).

6: Yield: quantitative. **IR** (ATR) $\tilde{\nu}$ = 1163 (P=O), 1101 (P–O^{i}Pr) cm^{-1}; **MS (MALDI)** *m/z*: 1484.4 $[NdCl_2(L1)_3 + 2\ NdCl_3]^+$, 726.3 $[NdCl_2(L1)_2]^+$; **Elemental analysis** in % (calculated) $C_{39}H_{63}Cl_3NdO_9P_3$ (1019.43 g/mol): C 46.10 (45.95), H 6.52 (6.23).

7: Yield: quantitative. **IR** (ATR) $\tilde{\nu}$ = 1160 (P=O), 1100 (P–O^{i}Pr) cm^{-1}; **MS (MALDI)** *m/z:* 1272.4 $[DyCl_2(L1)_3 + DyCl_3]^+$, 1002.5 $[DyCl_2(L1)_3]^+$, 746.3 $[DyCl_2(L1)_2]^+$; **Elemental analysis** in % (calculated) $C_{39}H_{63}Cl_3DyO_9P_3$ (251.33 g/mol): C 45.33 (45.14), H 6.28 (6.12).

8: Yield: quantitative. **IR** (ATR) $\tilde{\nu}$ = 1161 (P=O), 1100 (P–O^{i}Pr) cm^{-1}; **MS (MALDI)** *m/z*: 1006.6 $[ErCl_2(L1)_3]^+$, 750.4 $[ErCl_2(L1)_2]^+$; **Elemental analysis** in % (calculated) $C_{14}H_{21}NO_3$ (251.33 g/mol): C 45.12 (44.93), H 6.19 (6.09).

1.2.3 *L2, X = NO₃*

9: Yield: quantitative. **^{1}H-NMR** (400 MHz, $CDCl_3$): δ = 7.43 (d, 6H, $^3J_{HH}$ = 7.9 Hz, H3 + H5), 7.13 (d, 6H, $^3J_{HH}$ = 6.8 Hz, H2 + H6), 4.76–4.63 (m, 6H, C*H*$(CH_3)_2$), 3.18 (d, 6H, $^2J_{PH}$ = 21.9 Hz, CH_2), 1.26 (m, 36H, CH(C*H*$_3)_2$) ppm; **^{13}C{^{1}H}-NMR** (100 MHz, $CDCl_3$): δ = 131.8 (d, 6C, $^4J_{PC}$ = 3.1 Hz, C3 + C5), 131.7 (d, 6C, $^3J_{PC}$ = 6.8 Hz, C2 + C6), 130.0 (d, 3C, $^2J_{PC}$ = 10.3 Hz, C1), 121.3 (d, 3C, $^5J_{PC}$ = 4.9 Hz, C4), 73.6 (d, 6C, $^2J_{PC}$ = 7.7 Hz, *C*H$(CH_3)_2$), 33.6 (d, 3C, $^1J_{PC}$ = 140.8 Hz, CH_2), 24.0–23.9 (m, 12C, CH(*C*H$_3)_2$) ppm; **^{31}P{^{1}H}-NMR** (202 MHz, $CDCl_3$): δ = 22.3 (s) ppm; **IR** (ATR) $\tilde{\nu}$ = 1166 (P=O), 1096 (P–O^{i}Pr) cm^{-1}; **MS (MALDI)** *m/z*: 1269.0 $[La(NO_3)_2(L2)_3]^+$, 933.0 $[La(NO_3)_2(L2)_2]^+$; **Elemental analysis** in % (calculated) $C_{39}H_{60}Br_3LaN_3O_{18}P_3$ (1330.44 g/mol): C 35.26 (35.21), H 4.62 (4.55), N 3.08 (3.16).

10: Yield: quantitative. **IR** (ATR) $\tilde{\nu}$ = 1163 (P=O), 1095 (P–O^{i}Pr) cm^{-1}; **MS (MALDI)** *m/z*: 938.0 $[Nd(NO_3)_2(L2)_2]^+$; **Elemental analysis** in % (calculated) $C_{39}H_{60}Br_3NdN_3O_{18}P_3$ (1335.78 g/mol): C 35.32 (35.07), H 4.57 (4.53), N 3.19 (3.15).

11: Yield: quantitative. **IR** (ATR) $\tilde{\nu}$ = 1168 (P=O), 1095 (P–O^{i}Pr) cm^{-1}; **MS (MALDI)** *m/z*: 1292.0 $[Dy(NO_3)_2(L2)_3]^+$, 958.0 $[Dy(NO_3)_2(L2)_2]^+$; **Elemental analysis** in % (calculated) $C_{39}H_{60}Br_3DyN_3O_{18}P_3$ (1354.04 g/mol): C 34.57 (34.59), H 4.56 (4.47), N 3.14 (3.10).

12: Yield: quantitative. **IR** (ATR) $\tilde{\nu}$ = 1171 (P=O), 1097 (P–O^{i}Pr) cm^{-1}; **MS (MALDI)** *m/z*: 1296.2 $[Er(NO_3)_2(L2)_3]^+$, 960.2 $[Er(NO_3)_2(L2)_2]^+$; **Elemental analysis** in % (calculated) $C_{39}H_{60}Br_3ErN_3O_{18}P_3$ (1358.80 g/mol): C 34.70 (34.47), H 4.71 (4.45), N 3.04 (3.09).

1.2.4 *L2, X = Cl*

13: Yield: quantitative. **^{1}H-NMR** (400 MHz, $CDCl_3$): δ = 7.39 (d, 6H, $^3J_{HH}$ = 8.3 Hz, H3 + H5), 7.20 (dd, 6H, $^3J_{HH}$ = 8.4, $^4J_{HH}$ = 2.5 Hz, H2 + H6), 4.93–4.80 (m, 6H, C*H*$(CH_3)_2$), 3.31 (d, 6H, $^2J_{PH}$ = 22.3 Hz, CH_2), 1.26 (d, 18H, $^3J_{HH}$ = 6.2 Hz, CH(C*H*$_3)_2$), 1.22 (d, 18H, $^3J_{HH}$ = 6.2 Hz, CH(C*H*$_3)_2$) ppm; **^{13}C{^{1}H}-NMR** (100 MHz, $CDCl_3$): δ = 131.8 (d, 6C, $^3J_{PC}$ = 6.8 Hz, C2 + C6), 131.6 (d, 6C, $^4J_{PC}$ = 3.1 Hz, C3 + C5), 130.8 (d, 3C, $^2J_{PC}$ = 10.3 Hz, C1), 120.9 (d, 3C, $^5J_{PC}$ = 4.7 Hz, C4), 72.5 (d, 6C, $^2J_{PC}$ = 7.2 Hz, *C*H$(CH_3)_2$), 33.9 (d, 3C, $^1J_{PC}$ = 140.1 Hz, CH_2), 24.1 (d, 6C, $^3J_{PC}$ = 3.8 Hz, CH(*C*H$_3)_2$), 24.0 (d, 6C, $^3J_{PC}$ = 5.2 Hz, CH(*C*H$_3)_2$) ppm; **^{31}P{^{1}H}-NMR** (202 MHz, $CDCl_3$): δ = 23.2 (s) ppm; **IR** (ATR) $\tilde{\nu}$ = 1165 (P=O), 1097 (P–O^{i}Pr) cm^{-1};

MS (MALDI) *m/z*: 1705.3 $[LaCl_2(L2)_3 + 2\ LaCl_3]^+$, 1461.2 $[LaCl_2(L2)_3 + LaCl_3]^+$, 1215.3 $[LaCL_2(L2)_3]^+$, 879.2 $[LaCl_2(L2)_2]^+$; **Elemental analysis** in % (calculated) $C_{39}H_{60}Br_3LaO_9P_3$ (1250.79 g/mol): C 39.53 (37.45), H 4.89 (4.84).

14: Yield: quantitative. **IR** (ATR) $\tilde{\nu}$ = 1167 (P=O), 1097 (P–O^{i}Pr) cm^{-1}; **MS (MALDI)** *m/z*: 1722.3 $[NdCl_2(L2)_3 + 2\ NdCl_3]^+$, 1470.2 $[NdCl_2(L2)_3 + NdCl_3]^+$, 1218.3 $[NdCL_2(L2)_3]^+$, 884.2 $[NdCl_2(L2)_2]^+$; **Elemental analysis** in % (calculated) $C_{39}H_{60}Br_3NdO_9P_3$ (1256.12 g/mol): C 39.28 (37.29), H 4.81 (4.81).

15: Yield: quantitative. **IR** (ATR) $\tilde{\nu}$ = 1163 (P=O), 1096 (P–O^{i}Pr) cm^{-1}; **MS (MALDI)** *m/z*: 1507.3 $[DyCl_2(L2)_3 + DyCl_3]^+$, 1238.3 $[DyCL_2(L2)_3]^+$, 904.2 $[DyCl_2(L2)_2]^+$; **Elemental analysis** in % (calculated) $C_{39}H_{60}Br_3DyO_9P_3$ (1274.38 g/mol): C 38.83 (36.76), H 4.82 (4.75).

16: Yield: quantitative. **IR** (ATR) $\tilde{\nu}$ = 1164 (P=O), 1096 (P–O^{i}Pr) cm^{-1}; **MS (MALDI)** *m/z*: 1516.3 $[ErCl_2(L2)_3 + ErCl_3]^+$, 1244.3 $[ErCL_2(L2)_3]^+$, 908.3 $[ErCl_2(L2)_2]^+$; **Elemental analysis** in % (calculated) $C_{39}H_{60}Br_3ErO_9P_3$ (1279.14 g/mol): C 37.78 (36.62), H 4.73 (4.73).

1.2.5 *L3, X = Cl*

17: Yield: quantitative. **^{1}H-NMR** (500 MHz, $CDCl_3$): δ = 8.51 (d, 6H, $^3J_{HH}$ = 8.7 Hz, H4 + H8), 8.37 (d, 6H, $^3J_{HH}$ = 9.2 Hz, H1 + H5), 7.58–7.46 (m, 12H, H2 + H6, H3 + H7), 4.83–4.59 (m, 6H, C*H*$(CH_3)_2$), 4.36 (d, 6H, $^2J_{PH}$ = 22.8 Hz, CH_2), 1.16 (d, 18H, $^3J_{HH}$ = 6.1 Hz, CH(C*H*$_3)_2$), 0.98 (d, 18H, $^3J_{HH}$ = 6.1 Hz, CH(C*H*$_3)_2$) ppm; **^{13}C{^{1}H}-NMR** (100 MHz, C_6D_6): δ = 131.3 (d, 6C, $^4J_{PC}$ = 6.8 Hz, C13 + C14), 130.4 (d, 3C, $^5J_{PC}$ = 4.4 Hz, C10), 128.2 (d, 6C, $^6J_{PC}$ = 1.5 Hz, C3 + C7), 126.7 (d, 6C, $^5J_{PC}$ = 1.7 Hz, C4 + C8), 126.4 (d, 6C, $^4J_{PC}$ = 3.2 Hz, C1 + C5), 125.9 (d, 3C, $^2J_{PC}$ = 11.5 Hz, C9), 125.7 (d, 6C, $^5J_{PC}$ = 2.0 Hz, C2 + C6), 123.2 (d, 6C, $^3J_{PC}$ = 7.9 Hz, C11 + C12), 70.9 (d, 6C, $^2J_{PC}$ = 7.1 Hz, *C*H$(CH_3)_2$), 28.6 (d, 3C, $^1J_{PC}$ = 141.2 Hz, CH_2), 23.7 (d, 6C, $^3J_{PC}$ = 3.5 Hz, CH(*C*H$_3)_2$), 23.3 (d, 6C, $^3J_{PC}$ = 5.4 Hz, CH(*C*H$_3)_2$) ppm; **^{31}P{^{1}H}-NMR** (202 MHz, $CDCl_3$): δ = 23.0 ppm (s); **IR** (ATR) $\tilde{\nu}$ = 1160 (P=O), 1099 (P–O^{i}Pr) cm^{-1}; **Elemental analysis** in % (calculated) $C_{63}H_{72}Br_3Cl_3LaO_9P_3$ (1551.14 g/mol): C 48.84 (48.78), H 4.80 (4.68).

18: Yield: quantitative. **IR** (ATR) $\tilde{\nu}$ = 1154 (P=O), 1096 (P–O^{i}Pr) cm^{-1}; **Elemental analysis** in % (calculated) $C_{63}H_{72}Br_3Cl_3NdO_9P_3$ (1556.48 g/mol): C 48.31 (48.61), H 4.77 (4.66).

19: Yield: quantitative. **IR** (ATR) $\tilde{\nu}$ = 1153 (P=O), 1095 (P–O^{i}Pr) cm^{-1}; **Elemental analysis** in % (calculated) $C_{63}H_{72}Br_3Cl_3DyO_9P_3$ (1574.73 g/mol): C 48.31 (48.16), H 4.66 (4.61).

20: Yield: quantitative. **IR** (ATR) $\tilde{\nu}$ = 1154 (P=O), 1095 (P–O^{i}Pr) cm^{-1}; **Elemental analysis** in % (calculated) $C_{63}H_{72}Br_3Cl_3ErO_9P_3$ (1579.49 g/mol): C 47.55 (47.91), H 4.76 (4.59).

1.2.6 *L4, X = Cl*

21: Br ↔ Cl exchange (2:1) occurred between the ligand and the lanthanide salt. **^{1}H-NMR** (400 MHz, $CDCl_3$): δ = 7.37–7.27 (m, 12H, H2 + H6, H3 + H5; H2 + H6, H3 + H5), 4.99–4.86 (m, 6H, C*H*$(CH_3)_2$; C*H*$(CH_3)_2$), 4.53 (s, 2H, CH_2–Cl), 4.44 (s, 4H, CH_2–Br), 3.43 (d, 6H, $^2J_{PH}$ = 22.2 Hz, CH_2–P; CH_2–P), 1.28 (d, 18H, $^3J_{HH}$ = 6.1 Hz, CH(C*H*$_3)_2$; CH(C*H*$_3)_2$), 1.24 (d, 18H, $^3J_{HH}$ = 6.0 Hz, CH(C*H*$_3)_2$; CH(C*H*$_3)_2$) ppm; **^{13}C{^{1}H}-NMR** (100 MHz, $CDCl_3$): δ = 136.6 (d, 2C, $^5J_{PC}$ = 3.9 Hz, C4), 136.2 (d, 1C, $^5J_{PC}$ = 3.8 Hz, C4), 131.8 (d, 2C, $^2J_{PC}$ = 9.7 Hz, C1), 131.8 (d, 1C, $^2J_{PC}$ = 9.4 Hz, C1), 130.6–130.5 (m, 6C, C2 + C6; C2 + C6), 129.3 (d, 4C, $^4J_{PC}$ = 3.2 Hz, C3 + C5), 128.8 (d, 2C, $^4J_{PC}$ = 3.2 Hz, C3 + C5), 73.0 (s_{br}, 6C, *C*H$(CH_3)_2$, *C*H$(CH_3)_2$), 46.1 (s,

1C, CH_2–Cl), 34.2 (d, 3C, $^1J_{PC}$ = 139.7 Hz, CH_2–P; CH_2–P), 33.4 (s, 2C, CH_2–Br), 24.2 (d, 6C, $^3J_{PC}$ = 3.8 Hz, $CH(CH_3)_2$, $CH(CH_3)_2$), 24.1 (d, 6C, $^3J_{PC}$ = 5.3 Hz, $CH(CH_3)_2$, $CH(CH_3)_2$) ppm; **^{31}P{^{1}H}-NMR** (202 MHz, $CDCl_3$): δ = 23.73 (s, P), 23.67 (s,P) ppm; **IR** (ATR) $\tilde{\nu}$ = 1165 (P=O; P=O), 1098 (P–O^{i}Pr; P–O^{i}Pr) cm^{-1}; **MS (MALDI)** *m/z*: 1468.6 $[LaClBr(L4)_2(L4 - Br) + LaCl_3]^+$, 1424.7 $[LaCl_2(L4)_2(L4 - Br) + LaCl_3]^+$, 996.8 $[LaBr_2(L4)_2]^+$, 952.9 $[LaClBr(L4)_2]^+$; **Elemental analysis** in % (calculated) $C_{42}H_{66}Br_3Cl_3LaO_9P_3$ (1292.87 g/mol): C 39.16 (39.02), H 5.33 (5.15).

22: **IR** (ATR) $\tilde{\nu}$ = 1160 (P=O; P=O), 1096 (P–O^{i}Pr; P–O^{i}Pr) cm^{-1}; **MS (MALDI)** *m/z*: 1306.0 $[NdClBr(L4)_3]^+$, 1262.0 $[NdCl_2(L4)_3]^+$, 958.0 $[NdClBr(L4)_2]^+$, 912.0 $[NdCl_2(L4)_2]^+$; **Elemental analysis** in % (calculated) $C_{42}H_{66}Br_3Cl_3NdO_9P_3$ (1298.20 g/mol): C 39.68 (38.86), H 5.43 (5.12).

23: **IR** (ATR) $\tilde{\nu}$ = 1158 (P=O; P=O), 1096 (P–O^{i}Pr; P–O^{i}Pr) cm^{-1}; **MS (MALDI)** *m/z*: 1370.0 $[DyBr_2(L4)_3]^+$ 1326.0 $[DyClBr(L4)_3]^+$, 1280.1 $[DyCl_2(L4)_3]^+$, 1236.1 $[DyCl_2(L4)_2(L4)]^+$, 976.0 $[DyClBr(L4)_2]^+$, 932.0 $[DyCl_2(L4)_2]^+$; **Elemental analysis** in % (calculated) $C_{42}H_{66}Br_3Cl_3DyO_9P_3$ (1316.46 g/mol): C 38.44 (38.32), H 5.27 (5.05).

24: **IR** (ATR) $\tilde{\nu}$ = 1160 (P=O; P=O), 1096 (P–O^{i}Pr; P–O^{i}Pr) cm^{-1}; **MS (MALDI)** *m/z*: 1374.0 $[ErBr_2(L4)_3]^+$ 1330.1 $[ErClBr(L4)_3]^+$, 1284.1 $[ErCl_2(L4)_3]^+$, 1240.2 $[ErBr_2(L4)_3]^+$, 980.0 $[ErClBr(L4)_2]^+$, 935.0 $[ErCl_2(L4)_2]^+$; **Elemental analysis** in % (calculated) $C_{42}H_{66}Br_3Cl_3ErO_9P_3$ (1321.22 g/mol): C 39.68 (38.18), H 5.40 (5.03).

1.2.7 *L5, X = Cl*

25: **^{1}H-NMR** (400 MHz, $CDCl_3$): δ = 7.54–7.35 (m, 24H, H2 + H6; H2' + H6'; H3 + H5; H3' + H5'), 4.92–4.75 (m, 6H, C*H*$(CH_3)_2$), 3.33 (d, 6H, $^2J_{PH}$ = 21.8 Hz, CH_2), 1.29 (d, 18H, $^3J_{HH}$ = 6.0 Hz, $CH(CH_3)_2$), 1.24 (d, 18H, $^3J_{HH}$ = 6.0 Hz, $CH(CH_3)_2$) ppm; **^{13}C{^{1}H}-NMR** (100 MHz, $CDCl_3$): δ = 139.7–139.6 (m, 3C, C1'), 138.4 (d, 3C, $^5J_{PC}$ = 3.5 Hz, C1), 131.9 (s, 6C, C3' + C5'), 131.3 (d, 3C, $^2J_{PC}$ = 9.3 Hz, C4), 130.6 (d, 6C, $^3J_{PC}$ = 6.8 Hz, C3 + C5), 128.6 (s, 6C, C2' + C6'), 126.9 (d, 6C, $^4J_{PC}$ = 2.9 Hz, C2 + C6), 121.5 (s, 3C, C4'), 72.2–71.7 (m, 3C, *C*H$(CH_3)_2$), 34.3 (d, 3C, $^1J_{PC}$ = 139.9 Hz, CH_2), 24.2 (d, 6C, $^3J_{PC}$ = 3.7 Hz, $CH(CH_3)_2$), 24.0 (d, 6C, $^3J_{PC}$ = 5.2 Hz, $CH(CH_3)_2$) ppm; **^{31}P{^{1}H}-NMR** (202 MHz, $CDCl_3$): δ = 24.0 (s) ppm; **IR** (ATR) $\tilde{\nu}$ = 1142 (P=O), 1100 (P–O^{i}Pr) cm^{-1}; **MS (MALDI)** *m/z*: 1364.8 $[LaCl_2(L5)_3 - Br]^+$, 1030.9 $[LaCl_2(L5)_2]^+$; **Elemental analysis** in % (calculated) $C_{57}H_{72}Br_3Cl_3LaO_9P_3$ (1479.07 g/mol): C 46.62 (46.29), H 5.02 (4.91).

26: **IR** (ATR) $\tilde{\nu}$ 1142 (P=O), 1099 (P–O^{i}Pr) cm^{-1}; **MS (MALDI)** *m/z*: 1036.0 $[NdCl_2(L5)_2]^+$; **Elemental analysis** in % (calculated) $C_{57}H_{72}Br_3Cl_3NdO_9P_3$ (1484.41 g/mol): C 46.46 (46.12), H 5.61 (4.86).

27: **IR** (ATR) $\tilde{\nu}$ = 1143 (P=O), 1099 (P–O^{i}Pr) cm^{-1}; **MS (MALDI)** *m/z*: 1467.2 $[DyCl_2(L5)_3]^+$, 1056.0 $[DyCl_2(L5)_2]^+$; **Elemental analysis** in % (calculated) $C_{57}H_{72}Br_3Cl_3DyO_9P_3$ (1502.67 g/mol): C 46.65 (45.56), H 5.37 (4.83).

28: **IR** (ATR) $\tilde{\nu}$ = 1143 (P=O), 1099 (P–O^{i}Pr) cm^{-1}; **MS (MALDI)** *m/z*: 1470.1 $[ErCl_2(L5)_3]^+$, 1060.0 $[ErCl_2(L5)_2]^+$; **Elemental analysis** in % (calculated) $C_{57}H_{72}Br_3Cl_3ErO_9P_3$ (1507.43 g/mol): C 45.53 (45.42), H 5.12 (4.81).

1.2.8 *L6, X = Cl*

29: **^{1}H-NMR** (400 MHz, $CDCl_3$): δ = 4.98–4.85 (m, 6H, C*H*$(CH_3)_2$), 3.53 (d, 6H, $^{2}J_{PH}$ = 22.2 Hz, CH_2), 1.30 (d, 18H, $^{3}J_{HH}$ = 6.2 Hz, CH(C*H*$_3)_2$), 1.27 (d, 18H, $^{3}J_{HH}$ = 6.1 Hz, CH(C*H*$_3)_2$) ppm; **^{13}C{^{1}H}-NMR** (100 MHz, THF-d_8): δ = 147.8–146.9 (m, 2C, C3 + C5), 145.3–144.5 (m, 2C, C2 + C6), 114.0 (td, 1C, $^{2}J_{FC}$ = 18.5, $^{2}J_{PC}$ = 10.9 Hz, C1), 98.9 (td, 1C, $^{2}J_{FC}$ = 22.8, $^{5}J_{PC}$ = 5.4 Hz, C4), 73.1 (d, 2C, $^{2}J_{PC}$ = 6.8 Hz, *C*H$(CH_3)_2$), 24.4 (d, 2C, $^{3}J_{PC}$ = 3.7 Hz, CH(*C*H$_3)_2$), 24.2 (d, 2C, $^{3}J_{PC}$ = 5.3 Hz, CH(*C*H$_3)_2$), 23.9 (d, 1C, $^{1}J_{PC}$ = 143.1 Hz, CH_2) ppm; **^{19}F-NMR** (375 MHz, THF-d_8): δ = -134.5– -134.6 (m, 2F, F3 + F5), -138.3– -138.4 (m, 2F, F2 + F6) ppm; **^{31}P{^{1}H}-NMR** (202 MHz, THF-d_8): δ = 19.0 (s) ppm; **IR** (ATR) $\tilde{\nu}$ = 1145 (P=O), 1099 (P–O^{i}Pr) cm^{-1}; **MS (MALDI)** *m/z*: 1920.4 $[LaCl_2(L5)_3 + 2\ LaCl_3]^+$, 1674.6 $[LaCl_2(L5)_3 + LaCl_3]^+$; **Elemental analysis** in % (calculated) $C_{39}H_{48}Br_3Cl_3F_{12}LaO_9P_3$ (1466.67 g/mol): C 29.70 (31.94), H 3.30 (3.19).

For complexes **30**-**32** also species carrying **L6** with a Cl instead of a Br at the para position were detected in the MALDI spectra.

30: **IR** (ATR) $\tilde{\nu}$ = 1145 (P=O), 1099 (P–O^{i}Pr) cm^{-1}; **MS (MALDI)** *m/z*: 1236.0 $[NdCl_2(L6)(L6–Cl) + NdCl_3]^+$, 986.2 $[NdCl_2(L6)(L6–Cl)]^+$; **Elemental analysis** in % (calculated) $C_{39}H_{48}Br_3Cl_3F_{12}NdO_9P_3$ (1472.01 g/mol): C 31.95 (31.82), H 3.35 (3.29).

31: **IR** (ATR) $\tilde{\nu}$ = 1145 (P=O), 1099 (P–O^{i}Pr) cm^{-1}; **MS (MALDI)** *m/z*: 1274.1 $[DyCl_2(L6)(L6–Cl) + DyCl_3]^+$, 1006.2 $[DyCl_2(L6)(L6–Cl)]^+$; **Elemental analysis** in % (calculated) $C_{39}H_{48}Br_3Cl_3F_{12}DyO_9P_3$ (1490.27 g/mol): C 31.67 (31.43), H 3.55 (3.25).

32: **IR** (ATR) $\tilde{\nu}$ = 1144 (P=O), 1101 (P–O^{i}Pr) cm^{-1}; **MS (MALDI)** *m/z*: 1284.1 $[ErCl_2(L6)(L6–Cl) + ErCl_3]^+$, 1010.3 $[ErCl_2(L6)(L6–Cl)]^+$; **Elemental analysis** in % (calculated) $C_{39}H_{48}Br_3Cl_3F_{12}ErO_9P_3$ (1495.03 g/mol): C 32.17 (31.33), H3.72 (3.24).

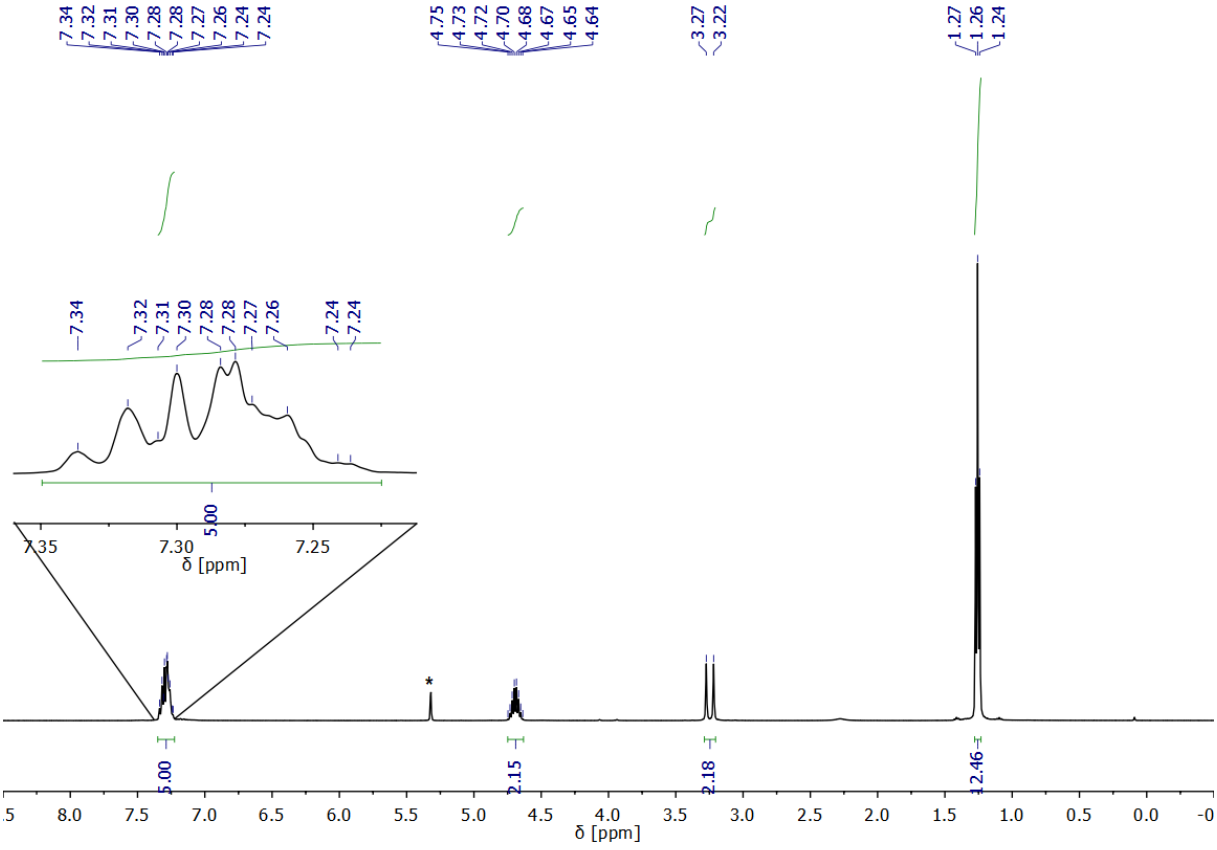

Figure 23: ^{1}H-NMR spectrum of **1** in CD_2Cl_2 (*).

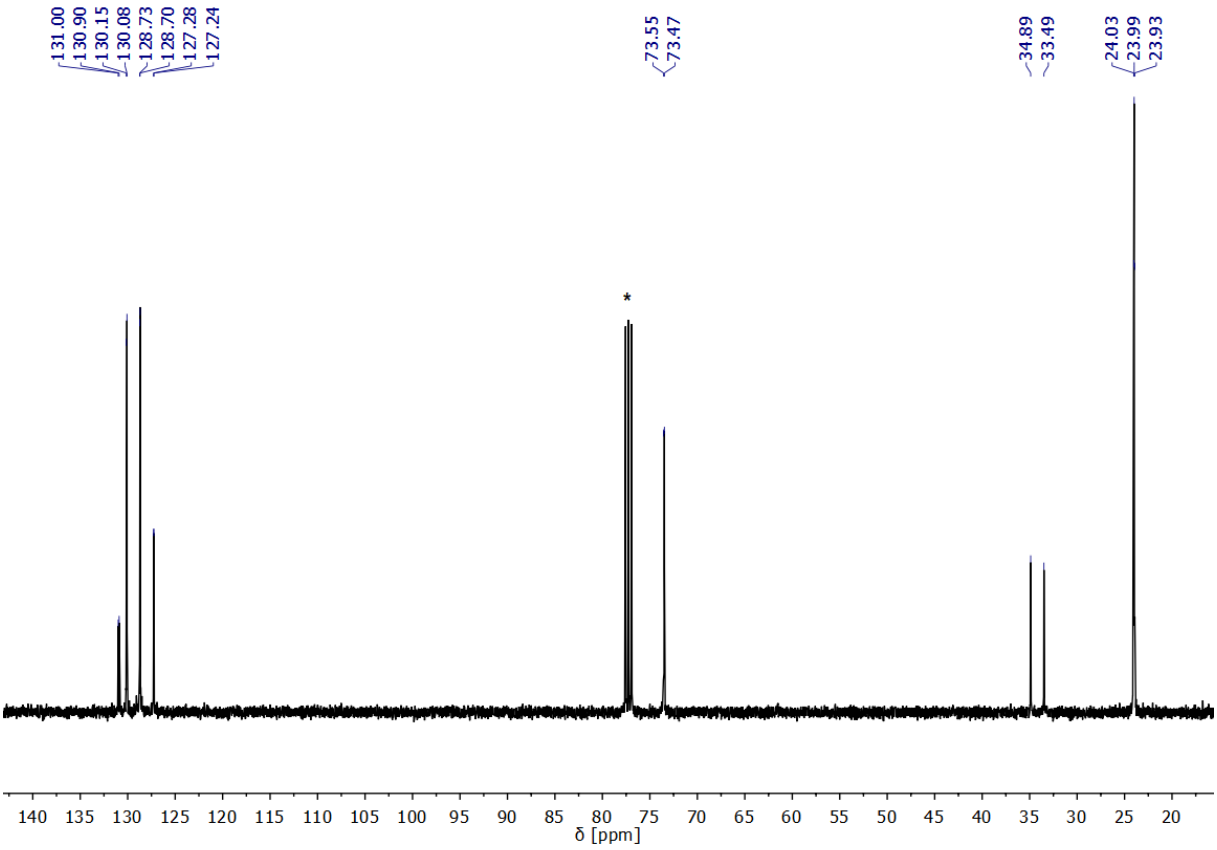

Figure 24: ^{13}C-NMR spectrum of **1** in $CDCl_3$ (*).

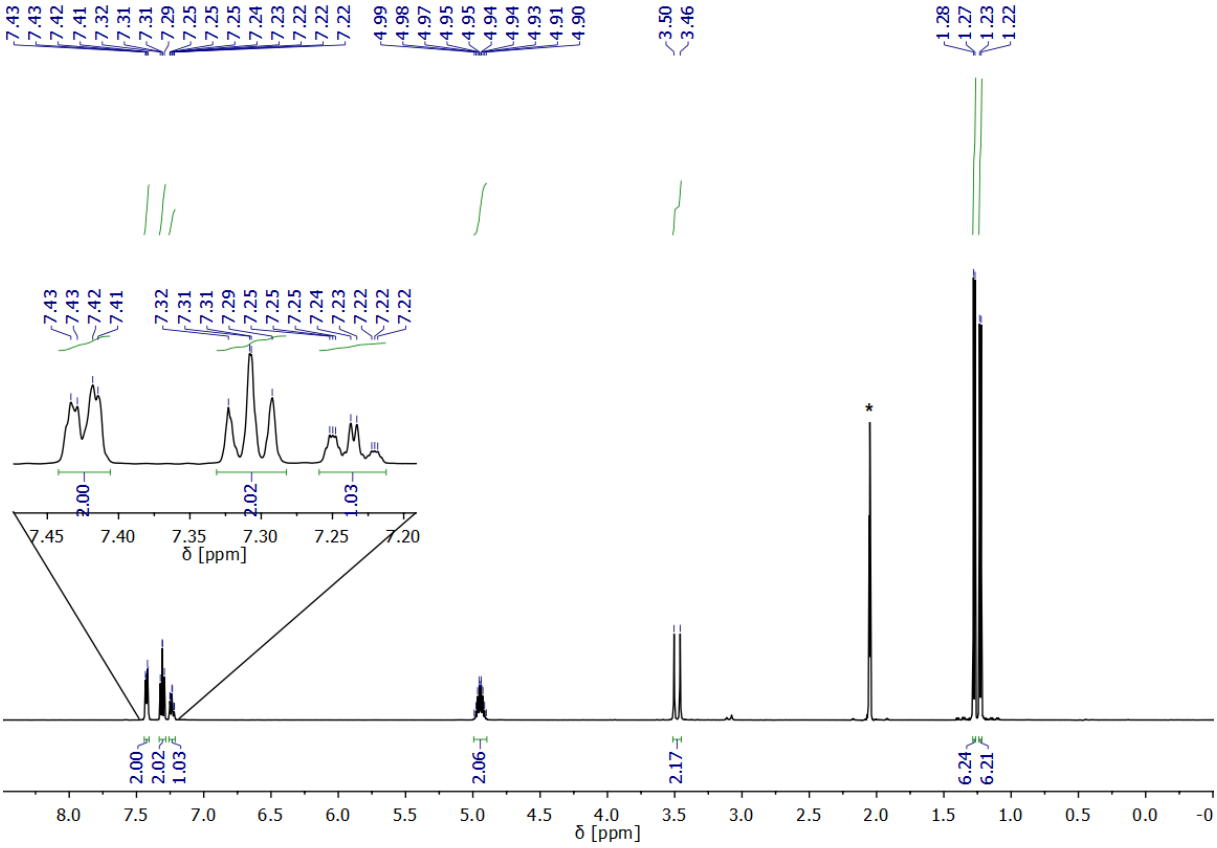

Figure 25: ^{1}H-NMR spectrum of **5** in acetone-d_6 (*).

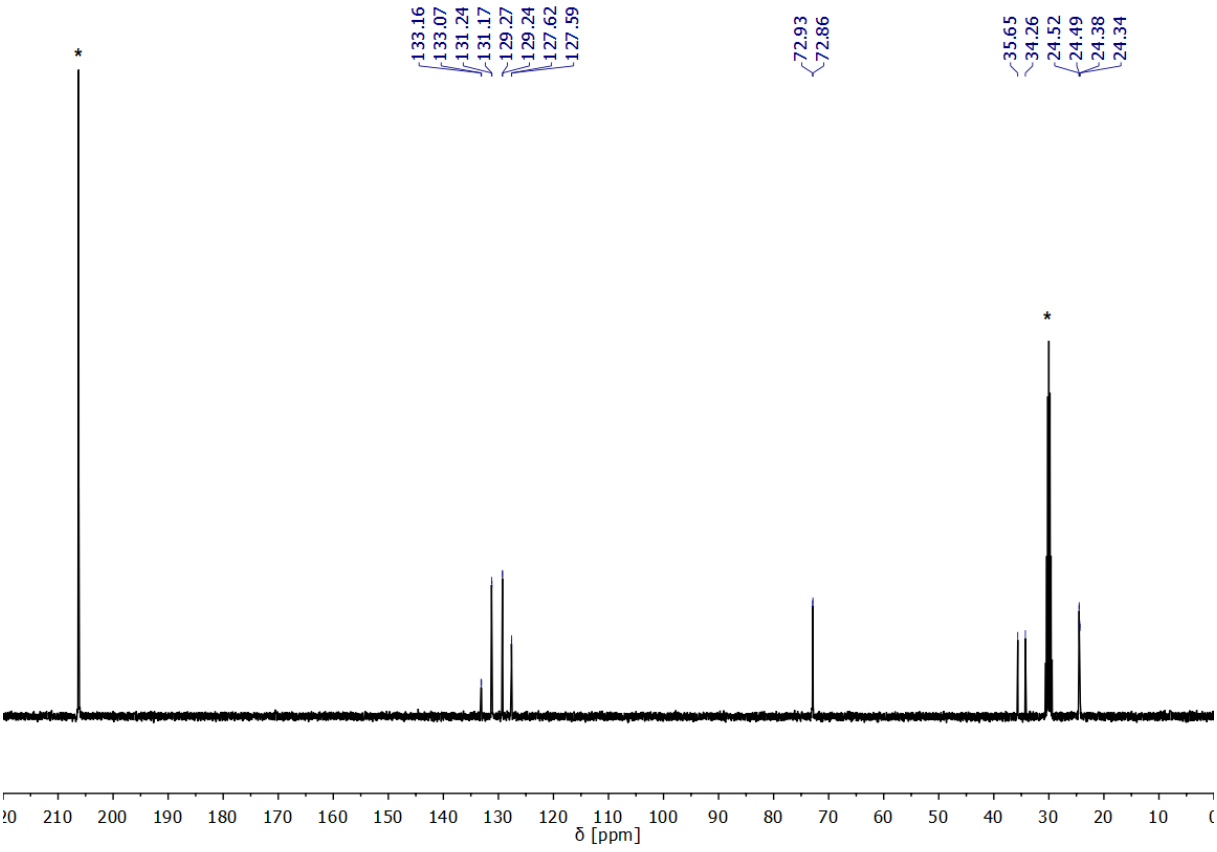

Figure 26: ^{13}C-NMR spectrum of **5** in acetone-d_6 (*).

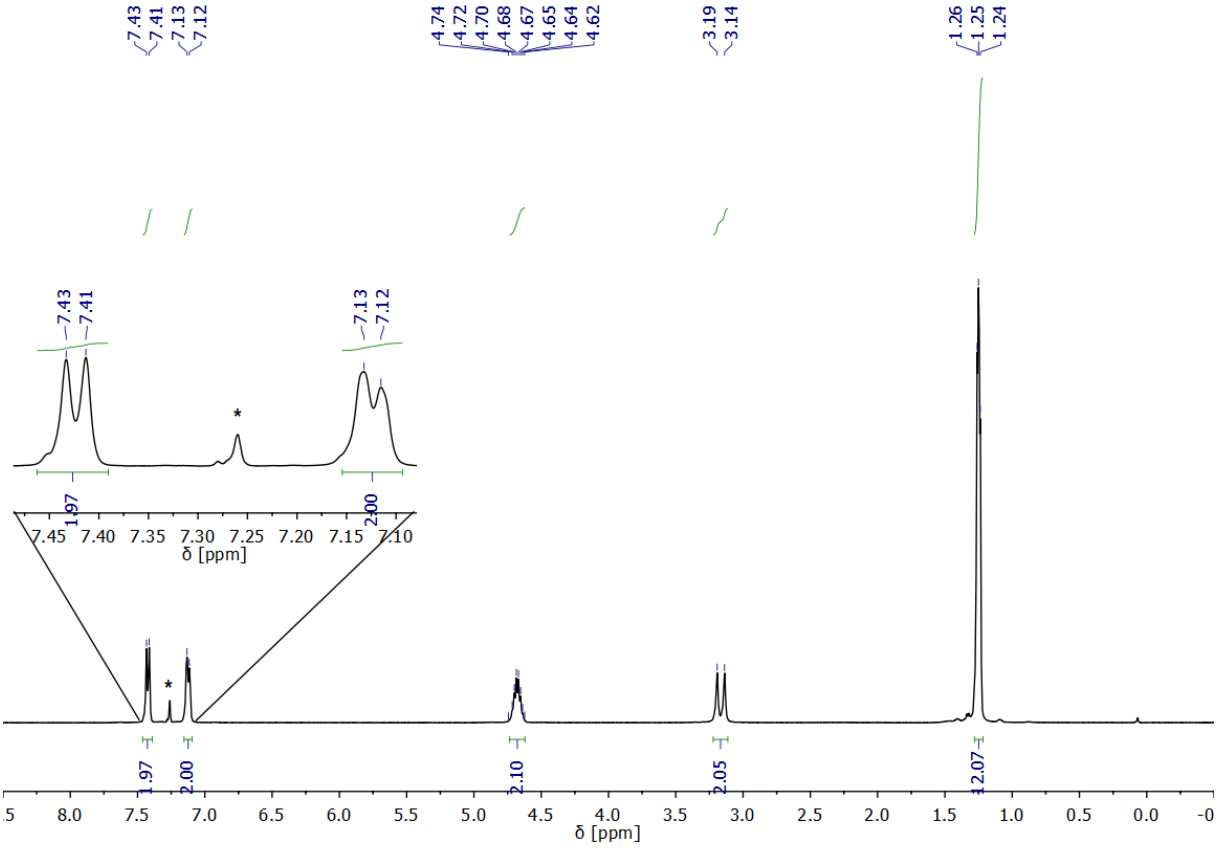

Figure 27: ^{1}H-NMR spectrum of **9** in $CDCl_3$ (*).

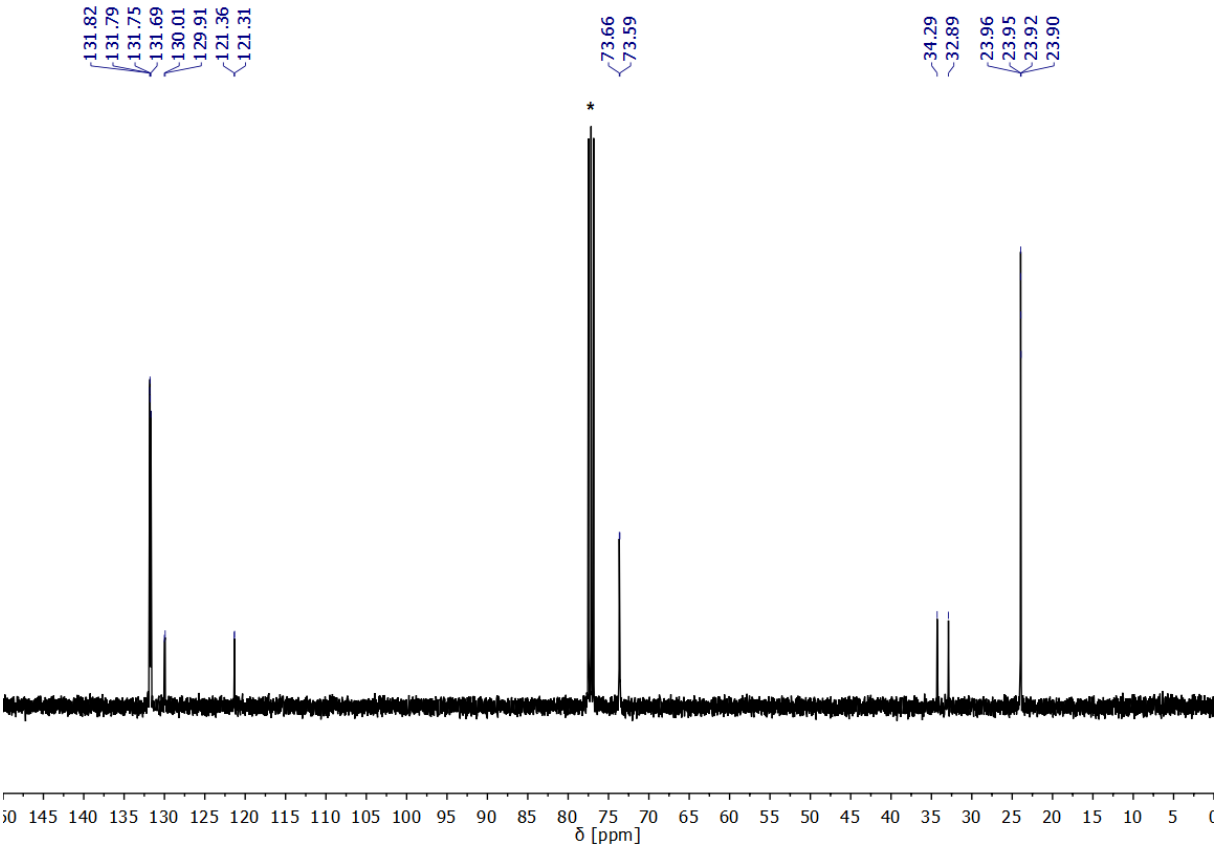

Figure 28: ^{13}C-NMR spectrum of **9** in $CDCl_3$ (*).

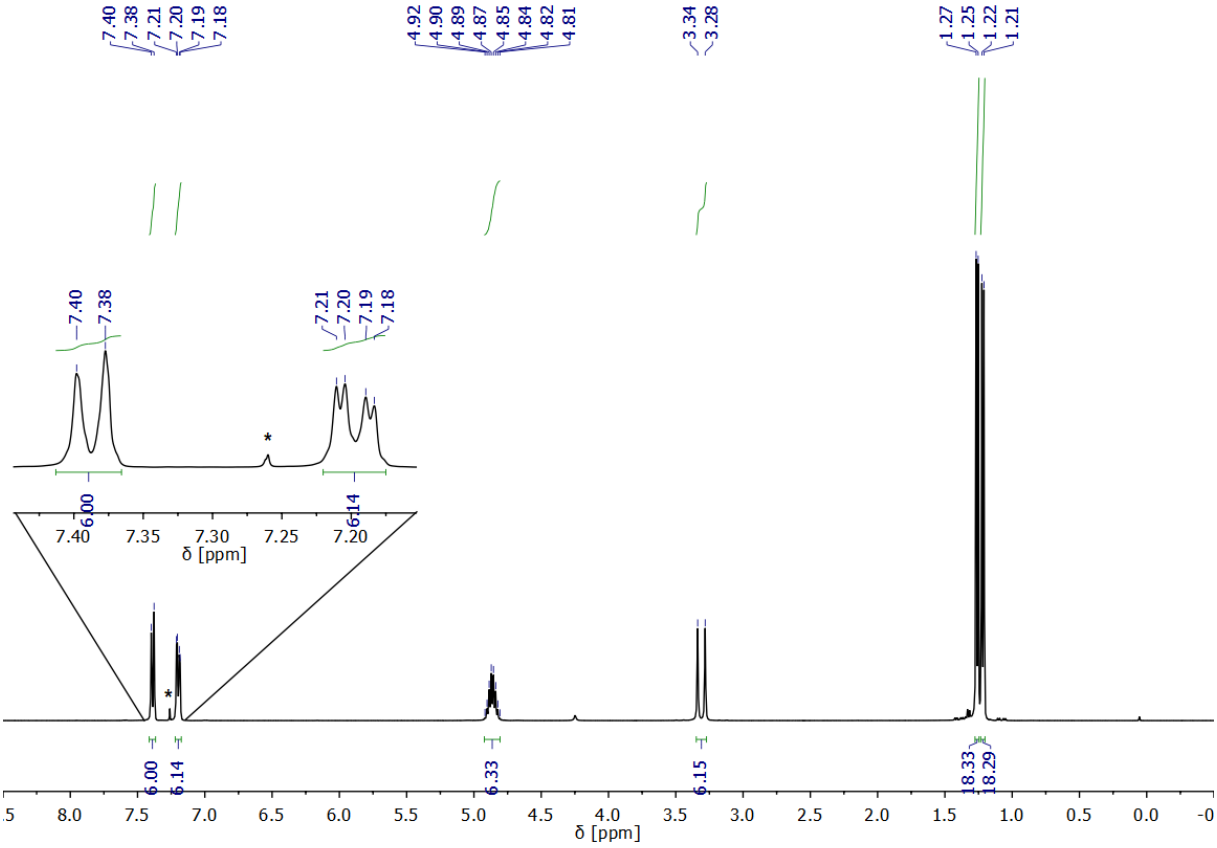

Figure 29: ^{1}H-NMR spectrum of **13** in $CDCl_3$ (*).

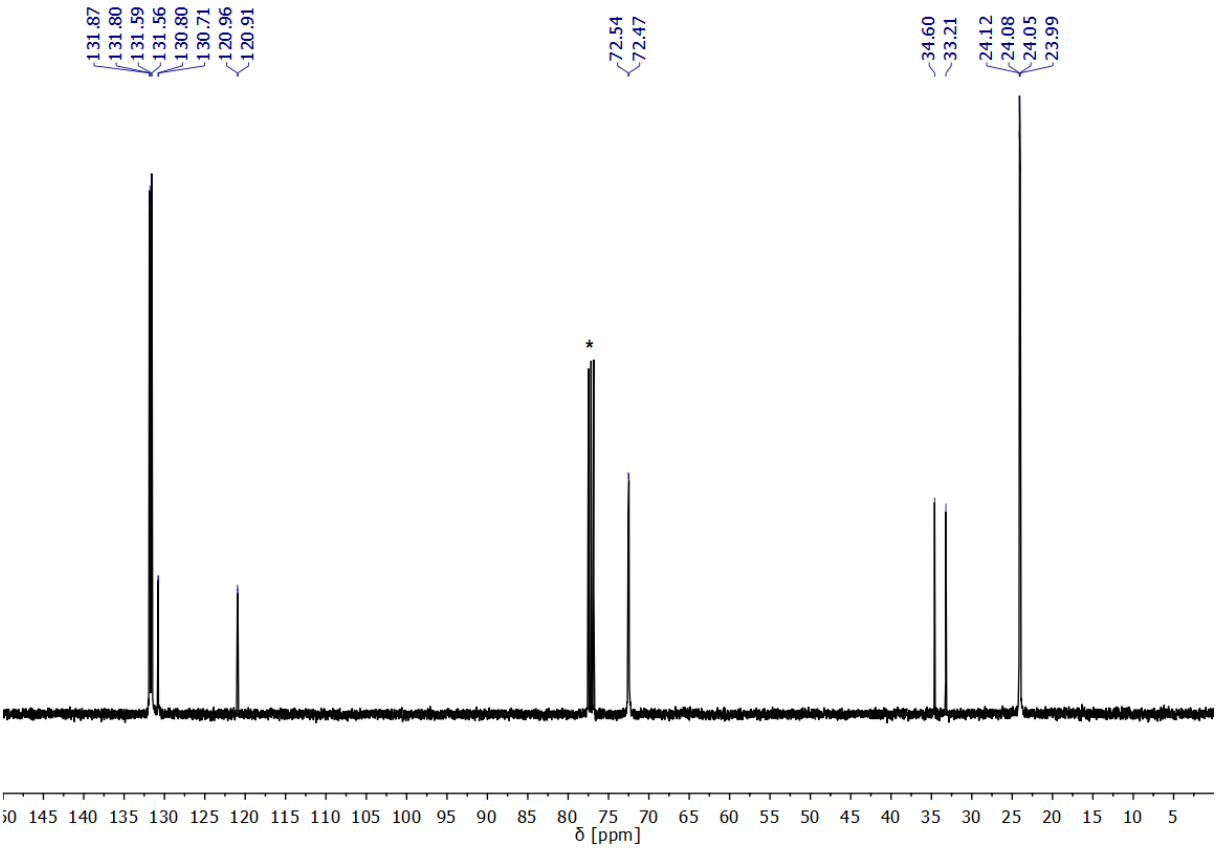

Figure 30: ^{13}C-NMR spectrum of **13** in $CDCl_3$ (*).

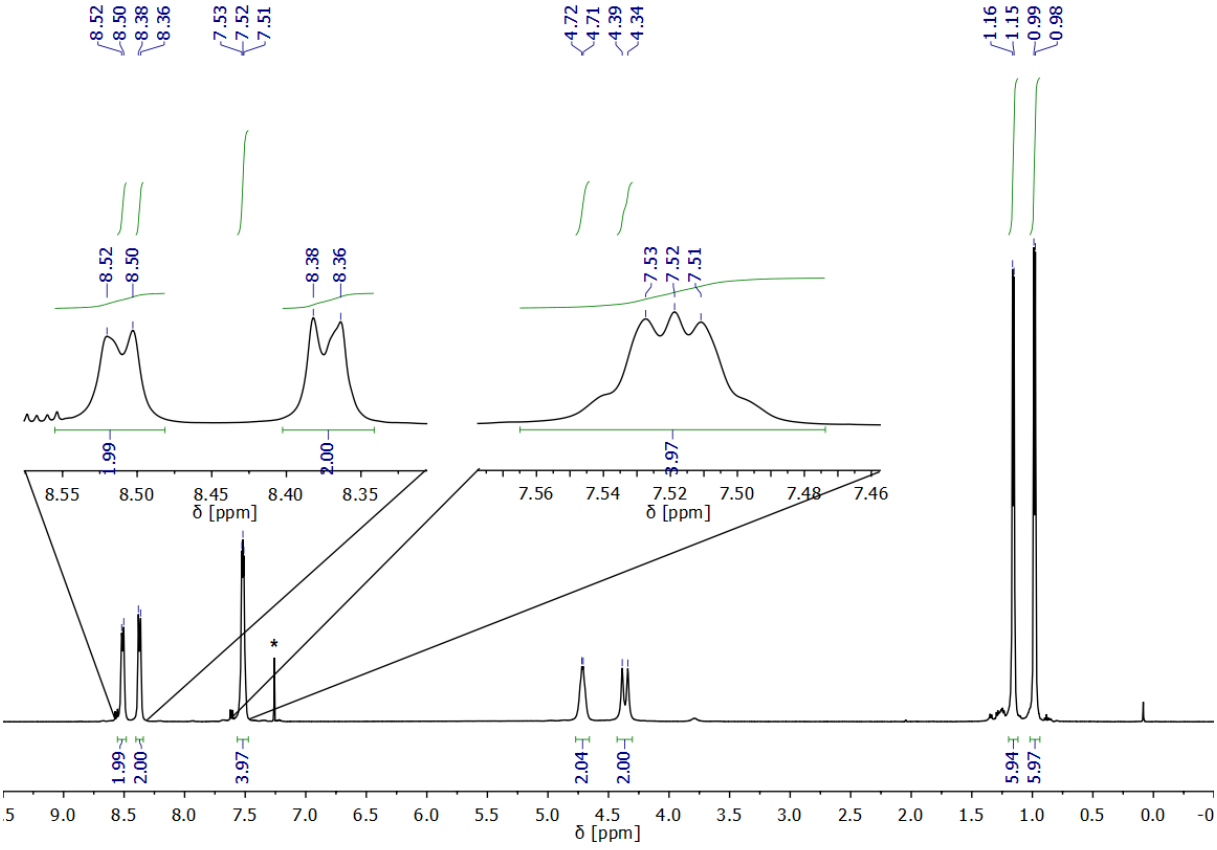

Figure 31: ^{1}H-NMR spectrum of **17** in $CDCl_3$ (*).

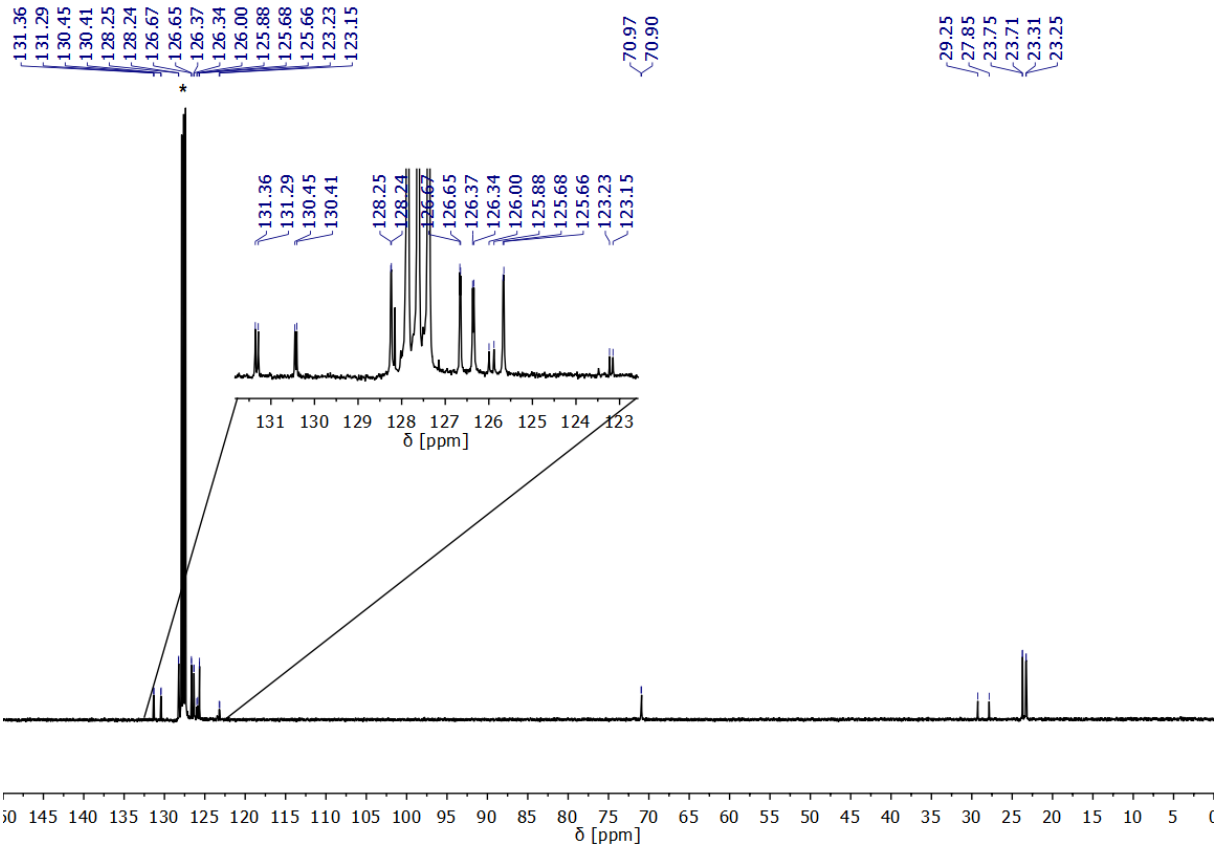

Figure 32: ^{13}C-NMR spectrum of **17** in C_6D_6 (*).

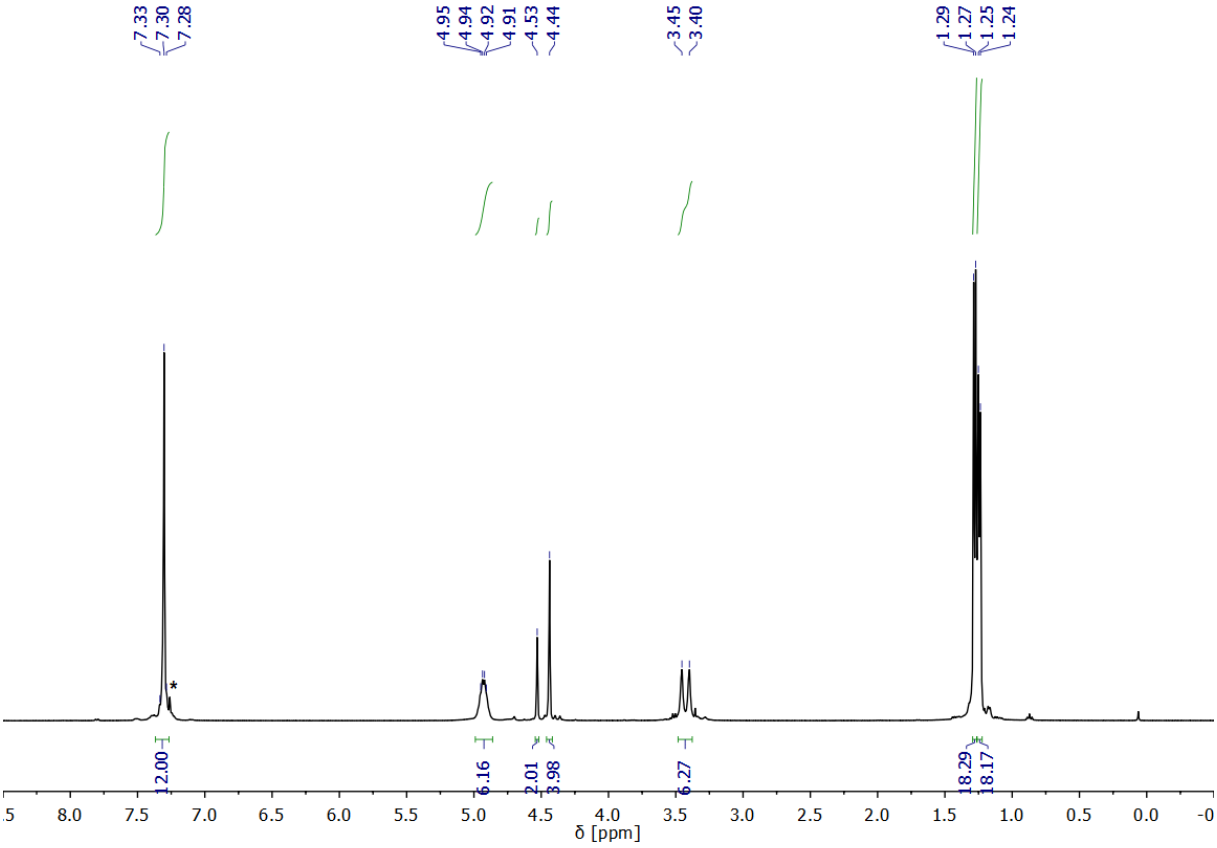

Figure 33: ^{1}H-NMR spectrum of **21** in $CDCl_3$ (*).

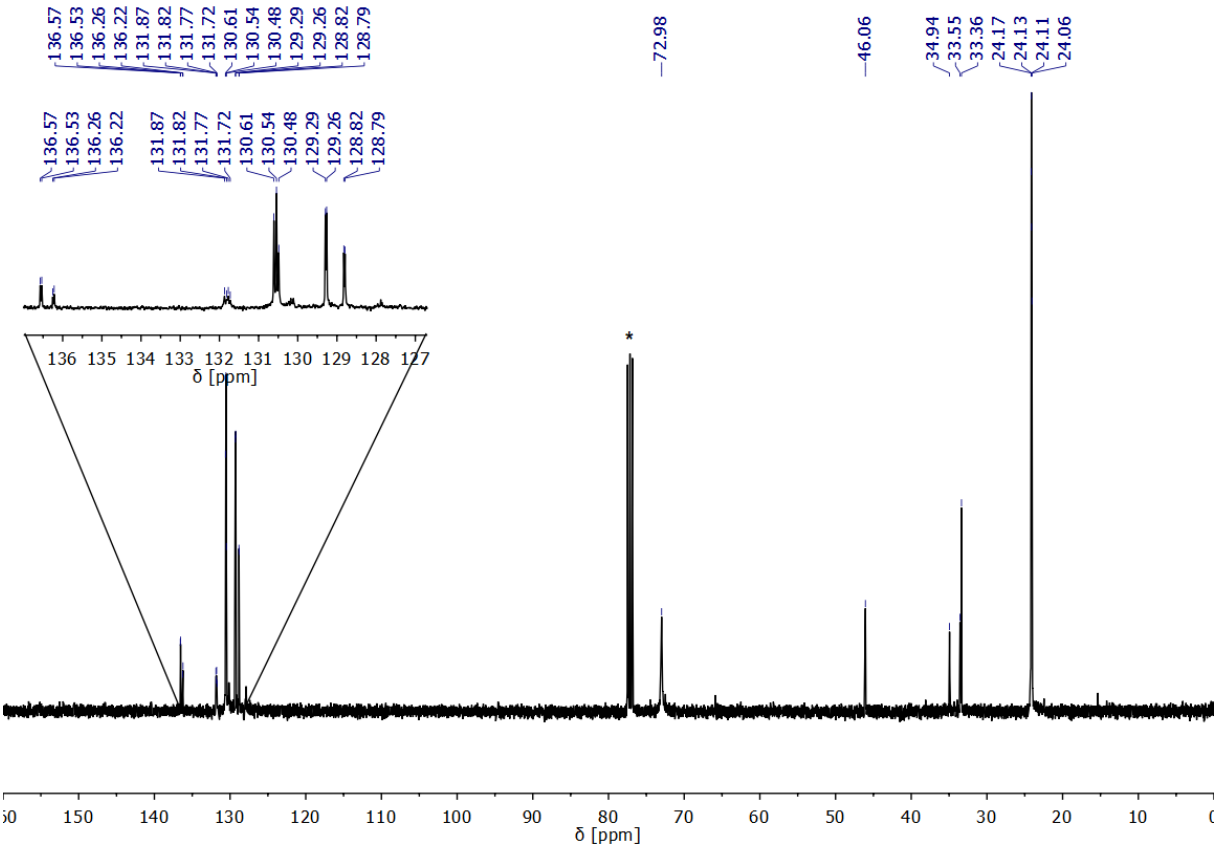

Figure 34: ^{13}C-NMR spectrum of **21** in $CDCl_3$ (*).

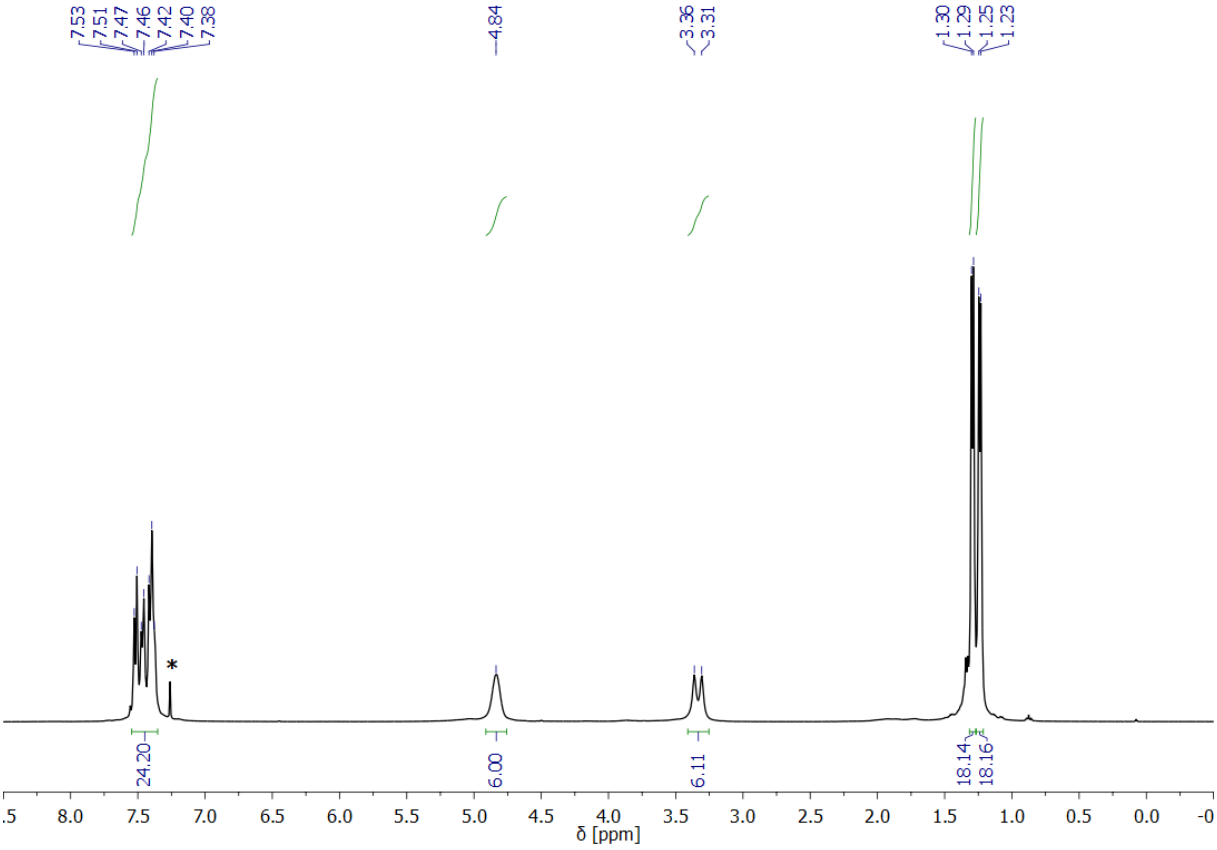

Figure 35: ^{1}H-NMR spectrum of **25** in $CDCl_3$ (*).

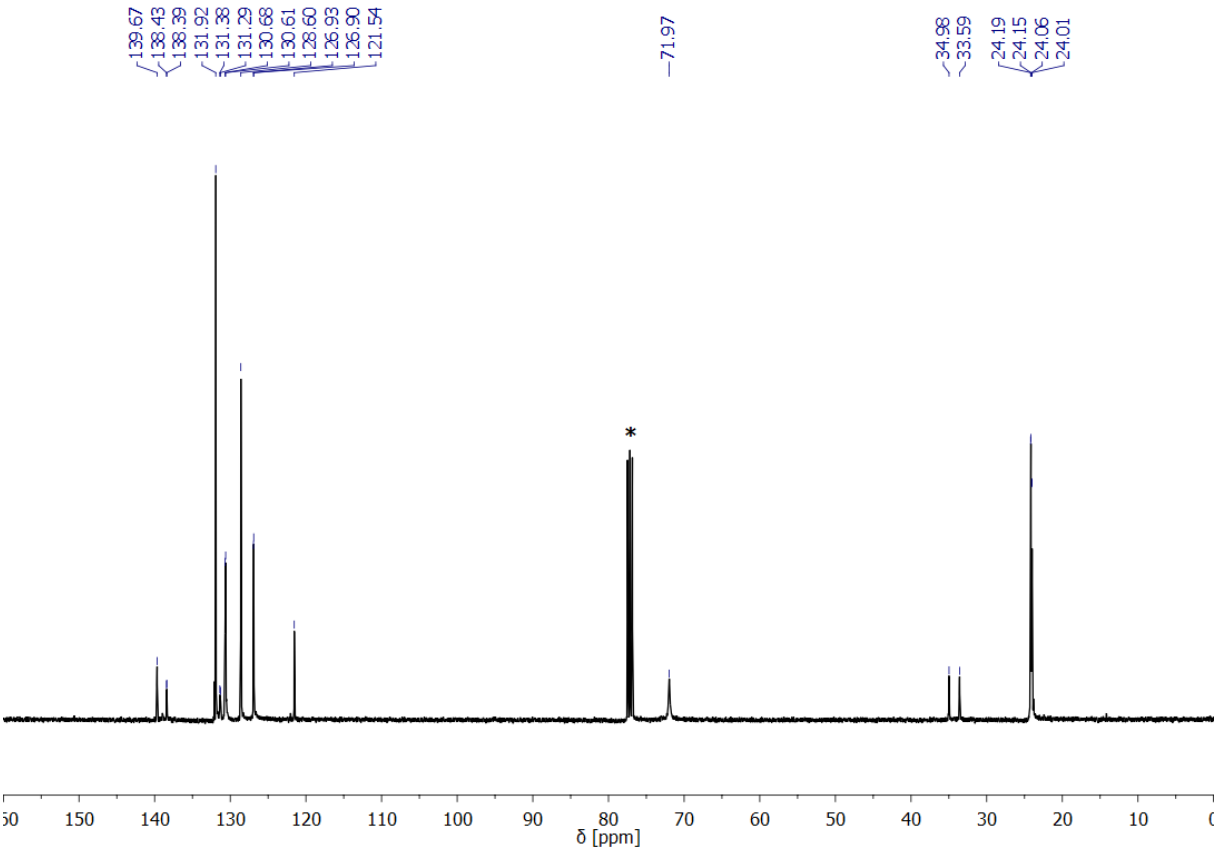

Figure 36: ^{13}C-NMR spectrum of **25** in $CDCl_3$ (*).

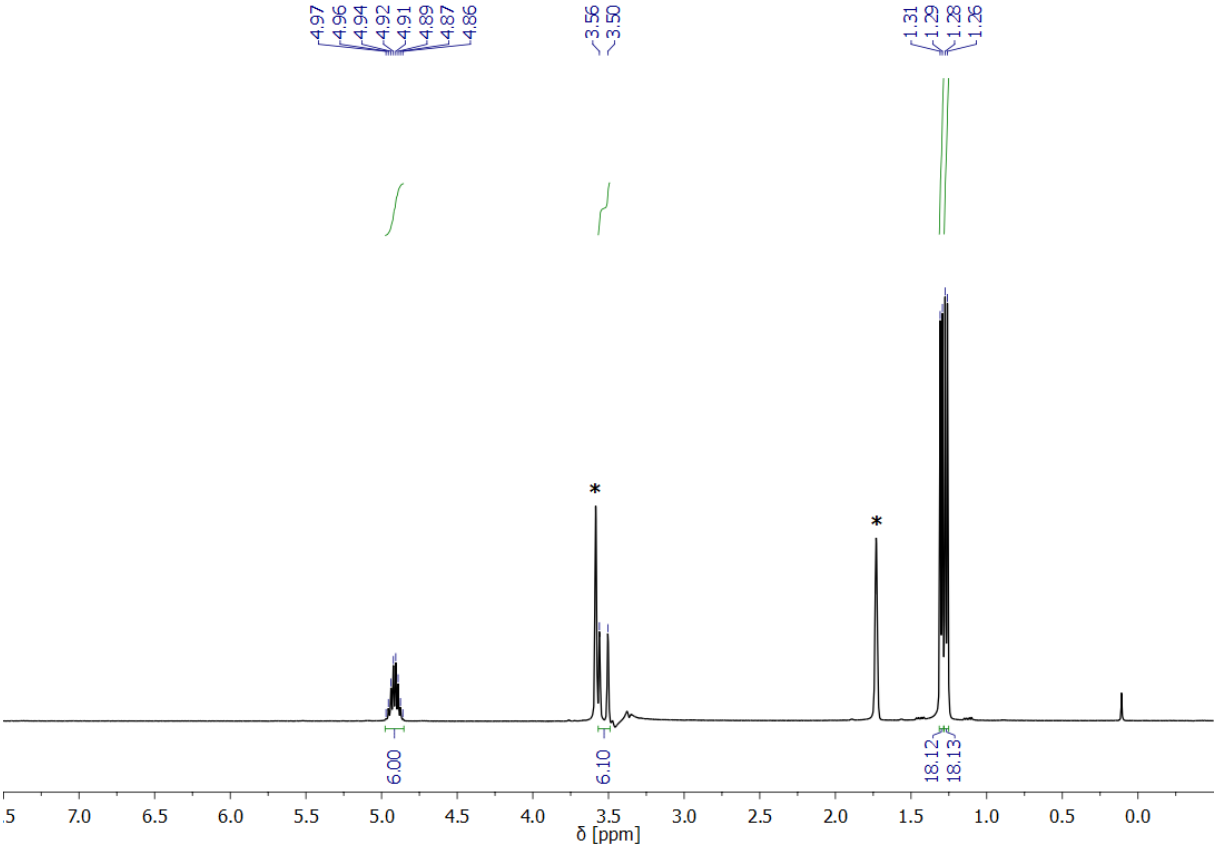

Figure 37: ^{1}H-NMR spectrum of **29** in THF-d_8 (*).

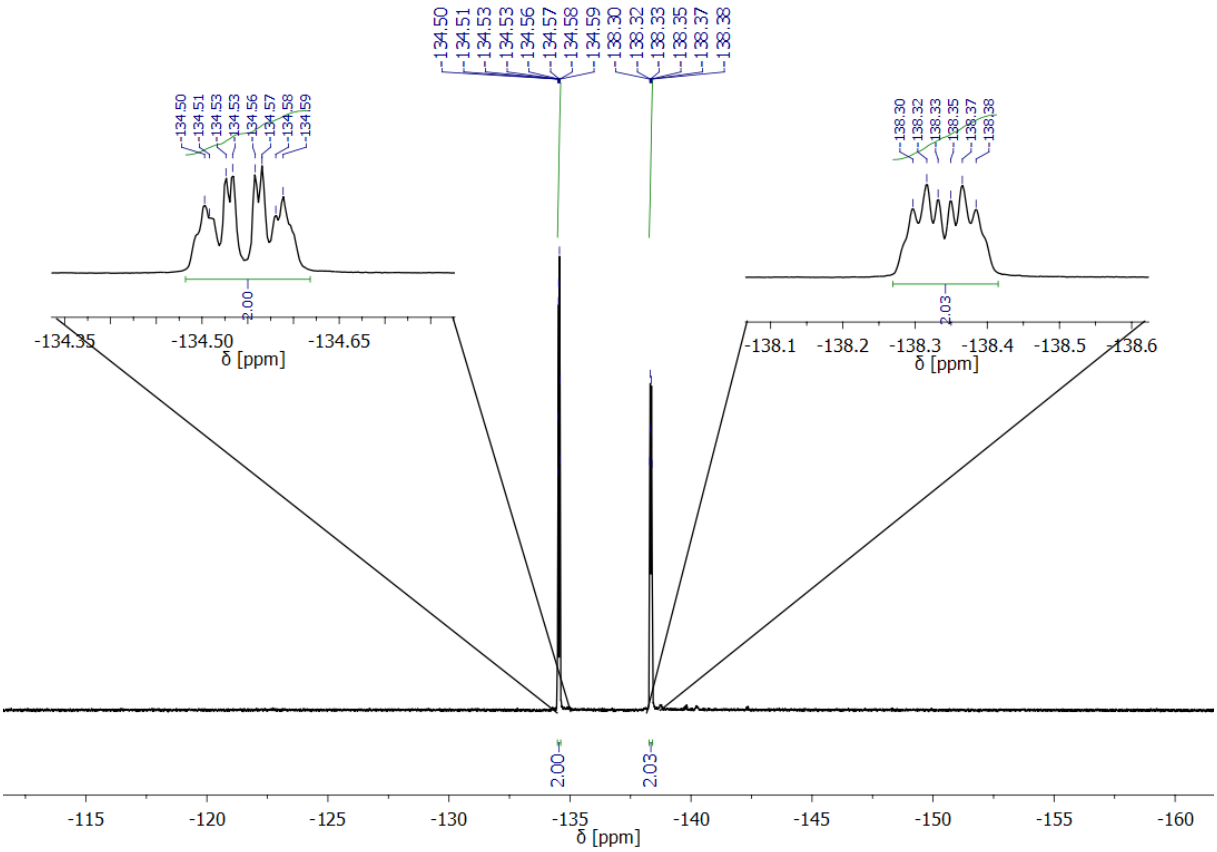

Figure 38: ^{19}F-NMR spectrum of **29** in THF-d_8.

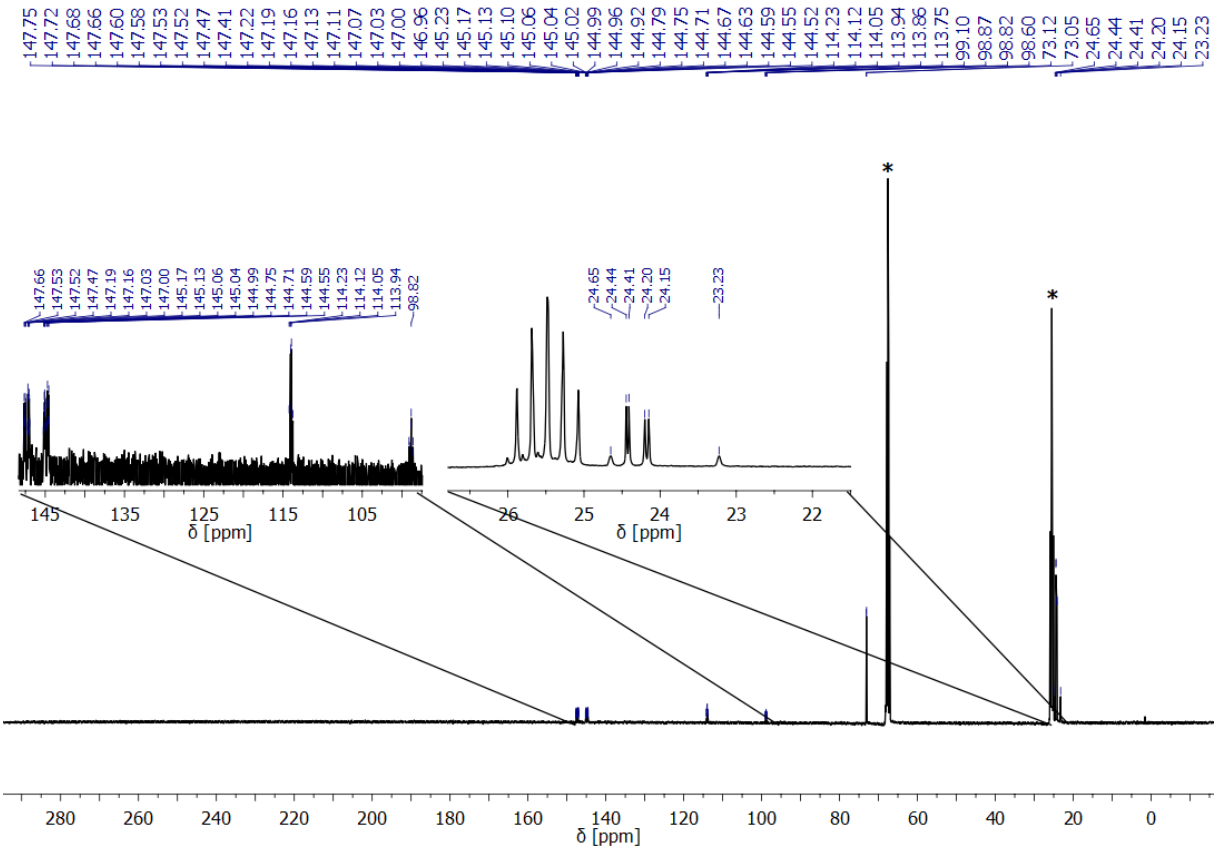

Figure 39: ^{13}C-NMR spectrum of **29** in THF-d_8 (*).

2 CRYSTALLOGRAPHIC DATA

Table 1: Crystallographic data for ligands **L3**, **L6** and complexes **2**, **3**, **5**, **10** and **13**.

Compound	**L3**	**L6**	**2**	**3**	**5**	**10**	**13**
CCDC number	2021050	2021051	2021052	2021053	2021054	2021055	2021056
formula	$C_{21}H_{24}BrO_3P$	$C_{13}H_{16}BrF_4O_3P$	$C_{39}H_{63}N_3NdO_{18}P_3$	$C_{39}H_{63}DyN_3O_{18}P_3$	$C_{78}H_{126}Cl_6La_2O_{18}P_6$	$C_{39}H_{60}Br_3N_3NdO_{18}P_3$	$C_{78}H_{120}Br_6Cl_6La_2O_{18}P_6$
MW [g mol^{-1}]	435.28	407.14	1099.07	1117.33	2028.12	1335.78	2501.53
T [K]	100(2)	100(2)	100(2)	100(2)	100(2)	100(2)	100(2)
space group	$P2_1/c$	$P\bar{1}$	$P2_1$	$P2_1/n$	$P\bar{1}$	$P\bar{1}$	$P2_1/c$
a [Å]	12.3927(5)	7.6543(6)	15.3724(7)	15.0470(5)	13.9287(4)	16.4380(3)	14.0316(6)
b [Å]	10.6107(4)	11.3119(8)	20.4341(6)	21.2523(7)	13.9984(4)	20.2226(3)	28.3123(14)
c [Å]	15.1024(5)	11.4882(9)	16.3173(6)	16.3650(6)	14.8205(4)	20.3926(3)	14.2123(6)
α [°]	90	115.082(6)	90	90	91.100(2)	61.9330(10)	90
β [°]	101.267(3)	97.147(6)	97.312(3)	97.899(3)	104.058(2)	70.4940(10)	116.555(3)
γ [°]	90	106.395(6)	90	90	119.094(2)	67.9000(10)	90
V [Å]^3	1947.62(13)	829.47(12)	5083.9(3)	5183.6(3)	2417.10(13)	5439.20(16)	5050.4(4)
Z	4	2	4	4	1	4	2
μ [mm^{-1}]	3.799	4.765	1.185	1.602	9.676	11.279	3.520
θ_{max} [°]	70.845	69.479	25.720	25.719	71.350	69.474	26.823
reflections measured	8933	6462	35429	29769	19757	61374	24997
reflections unique	3618	3010	19054	9767	9025	19710	10707
R_{int}	0.0127	0.0322	0.0381	0.0174	0.0233	0.0213	0.0388
data/restr./ para.	3618/39/263	3010/0/203	19054/329/1340	9767/42/676	9025/0/508	19710/106/1309	10707/105/574
R_1 [I>2σ(I)][a]	0.0368	0.0392	0.0432	0.0286	0.0272	0.0341	0.0531
wR_2 (all)[b]	0.0827	0.1087	0.1162	0.0751	0.0755	0.0902	0.1346
$\Delta\rho_{fin}$ [eÅ^{-3}]	0.88/–2.00	0.73/–0.80	1.59/–0.92	0.72/–0.62	0.53/–0.81	1.39/–1.37	1.46/–1.34

[a] $R_1 = (\Sigma||F_o|-|F_c||)/(\Sigma|F_o|)$; [b] $wR_2 = [\{\Sigma w(F_o^2-F_c^2)^2\}/\{\Sigma w(F_o^2)^2\}]^{1/2}$.

Table 2: Crystallographic data for complexes **14**, **17**-**20** and **31**, **32**.

Compound	**14**	**17**	**18**	**19**	**20**	**31**	**32**
CCDC number	2021057	2021058	2021059	2021060	2021061	2021062	2021063
formula	$C_{78}H_{120}Br_6Cl_6Nd_2O_{18}P_6$	$C_{88}H_{108}Br_4Cl_6La_2O_{14}P_4 \cdot C_{14}H_8Br_2 \cdot 2EtOH$	$C_{63}H_{74}Br_3Cl_3NdO_{10}P_3$	$C_{44}H_{54}Br_2Cl_3DyO_7P_2 \cdot 2EtOH$	$C_{63}H_{72}Br_3Cl_3ErO_9P_3 \cdot THF$	$C_{39}H_{50}Br_3Cl_2DyF_{12}O_{10}P_3 \cdot Cl^-$	$C_{39}H_{50}Br_3Cl_2ErF_{12}O_{10}P_3 \cdot Cl^-$
MW [g mol^{-1}]	2512.19	2751.94	1574.45	1277.61	1651.56	1508.28	1513.04
T [K]	100(2)	100(2)	100(2)	100(2)	100(2)	100(2)	100(2)
space group	$P2_1/c$	$P\bar{1}$	$P\bar{1}$	$P\bar{1}$	$P2_1/c$	$P\bar{1}$	$P\bar{1}$
a [Å]	14.0648(5)	13.4455(5)	13.6169(7)	11.2192(4)	21.3578(6)	13.4866(11)	13.3443(19)
b [Å]	29.1214(12)	13.7347(6)	15.8635(9)	13.3757(4)	16.3493(6)	14.6216(17)	14.464(2)
c [Å]	14.2850(5)	15.6918(7)	17.6449(8)	20.6992(7)	22.3921(6)	16.3342(12)	16.184(2)
α [°]	90	97.274(3)	103.591(4)	101.357(3)	90	110.663(7)	111.069(10)
β [°]	116.184(2)	98.726(3)	100.785(4)	98.671(3)	115.243(2)	110.117(6)	110.094(11)
γ [°]	90	93.626(3)	111.726(4)	111.903(2)	90	91.210(8)	91.110(12)
V [Å]^3	5250.5(3)	2831.1(2)	3279.2(3)	2738.39(17)	7072.3(4)	2792.5(5)	2700.8(7)
Z	2	1	2	2	4	2	2
μ [mm^{-1}]	12.863	3.120	2.870	11.311	6.311	3.802	4.102
θ_{max} [°]	69.496	26.834	26.851	71.152	69.984	25.798	25.829
reflections measured	24449	22575	26012	20943	26810	19255	18654
reflections unique	9520	11968	13831	10129	12928	10468	10143
R_{int}	0.0879	0.0386	0.0629	0.0275	0.0515	0.0544	0.0668
data/restr./ para.	9520/0/535	11968/95/738	13831/0/768	10129/49/627	12928/0/796	10468/0/660	10143/18/653
R_1 [I>2σ(I)][a]	0.0995	0.0437	0.0477	0.0470	0.0661	0.0597	0.0594
wR_2 (all)[b]	0.3002	0.1035	0.1140	0.1317	0.1915	0.1435	0.1681
$\Delta\rho_{fin}$ [eÅ^{-3}]	2.64/–1.26	1.05/–1.50	1.36/–1.02	1.78/–1.64	3.05/–1.45	1.38/–1.64	2.32/–1.25
[a] $R_1 = (\Sigma\|F_o\|-\|F_c\|)/(\Sigma\|F_o\|)$; [b] w$R_2 = [\{\Sigma w(F_o^2-F_c^2)^2\}/\{\Sigma w(F_o^2)^2\}]^{1/2}$.							

2.1 L3

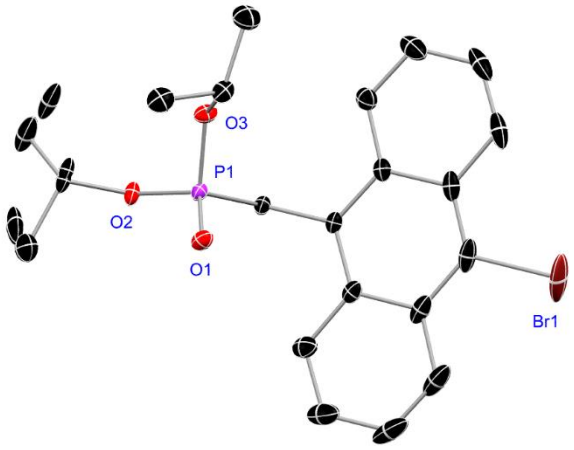

Figure 40: Asymmetric unit of **L3**.

L3 crystalizes in the space group $P2_1/c$. One of the two *i*Pr residues is disordered about two positions. They are refined with distance restraints and restraints for the anisotropic displacement parameters. One carbon atom and its corresponding disordered congener are constrained to be the same. The occupancy of the minor position refined to 0.340(17).

Bond lengths [Å] and angles [°] of **L3**.

Br(1)–C(9)	*1.910(2)*	*C(11)–C(12)*	*1.348(4)*
O(1)–P(1)	*1.4679(17)*	*C(12)–C(13)*	*1.417(4)*
P(1)–O(2)	*1.5782(16)*	*C(13)–C(14)*	*1.358(4)*
P(1)–O(3)	*1.5829(16)*	*C(14)–C(15)*	*1.426(3)*
P(1)–C(1)	*1.805(2)*	*O(2)–C(16)*	*1.471(7)*
C(1)–C(2)	*1.518(3)*	*O(2)–C(16A)*	*1.477(14)*
C(2)–C(15)	*1.405(3)*	*C(16)–C(18)*	*1.503(9)*
C(2)–C(3)	*1.409(3)*	*C(16)–C(17)*	*1.509(7)*
C(3)–C(4)	*1.424(4)*	*C(16A)–C(17A)*	*1.503(16)*
C(3)–C(8)	*1.446(3)*	*C(16A)–C(18A)*	*1.505(14)*
O(3)–C(19)	*1.470(3)*	*C(20)–C(19)*	*1.513(4)*
C(4)–C(5)	*1.360(4)*	*C(21)–C(19)*	*1.513(4)*
C(5)–C(6)	*1.413(4)*		
C(8)–C(9)	*1.400(4)*		
C(8)–C(7)	*1.427(4)*	*O(1)–P(1)–O(2)*	*116.40(10)*
C(7)–C(6)	*1.352(5)*	*O(1)–P(1)–O(3)*	*113.59(9)*
C(9)–C(10)	*1.394(4)*	*O(2)–P(1)–O(3)*	*103.63(9)*
C(10)–C(11)	*1.432(4)*	*O(1)–P(1)–C(1)*	*116.07(10)*
C(10)–C(15)	*1.449(3)*	*O(2)–P(1)–C(1)*	*98.97(9)*

O(3)–P(1)–C(1)	*106.41(10)*	*C(11)–C(10)–C(15)*	*118.3(2)*
C(2)–C(1)–P(1)	*114.22(15)*	*C(12)–C(11)–C(10)*	*121.8(2)*
C(15)–C(2)–C(3)	*120.5(2)*	*C(11)–C(12)–C(13)*	*120.2(2)*
C(15)–C(2)–C(1)	*120.1(2)*	*C(14)–C(13)–C(12)*	*120.2(3)*
C(3)–C(2)–C(1)	*119.4(2)*	*C(13)–C(14)–C(15)*	*122.3(2)*
C(2)–C(3)–C(4)	*122.6(2)*	*C(2)–C(15)–C(14)*	*122.5(2)*
C(2)–C(3)–C(8)	*120.1(2)*	*C(2)–C(15)–C(10)*	*120.3(2)*
C(4)–C(3)–C(8)	*117.3(2)*	*C(14)–C(15)–C(10)*	*117.2(2)*
C(19)–O(3)–P(1)	*123.47(15)*	*C(16)–O(2)–P(1)*	*121.7(5)*
C(5)–C(4)–C(3)	*122.1(3)*	*C(16A)–O(2)–P(1)*	*118.6(10)*
C(4)–C(5)–C(6)	*120.4(3)*	*O(2)–C(16)–C(18)*	*106.5(6)*
C(9)–C(8)–C(7)	*124.1(2)*	*O(2)–C(16)–C(17)*	*109.2(5)*
C(9)–C(8)–C(3)	*117.6(2)*	*C(18)–C(16)–C(17)*	*112.7(7)*
C(7)–C(8)–C(3)	*118.3(3)*	*O(2)–C(16A)–C(17A)*	*105.3(13)*
C(6)–C(7)–C(8)	*121.9(3)*	*O(2)–C(16A)–C(18A)*	*107.4(11)*
C(7)–C(6)–C(5)	*120.0(3)*	*C(17A)–C(16A)–C(18A)*	*114.7(15)*
C(10)–C(9)–C(8)	*123.9(2)*	*O(3)–C(19)–C(20)*	*109.74(19)*
C(10)–C(9)–Br(1)	*118.0(2)*	*O(3)–C(19)–C(21)*	*105.8(2)*
C(8)–C(9)–Br(1)	*118.1(2)*	*C(20)–C(19)–C(21)*	*113.0(2)*
C(9)–C(10)–C(11)	*124.2(2)*		
C(9)–C(10)–C(15)	*117.6(2)*		

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Structure factors have been supplied for datablock(s) sv0839

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Datablock: sv0839

Bond precision: C-C = 0.0035 A Wavelength=1.54186

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	alpha=90	*beta=101.267(3)*	*gamma=90*

Temperature: *100 K*

	Calculated	*Reported*
Volume	*1947.62(13)*	*1947.62(13)*
Space group	*P 21/c*	*P 21/c*
Hall group	*-P 2ybc*	*-P 2ybc*

Moiety formula	*C21 H24 Br O3 P*	*?*
Sum formula	*C21 H24 Br O3 P*	*C21 H24 Br O3 P*
Mr	*435.27*	*435.28*
Dx,g cm-3	*1.485*	*1.484*
Z	*4*	*4*
Mu (mm-1)	*3.799*	*3.799*
F000	*896.0*	*896.0*
F000'	*896.51*	
h,k,lmax	*15,13,18*	*15,12,18*
Nref	*3746*	*3618*
Tmin,Tmax	*0.477,0.710*	*0.399,0.717*
Tmin'	*0.368*	

Correction method= # Reported T Limits: Tmin=0.399 Tmax=0.717
AbsCorr = MULTI-SCAN

Data completeness= 0.966 *Theta(max)= 70.845*

R(reflections)= 0.0368(3420) *wR2(reflections)= 0.0827(3618)*

S = 1.072 *Npar= 263*

The following ALERTS were generated. Each ALERT has the format ***test-name_ALERT_alert-type_alert-level****.*

Click on the hyperlinks for more details of the test.

Alert level C

PLAT213_ALERT_2_C Atom Br1 has ADP max/min Ratio	*3.7 prolat*
PLAT213_ALERT_2_C Atom C17A has ADP max/min Ratio	*3.1 prolat*
PLAT220_ALERT_2_C NonSolvent Resd 1 C Ueq(max) / Ueq(min) Range	*3.1 Ratio*
PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L= 0.600	*47 Report*
PLAT918_ALERT_3_C Reflection(s) with I(obs) much Smaller I(calc) .	*1 Check*
PLAT972_ALERT_2_C Check Calcd Resid. Dens. 1.05A From Br1	*-1.86 eA-3*

Alert level G

PLAT002_ALERT_2_G Number of Distance or Angle Restraints on AtSite	*7 Note*
PLAT003_ALERT_2_G Number of Uiso or Uij Restrained non-H Atoms ...	*5 Report*
PLAT171_ALERT_4_G The CIF-Embedded .res File Contains EADP Records	*1 Report*
PLAT175_ALERT_4_G The CIF-Embedded .res File Contains SAME Records	*1 Report*
PLAT178_ALERT_4_G The CIF-Embedded .res File Contains SIMU Records	*2 Report*
PLAT187_ALERT_4_G The CIF-Embedded .res File Contains RIGU Records	*1 Report*
PLAT301_ALERT_3_G Main Residue Disorder .....(Resd 1)	*12% Note*
PLAT720_ALERT_4_G Number of Unusual/Non-Standard Labels	*1 Note*
PLAT793_ALERT_4_G Model has Chirality at P1 (Centro SPGR)	*R Verify*
PLAT860_ALERT_3_G Number of Least-Squares Restraints	*39 Note*
PLAT883_ALERT_1_G No Info/Value for _atom_sites_solution_primary .	*Please Do !*
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L= 0.600	*82 Note*
PLAT941_ALERT_3_G Average HKL Measurement Multiplicity	*2.5 Low*
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density.	*12 Info*

2.2 L6

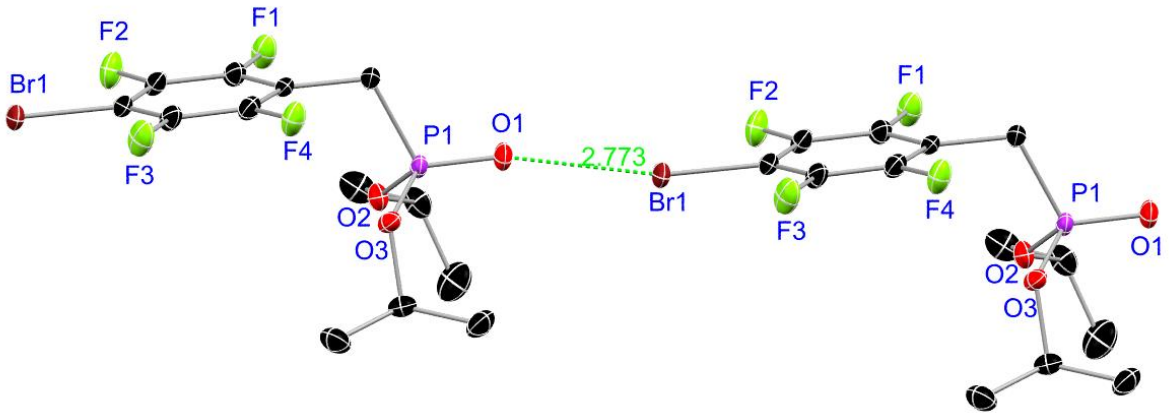

Figure 41: Asymmetric unit of **L6**.

L6 crystalizes in the space group $P\bar{1}$. The asymmetric unit contains one molecule. The -I-effect of the fluoride substituents most likely causes a depletion of electron density at the bromide substituent of each molecule. To compensate for this lack of electron density, the P=O oxygen atom of a neighboring molecule donates electron density to the bromide forming a so-called halogen bond. Thus, the O···Br distance (2.773 Å) is shorter than the sum of the van der Waals radii of both atoms (3.35 Å) causing an A-Alert upon cif-file check.

Bond lengths [Å] and angles [°] of **L6**.

Br(1)–C(5)	*1.873(3)*	*C(8)–C(9)*	*1.509(4)*
P(1)–O(1)	*1.4653(18)*	*C(8)–C(10)*	*1.509(5)*
P(1)–C(1)	*1.803(3)*	*C(11)–C(12)*	*1.509(4)*
P(1)–O(2)	*1.5824(18)*	*C(11)–C(13)*	*1.515(4)*
P(1)–O(3)	*1.5681(18)*		
F(1)–C(3)	*1.347(3)*	*O(1)–P(1)–C(1)*	*112.97(11)*
C(1)–C(2)	*1.503(3)*	*O(1)–P(1)–O(2)*	*113.88(11)*
F(2)–C(4)	*1.339(3)*	*O(1)–P(1)–O(3)*	*117.66(10)*
O(2)–C(8)	*1.473(3)*	*O(2)–P(1)–C(1)*	*106.92(11)*
C(2)–C(3)	*1.382(4)*	*O(3)–P(1)–C(1)*	*100.12(11)*
C(2)–C(7)	*1.392(4)*	*O(3)–P(1)–O(2)*	*103.83(10)*
F(3)–C(6)	*1.340(3)*	*C(2)–C(1)–P(1)*	*113.60(18)*
O(3)–C(11)	*1.471(3)*	*C(8)–O(2)–P(1)*	*122.44(16)*
C(3)–C(4)	*1.375(4)*	*C(3)–C(2)–C(1)*	*122.5(2)*
F(4)–C(7)	*1.339(3)*	*C(3)–C(2)–C(7)*	*116.2(2)*
C(4)–C(5)	*1.388(4)*	*C(7)–C(2)–C(1)*	*121.3(2)*
C(5)–C(6)	*1.385(4)*	*C(11)–O(3)–P(1)*	*122.88(15)*
C(6)–C(7)	*1.377(4)*	*F(1)–C(3)–C(2)*	*119.4(2)*

F(1)–C(3)–C(4)	*117.9(2)*	*F(4)–C(7)–C(2)*	*119.6(2)*
C(4)–C(3)–C(2)	*122.7(2)*	*F(4)–C(7)–C(6)*	*118.5(2)*
F(2)–C(4)–C(3)	*119.4(2)*	*C(6)–C(7)–C(2)*	*121.9(2)*
F(2)–C(4)–C(5)	*120.1(2)*	*O(2)–C(8)–C(10)*	*108.7(2)*
C(3)–C(4)–C(5)	*120.5(2)*	*C(10)–C(8)–C(9)*	*113.6(3)*
C(4)–C(5)–Br(1)	*121.3(2)*	*O(3)–C(11)–C(12)*	*104.9(2)*
C(6)–C(5)–Br(1)	*120.9(2)*	*O(3)–C(11)–C(13)*	*108.7(2)*
C(6)–C(5)–C(4)	*117.7(2)*	*C(12)–C(11)–C(13)*	*113.5(2)*
F(3)–C(6)–C(5)	*120.1(2)*		
F(3)–C(6)–C(7)	*118.9(2)*		
C(7)–C(6)–C(5)	*121.0(2)*		

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No syntax errors found. CIF dictionary Interpreting this report

Datablock: sv0908

Bond precision: C-C = 0.0050 A Wavelength=1.54186

Cell:	a=7.6543(6)	b=11.3119(8)	c=11.4882(9)
	alpha=115.082(6)	beta=97.147(6)	gamma=106.395(6)
Temperature:	100 K		

	Calculated	Reported
Volume	829.47(13)	829.47(12)
Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	C13 H16 Br F4 O3 P	C13 H16 Br F4 O3 P
Sum formula	C13 H16 Br F4 O3 P	C13 H16 Br F4 O3 P
Mr	407.13	407.14
Dx,g cm-3 1.630		1.630
Z	2	2
Mu (mm-1)	4.765	4.765
F000	408.0	408.0
F000’	408.56	
h,k,lmax	9,13,13	9,13,13

Nref	3117	3010
Tmin,Tmax	0.275,0.565	0.132,0.536
Tmin'	0.164	

Correction method= # Reported T Limits: Tmin=0.132 Tmax=0.536 AbsCorr = MULTI-SCAN

Data completeness= 0.966 Theta(max)= 69.479

R(reflections)= 0.0392(2882) wR2(reflections)= 0.1087(3010)

S = 1.052 Npar= 203

The following ALERTS were generated. Each ALERT has the format

***test-name_ALERT_alert-type_alert-level**.*

Click on the hyperlinks for more details of the test.

Alert level A

PLAT431_ALERT_2_A Short Inter HL..A Contact Br1 ..O1 . 2.78 Ang.

x,1+y,z = 1_565 Check

Alert level C

PLAT029_ALERT_3_C _diffrn_measured_fraction_theta_full value Low . 0.977 Why?
PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L= 0.600 70 Report
PLAT918_ALERT_3_C Reflection(s) with I(obs) much Smaller I(calc) . 2 Check
PLAT939_ALERT_3_C Large Value of Not (SHELXL) Weight Optimized S . 10.27 Check

---

Alert level G

PLAT154_ALERT_1_G The s.u.'s on the Cell Angles are Equal ..(Note) 0.006 Degree
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L= 0.600 38 Note
PLAT933_ALERT_2_G Number of OMIT Records in Embedded .res File ... 17 Note
PLAT941_ALERT_3_G Average HKL Measurement Multiplicity ..... 2.1 Low
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density. 3 Info

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2.3 Complex 2

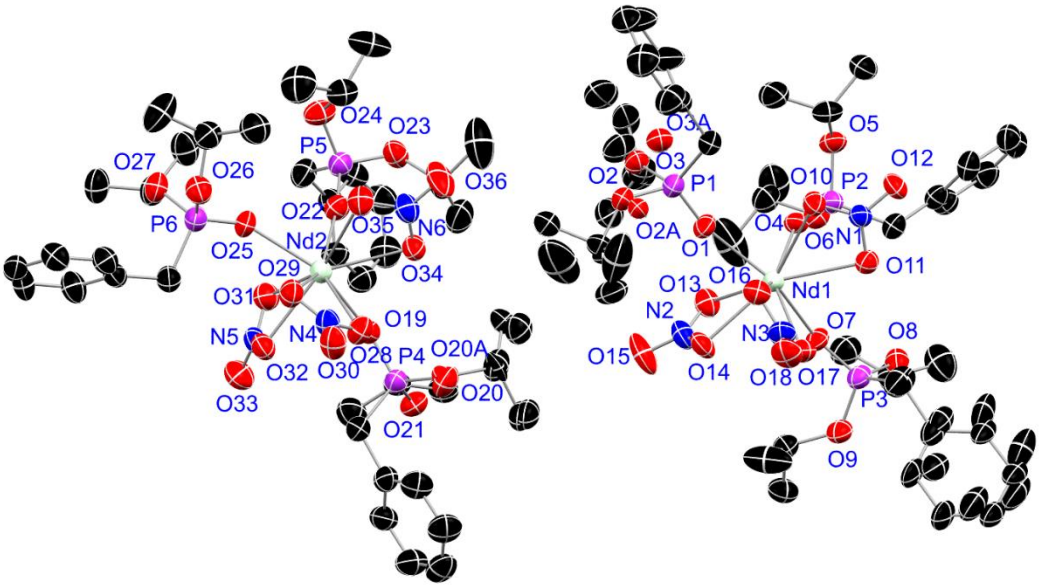

Figure 42: Asymmetric unit of **2**.

2 crystalizes in the space group $P2_1$ and the asymmetric unit contains two molecules. Three of the twelve *i*Pr residues as well as their bridging O-atoms are disordered about two positions. They are refined with distance restraints and partially with restraints for the anisotropic displacement parameters. One *i*Pr carbon atom and its disordered counterpart are constrained to be the same. The occupancy of the minor positions refined to 0.468(27), 0.450(22) and 0.476(24), respectively. Also, one of the phenyl rings is disordered about two positions and they are refined with distance restraints and restraints for the anisotropic displacement parameters. Two of the ring carbon atoms and their disordered congeners are constrained to be the same. The minor position refined to 0.185(31).

Bond lengths [Å] and angles [°] of **2**.

Nd(1)–O(1)	2.349(5)	N(1)–O(10)	1.275(9)
Nd(1)–O(4)	2.397(6)	N(1)–O(12)	1.206(9)
Nd(1)–O(7)	2.386(5)	N(1)–O(11)	1.278(9)
Nd(1)–O(10)	2.565(6)	O(1)–P(1)	1.479(6)
Nd(1)–O(11)	2.540(6)	P(1)–O(2)	1.485(12)
Nd(1)–O(13)	2.521(6)	P(1)–O(2A)	1.635(12)
Nd(1)–O(14)	2.501(6)	P(1)–O(3)	1.488(11)
Nd(1)–O(16)	2.531(6)	P(1)–O(3A)	1.706(18)
Nd(1)–O(17)	2.526(7)	O(2)–C(8)	1.431(18)
C(1)–P(1)	1.754(10)	C(8)–C(9)	1.47(2)
C(1)–C(2)	1.506(13)	C(8)–C(10)	1.49(2)

O(2A)–C(8A)	1.410(19)	P(4)–O(19)	1.478(6)
C(8A)–C(9A)	1.45(2)	P(4)–C(40)	1.788(10)
C(8A)–C(10A)	1.43(2)	P(4)–O(21)	1.550(7)
O(3)–C(11)	1.47(2)	O(20)–C(47)	1.457(18)
C(11)–C(12)	1.50(3)	C(47)–C(48)	1.46(4)
C(11)–C(13)	1.48(3)	C(47)–C(49)	1.51(3)
O(3A)–C(11A)	1.48(2)	O(20A)–C(47A)	1.47(2)
C(11A)–C(12A)	1.50(3)	C(47A)–C(48A)	1.48(4)
C(11A)–C(13A)	1.49(3)	C(47A)–C(49A)	1.52(3)
Nd(2)–O(19)	2.400(6)	N(4)–O(28)	1.279(10)
Nd(2)–O(22)	2.376(6)	N(4)–O(29)	1.260(10)
Nd(2)–O(25)	2.377(6)	N(4)–O(30)	1.190(10)
Nd(2)–O(28)	2.514(7)	C(4)–C(3)	1.383(16)
Nd(2)–O(29)	2.564(7)	C(7)–C(6)	1.384(16)
Nd(2)–O(32)	2.504(6)	O(7)–P(3)	1.487(6)
Nd(2)–O(31)	2.544(7)	O(6)–C(24)	1.488(12)
Nd(2)–O(35)	2.532(7)	P(6)–O(25)	1.483(6)
Nd(2)–O(34)	2.528(6)	P(6)–O(26)	1.561(7)
C(2)–C(7)	1.390(17)	P(6)–O(27)	1.557(7)
C(2)–C(3)	1.350(15)	P(6)–C(66)	1.783(10)
N(2)–O(13)	1.265(11)	N(6)–O(36)	1.190(10)
N(2)–O(14)	1.260(10)	N(6)–O(35)	1.282(10)
N(2)–O(15)	1.206(10)	N(6)–O(34)	1.268(10)
P(2)–O(5)	1.553(6)	O(8)–C(34)	1.479(11)
P(2)–O(4)	1.491(6)	O(8)–P(3)	1.565(7)
P(2)–O(6)	1.559(6)	O(9)–C(37)	1.467(11)
P(2)–C(14)	1.772(9)	O(9)–P(3)	1.553(7)
N(5)–O(32)	1.280(10)	C(14)–C(15)	1.511(13)
N(5)–O(31)	1.273(11)	C(15)–C(20)	1.383(14)
N(5)–O(33)	1.225(10)	C(15)–C(16)	1.403(14)
C(5)–C(4)	1.370(18)	C(20)–C(19)	1.392(15)
C(5)–C(6)	1.375(18)	C(16)–C(17)	1.394(14)
O(5)–C(21)	1.472(11)	O(16)–N(3)	1.271(10)
P(5)–O(22)	1.482(7)	O(18)–N(3)	1.213(10)
P(5)–O(23)	1.529(7)	C(18)–C(17)	1.367(16)
P(5)–O(24)	1.573(7)	C(18)–C(19)	1.361(15)
P(5)–C(53)	1.773(10)	O(17)–N(3)	1.273(11)
P(4)–O(20)	1.546(15)	C(40)–C(41)	1.516(13)
P(4)–O(20A)	1.538(17)	C(42)–C(41)	1.389(15)

C(42)–C(43)	1.372(16)	C(35)–C(34)	1.490(16)
C(41)–C(46)	1.366(14)	C(37)–C(38)	1.507(13)
C(43)–C(44)	1.380(17)	C(37)–C(39)	1.495(14)
C(46)–C(45)	1.379(15)	P(3)–C(27)	1.78(3)
C(45)–C(44)	1.388(16)	P(3)–C(27A)	1.779(12)
C(22)–C(21)	1.497(13)	C(27)–C(28)	1.53(3)
C(21)–C(23)	1.464(14)	C(28)–C(29)	1.41(3)
O(21)–C(50)	1.486(11)	C(28)–C(33)	1.37(3)
O(23)–C(60)	1.470(12)	C(29)–C(30)	1.39(3)
C(24)–C(25)	1.469(16)	C(30)–C(31)	1.36(4)
C(24)–C(26)	1.484(18)	C(31)–C(32)	1.36(4)
O(24)–C(63)	1.460(12)	C(32)–C(33)	1.39(4)
O(26)–C(73)	1.456(13)	C(27A)–C(28A)	1.534(15)
O(27)–C(76)	1.453(12)	C(28A)–C(29A)	1.40(2)
C(50)–C(51)	1.504(14)	C(28A)–C(33A)	1.37(2)
C(50)–C(52)	1.480(13)	C(29A)–C(30A)	1.40(2)
C(53)–C(54)	1.485(14)	C(30A)–C(31A)	1.36(3)
C(54)–C(55)	1.395(14)	C(31A)–C(32A)	1.36(3)
C(54)–C(59)	1.372(14)	C(32A)–C(33A)	1.38(2)
C(55)–C(56)	1.384(15)		
C(56)–C(57)	1.368(16)	N(1)–Nd(1)–N(3)	86.5(2)
C(57)–C(58)	1.370(17)	O(1)–Nd(1)–N(1)	100.2(2)
C(58)–C(59)	1.418(16)	O(1)–Nd(1)–N(2)	75.2(2)
C(60)–C(61)	1.514(19)	O(1)–Nd(1)–O(4)	87.2(2)
C(60)–C(62)	1.489(19)	O(1)–Nd(1)–O(7)	151.4(2)
C(63)–C(64)	1.497(17)	O(1)–Nd(1)–O(10)	75.4(2)
C(63)–C(65)	1.490(16)	O(1)–Nd(1)–O(11)	125.4(2)
C(66)–C(67)	1.507(13)	O(1)–Nd(1)–O(13)	75.3(2)
C(67)–C(68)	1.387(13)	O(1)–Nd(1)–O(14)	80.4(2)
C(67)–C(72)	1.370(14)	O(1)–Nd(1)–O(16)	75.2(2)
C(68)–C(69)	1.396(14)	O(1)–Nd(1)–O(17)	123.0(2)
C(69)–C(70)	1.355(15)	O(1)–Nd(1)–N(3)	99.3(2)
C(70)–C(71)	1.378(15)	N(2)–Nd(1)–N(1)	173.5(2)
C(71)–C(72)	1.386(14)	N(2)–Nd(1)–N(3)	98.7(2)
C(73)–C(74)	1.510(15)	O(4)–Nd(1)–N(1)	70.7(2)
C(73)–C(75)	1.502(14)	O(4)–Nd(1)–N(2)	104.2(2)
C(76)–C(77)	1.506(13)	O(4)–Nd(1)–O(10)	74.2(2)
C(76)–C(78)	1.498(16)	O(4)–Nd(1)–O(11)	77.22(19)
C(36)–C(34)	1.511(15)	O(4)–Nd(1)–O(13)	78.9(2)

O(4)–Nd(1)–O(14)	129.4(2)	O(16)–Nd(1)–O(11)	89.9(2)
O(4)–Nd(1)–O(16)	146.5(2)	O(16)–Nd(1)–N(3)	25.0(2)
O(4)–Nd(1)–O(17)	147.3(2)	O(17)–Nd(1)–N(1)	90.0(2)
O(4)–Nd(1)–N(3)	157.1(2)	O(17)–Nd(1)–N(2)	96.4(2)
O(7)–Nd(1)–N(1)	100.4(2)	O(17)–Nd(1)–O(10)	99.6(2)
O(7)–Nd(1)–N(2)	82.5(2)	O(17)–Nd(1)–O(11)	74.7(2)
O(7)–Nd(1)–O(4)	81.2(2)	O(17)–Nd(1)–O(16)	50.0(2)
O(7)–Nd(1)–O(10)	125.27(19)	O(17)–Nd(1)–N(3)	25.0(2)
O(7)–Nd(1)–O(11)	77.4(2)	C(2)–C(1)–P(1)	115.8(7)
O(7)–Nd(1)–O(13)	76.8(2)	O(10)–N(1)–Nd(1)	59.2(4)
O(7)–Nd(1)–O(14)	87.0(2)	O(10)–N(1)–O(11)	115.7(6)
O(7)–Nd(1)–O(16)	126.5(2)	O(12)–N(1)–Nd(1)	167.0(6)
O(7)–Nd(1)–O(17)	76.6(2)	O(12)–N(1)–O(10)	122.2(7)
O(7)–Nd(1)–N(3)	101.6(2)	O(12)–N(1)–O(11)	122.1(7)
O(10)–Nd(1)–N(1)	25.27(19)	O(11)–N(1)–Nd(1)	58.1(4)
O(10)–Nd(1)–N(2)	150.5(2)	P(1)–O(1)–Nd(1)	161.2(4)
O(10)–Nd(1)–N(3)	86.1(2)	O(1)–P(1)–C(1)	113.1(4)
O(11)–Nd(1)–N(1)	25.27(19)	O(1)–P(1)–O(2)	118.0(6)
O(11)–Nd(1)–N(2)	159.3(2)	O(1)–P(1)–O(2A)	110.2(6)
O(11)–Nd(1)–O(10)	50.08(19)	O(1)–P(1)–O(3)	112.4(6)
O(11)–Nd(1)–N(3)	81.2(2)	O(1)–P(1)–O(3A)	109.2(6)
O(13)–Nd(1)–N(1)	149.4(2)	O(2)–P(1)–C(1)	115.7(8)
O(13)–Nd(1)–N(2)	25.4(2)	O(2)–P(1)–O(3)	76.4(10)
O(13)–Nd(1)–O(10)	140.8(2)	O(2A)–P(1)–C(1)	97.7(6)
O(13)–Nd(1)–O(11)	147.0(2)	O(2A)–P(1)–O(3A)	130.4(9)
O(13)–Nd(1)–O(16)	122.1(2)	O(3)–P(1)–C(1)	116.5(7)
O(13)–Nd(1)–O(17)	118.2(2)	O(3A)–P(1)–C(1)	93.4(7)
O(13)–Nd(1)–N(3)	124.0(2)	C(8)–O(2)–P(1)	135.3(14)
O(14)–Nd(1)–N(1)	159.8(2)	O(2)–C(8)–C(9)	111(2)
O(14)–Nd(1)–N(2)	25.2(2)	O(2)–C(8)–C(10)	96.8(16)
O(14)–Nd(1)–O(10)	145.2(2)	C(9)–C(8)–C(10)	105(2)
O(14)–Nd(1)–O(11)	146.9(2)	C(8A)–O(2A)–P(1)	119.3(14)
O(14)–Nd(1)–O(13)	50.6(2)	O(2A)–C(8A)–C(9A)	115.5(19)
O(14)–Nd(1)–O(16)	76.1(2)	O(2A)–C(8A)–C(10A)	133(2)
O(14)–Nd(1)–O(17)	73.3(2)	C(10A)–C(8A)–C(9A)	102.0(19)
O(14)–Nd(1)–N(3)	73.5(2)	C(11)–O(3)–P(1)	121.7(13)
O(16)–Nd(1)–N(1)	84.5(2)	O(3)–C(11)–C(12)	105(2)
O(16)–Nd(1)–N(2)	98.6(2)	O(3)–C(11)–C(13)	108.8(17)
O(16)–Nd(1)–O(10)	73.8(2)	C(13)–C(11)–C(12)	113.4(18)

C(11A)–O(3A)–P(1)	122.5(17)	O(28)–Nd(2)–N(6)	78.4(2)
O(3A)–C(11A)–C(12A)	104(2)	O(28)–Nd(2)–O(29)	50.0(2)
O(3A)–C(11A)–C(13A)	111(2)	O(28)–Nd(2)–O(31)	126.7(2)
C(13A)–C(11A)–C(12A)	111(3)	O(28)–Nd(2)–O(35)	84.3(2)
N(5)–Nd(2)–N(4)	102.5(2)	O(28)–Nd(2)–O(34)	71.1(2)
N(5)–Nd(2)–N(6)	165.2(2)	O(29)–Nd(2)–N(5)	99.5(2)
N(6)–Nd(2)–N(4)	84.3(2)	O(29)–Nd(2)–N(4)	24.9(2)
O(19)–Nd(2)–N(5)	67.6(2)	O(29)–Nd(2)–N(6)	92.2(2)
O(19)–Nd(2)–N(4)	97.7(2)	O(32)–Nd(2)–N(5)	25.8(2)
O(19)–Nd(2)–N(6)	98.6(2)	O(32)–Nd(2)–N(4)	76.9(2)
O(19)–Nd(2)–O(28)	74.5(2)	O(32)–Nd(2)–N(6)	158.9(2)
O(19)–Nd(2)–O(29)	119.8(2)	O(32)–Nd(2)–O(28)	80.5(2)
O(19)–Nd(2)–O(32)	74.6(2)	O(32)–Nd(2)–O(29)	74.9(2)
O(19)–Nd(2)–O(31)	72.9(2)	O(32)–Nd(2)–O(31)	50.8(2)
O(19)–Nd(2)–O(35)	123.9(2)	O(32)–Nd(2)–O(35)	151.6(2)
O(19)–Nd(2)–O(34)	73.5(2)	O(32)–Nd(2)–O(34)	141.7(2)
O(22)–Nd(2)–N(5)	100.0(2)	O(31)–Nd(2)–N(5)	25.7(2)
O(22)–Nd(2)–N(4)	157.5(2)	O(31)–Nd(2)–N(4)	127.7(2)
O(22)–Nd(2)–N(6)	73.7(2)	O(31)–Nd(2)–N(6)	147.3(2)
O(22)–Nd(2)–O(19)	90.2(2)	O(31)–Nd(2)–O(29)	119.8(2)
O(22)–Nd(2)–O(25)	85.5(2)	O(35)–Nd(2)–N(5)	168.3(2)
O(22)–Nd(2)–O(28)	145.7(2)	O(35)–Nd(2)–N(4)	79.1(2)
O(22)–Nd(2)–O(29)	148.8(2)	O(35)–Nd(2)–N(6)	25.5(2)
O(22)–Nd(2)–O(32)	125.5(2)	O(35)–Nd(2)–O(29)	77.0(2)
O(22)–Nd(2)–O(31)	74.8(2)	O(35)–Nd(2)–O(31)	149.0(2)
O(22)–Nd(2)–O(35)	79.0(2)	O(34)–Nd(2)–N(5)	140.9(2)
O(22)–Nd(2)–O(34)	75.2(2)	O(34)–Nd(2)–N(4)	86.9(2)
O(25)–Nd(2)–N(5)	86.4(2)	O(34)–Nd(2)–N(6)	25.1(2)
O(25)–Nd(2)–N(4)	96.5(2)	O(34)–Nd(2)–O(29)	103.6(2)
O(25)–Nd(2)–N(6)	106.1(2)	O(34)–Nd(2)–O(31)	134.3(2)
O(25)–Nd(2)–O(19)	152.5(2)	O(34)–Nd(2)–O(35)	50.4(2)
O(25)–Nd(2)–O(28)	121.7(2)	C(7)–C(2)–C(1)	119.8(10)
O(25)–Nd(2)–O(29)	71.7(2)	C(3)–C(2)–C(1)	121.9(10)
O(25)–Nd(2)–O(32)	85.9(2)	C(3)–C(2)–C(7)	118.3(10)
O(25)–Nd(2)–O(31)	79.8(2)	O(13)–N(2)–Nd(1)	58.7(4)
O(25)–Nd(2)–O(35)	81.9(2)	O(14)–N(2)–Nd(1)	57.8(4)
O(25)–Nd(2)–O(34)	130.7(2)	O(14)–N(2)–O(13)	116.3(7)
O(28)–Nd(2)–N(5)	102.1(2)	O(15)–N(2)–Nd(1)	174.2(7)
O(28)–Nd(2)–N(4)	25.2(2)	O(15)–N(2)–O(13)	121.9(9)

O(15)–N(2)–O(14)	121.8(9)	O(28)–N(4)–Nd(2)	56.7(4)
O(5)–P(2)–O(6)	103.4(4)	O(29)–N(4)–Nd(2)	58.8(4)
O(5)–P(2)–C(14)	109.4(4)	O(29)–N(4)–O(28)	115.5(7)
O(4)–P(2)–O(5)	112.6(4)	O(30)–N(4)–Nd(2)	176.3(6)
O(4)–P(2)–O(6)	114.6(4)	O(30)–N(4)–O(28)	121.3(8)
O(4)–P(2)–C(14)	111.7(4)	O(30)–N(4)–O(29)	123.1(8)
O(6)–P(2)–C(14)	104.5(4)	C(5)–C(4)–C(3)	121.8(12)
O(32)–N(5)–Nd(2)	58.3(4)	C(6)–C(7)–C(2)	120.4(13)
O(31)–N(5)–Nd(2)	60.0(4)	P(3)–O(7)–Nd(1)	149.4(4)
O(31)–N(5)–O(32)	115.9(7)	C(24)–O(6)–P(2)	120.1(6)
O(33)–N(5)–Nd(2)	164.6(7)	O(25)–P(6)–O(26)	114.9(4)
O(33)–N(5)–O(32)	122.3(9)	O(25)–P(6)–O(27)	111.6(4)
O(33)–N(5)–O(31)	121.8(9)	O(25)–P(6)–C(66)	112.6(4)
C(4)–C(5)–C(6)	117.3(11)	O(26)–P(6)–C(66)	103.4(5)
C(21)–O(5)–P(2)	123.6(6)	O(27)–P(6)–O(26)	104.1(4)
O(22)–P(5)–O(23)	111.7(4)	O(27)–P(6)–C(66)	109.7(5)
O(22)–P(5)–O(24)	113.4(4)	O(36)–N(6)–Nd(2)	173.0(8)
O(22)–P(5)–C(53)	113.8(5)	O(36)–N(6)–O(35)	121.5(9)
O(23)–P(5)–O(24)	106.1(4)	O(36)–N(6)–O(34)	123.2(8)
O(23)–P(5)–C(53)	107.9(5)	O(35)–N(6)–Nd(2)	58.0(4)
O(24)–P(5)–C(53)	103.4(4)	O(34)–N(6)–Nd(2)	57.8(4)
P(2)–O(4)–Nd(1)	153.7(4)	O(34)–N(6)–O(35)	115.3(7)
O(20)–P(4)–C(40)	102.3(9)	C(5)–C(6)–C(7)	121.1(14)
O(20)–P(4)–O(21)	108.2(12)	C(34)–O(8)–P(3)	124.9(6)
O(20A)–P(4)–C(40)	102.6(13)	C(37)–O(9)–P(3)	124.7(5)
O(20A)–P(4)–O(21)	102.7(15)	N(1)–O(10)–Nd(1)	95.5(4)
O(19)–P(4)–O(20)	111.9(14)	N(1)–O(11)–Nd(1)	96.7(4)
O(19)–P(4)–O(20A)	116.8(16)	N(2)–O(13)–Nd(1)	95.9(5)
O(19)–P(4)–C(40)	112.8(4)	N(2)–O(14)–Nd(1)	97.0(5)
O(19)–P(4)–O(21)	112.5(4)	C(15)–C(14)–P(2)	117.2(6)
O(21)–P(4)–C(40)	108.5(4)	C(20)–C(15)–C(14)	120.5(9)
C(47)–O(20)–P(4)	130(2)	C(20)–C(15)–C(16)	118.6(9)
O(20)–C(47)–C(49)	108(2)	C(16)–C(15)–C(14)	120.9(9)
C(48)–C(47)–O(20)	112(2)	C(15)–C(20)–C(19)	121.0(10)
C(48)–C(47)–C(49)	118(5)	C(17)–C(16)–C(15)	119.7(10)
C(47A)–O(20A)–P(4)	127(3)	N(3)–O(16)–Nd(1)	97.8(5)
O(20A)–C(47A)–C(48A)	110(3)	C(19)–C(18)–C(17)	121.2(10)
O(20A)–C(47A)–C(49A)	105(2)	C(18)–C(17)–C(16)	120.1(11)
C(48A)–C(47A)–C(49A)	113(4)	N(3)–O(17)–Nd(1)	97.9(5)

P(4)–O(19)–Nd(2)	141.4(4)	C(62)–C(60)–C(61)	112.6(11)
C(18)–C(19)–C(20)	119.4(10)	O(24)–C(63)–C(64)	106.8(10)
C(41)–C(40)–P(4)	113.8(7)	O(24)–C(63)–C(65)	108.5(10)
C(43)–C(42)–C(41)	122.2(11)	C(65)–C(63)–C(64)	112.8(12)
C(42)–C(41)–C(40)	119.8(10)	C(67)–C(66)–P(6)	113.5(7)
C(46)–C(41)–C(40)	121.9(9)	C(68)–C(67)–C(66)	120.0(9)
C(46)–C(41)–C(42)	118.3(10)	C(72)–C(67)–C(66)	122.4(9)
C(42)–C(43)–C(44)	118.8(11)	C(72)–C(67)–C(68)	117.5(9)
C(41)–C(46)–C(45)	120.8(10)	C(67)–C(68)–C(69)	121.4(10)
C(46)–C(45)–C(44)	120.2(11)	C(70)–C(69)–C(68)	119.2(9)
C(43)–C(44)–C(45)	119.8(10)	C(69)–C(70)–C(71)	120.9(9)
P(5)–O(22)–Nd(2)	171.8(4)	C(70)–C(71)–C(72)	119.1(9)
C(23)–C(21)–O(5)	108.2(8)	C(67)–C(72)–C(71)	121.9(9)
C(23)–C(21)–C(22)	112.1(8)	O(26)–C(73)–C(74)	107.0(10)
C(50)–O(21)–P(4)	124.2(6)	O(26)–C(73)–C(75)	107.1(9)
C(60)–O(23)–P(5)	125.3(6)	C(75)–C(73)–C(74)	114.7(10)
C(25)–C(24)–O(6)	107.6(9)	O(27)–C(76)–C(77)	106.7(9)
C(25)–C(24)–C(26)	111.8(11)	O(27)–C(76)–C(78)	108.9(9)
C(26)–C(24)–O(6)	107.8(10)	C(78)–C(76)–C(77)	113.1(10)
C(63)–O(24)–P(5)	119.2(7)	N(5)–O(32)–Nd(2)	96.0(5)
P(6)–O(25)–Nd(2)	150.1(4)	N(5)–O(31)–Nd(2)	94.3(5)
C(73)–O(26)–P(6)	123.9(7)	N(6)–O(35)–Nd(2)	96.5(5)
C(76)–O(27)–P(6)	122.9(6)	O(8)–C(34)–C(36)	109.8(8)
N(4)–O(28)–Nd(2)	98.1(5)	O(8)–C(34)–C(35)	106.0(8)
N(4)–O(29)–Nd(2)	96.3(5)	C(35)–C(34)–C(36)	113.6(11)
O(21)–C(50)–C(51)	104.4(7)	N(6)–O(34)–Nd(2)	97.1(5)
C(52)–C(50)–O(21)	109.6(8)	O(9)–C(37)–C(38)	105.6(7)
C(52)–C(50)–C(51)	114.4(9)	O(9)–C(37)–C(39)	109.0(10)
C(54)–C(53)–P(5)	114.2(7)	C(39)–C(37)–C(38)	113.7(9)
C(55)–C(54)–C(53)	121.1(10)	O(7)–P(3)–O(8)	114.0(4)
C(59)–C(54)–C(53)	119.1(9)	O(7)–P(3)–O(9)	113.0(4)
C(59)–C(54)–C(55)	119.8(10)	O(7)–P(3)–C(27)	113(2)
C(56)–C(55)–C(54)	119.8(10)	O(7)–P(3)–C(27A)	110.0(6)
C(57)–C(56)–C(55)	120.6(11)	O(8)–P(3)–C(27)	102(3)
C(56)–C(57)–C(58)	120.5(11)	O(8)–P(3)–C(27A)	104.6(9)
C(57)–C(58)–C(59)	119.5(11)	O(9)–P(3)–O(8)	106.2(4)
C(54)–C(59)–C(58)	119.7(10)	O(9)–P(3)–C(27)	108(2)
O(23)–C(60)–C(61)	108.0(9)	O(9)–P(3)–C(27A)	108.6(7)
O(23)–C(60)–C(62)	107.3(11)	C(28)–C(27)–P(3)	117(4)

C(29)–C(28)–C(27)	117(4)	C(31A)–C(30A)–C(29A)	118(2)
C(33)–C(28)–C(27)	122(4)	C(30A)–C(31A)–C(32A)	123.3(16)
C(33)–C(28)–C(29)	120(3)	C(31A)–C(32A)–C(33A)	119(2)
C(30)–C(29)–C(28)	119(4)	C(28A)–C(33A)–C(32A)	120(2)
C(31)–C(30)–C(29)	118(4)	C(2)–C(3)–C(4)	120.9(12)
C(32)–C(31)–C(30)	125(4)	O(16)–N(3)–Nd(1)	57.3(4)
C(31)–C(32)–C(33)	117(4)	O(16)–N(3)–O(17)	114.3(7)
C(28)–C(33)–C(32)	121(4)	O(18)–N(3)–Nd(1)	177.7(7)
C(28A)–C(27A)–P(3)	113.7(11)	O(18)–N(3)–O(16)	121.9(9)
C(29A)–C(28A)–C(27A)	118.4(15)	O(18)–N(3)–O(17)	123.8(8)
C(33A)–C(28A)–C(27A)	121.3(16)	O(17)–N(3)–Nd(1)	57.0(4)
C(33A)–C(28A)–C(29A)	120.3(15)		
C(30A)–C(29A)–C(28A)	119.9(19)		

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) i2902

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: i2902

Bond precision:	C-C = 0.0159 A	Wavelength=0.71073	
Cell:	a=15.3724(7)	b=20.4341(6)	c=16.3173(6)
	alpha=90	beta=97.312(3)	gamma=90
Temperature:	100 K		

	Calculated	Reported
Volume	5083.9(3)	5083.9(3)
Space group	P 21	P 1 21 1
Hall group	P 2yb	P 2yb
Moiety formula	C39 H63 N3 Nd O18 P3	C39 H63 N3 Nd O18 P3
Sum formula	C39 H63 N3 Nd O18 P3	C39 H63 N3 Nd O18 P3
Mr	1099.08	1099.07
Dx,g cm-3	1.436	1.436
Z	4	4
Mu (mm-1)	1.185	1.185

F000	2268.0	2268.0
F000'	2269.51	
h,k,lmax	18,24,19	18,24,19
Nref	19375[9978]	19054
Tmin,Tmax	0.855,0.965	0.326,0.918
Tmin'	0.798	

Correction method= # Reported T Limits: Tmin=0.326 Tmax=0.918 AbsCorr = MULTI-SCAN

Data completeness= 1.91/0.98 Theta(max)= 25.720 R(reflections)= 0.0432(16024)

wR2(reflections)= 0.1162(19054)

S = 1.035 Npar= 1340

The following ALERTS were generated. Each ALERT has the format

test-name_ALERT_alert-type_alert-level.

Click on the hyperlinks for more details of the test.

Alert level C

PLAT090_ALERT_3_C	Poor Data / Parameter Ratio (Zmax > 18)	7.37 Note
PLAT220_ALERT_2_C	NonSolvent Resd 1 C Ueq(max) / Ueq(min) Range	3.5 Ratio
PLAT222_ALERT_3_C	NonSolvent Resd 1 H Uiso(max)/Uiso(min) Range	4.2 Ratio
PLAT234_ALERT_4_C	Large Hirshfeld Difference O2A--C8A .	0.18 Ang.
PLAT241_ALERT_2_C	High 'MainMol' Ueq as Compared to Neighbors of	C6 Check
PLAT241_ALERT_2_C	High 'MainMol' Ueq as Compared to Neighbors of	C7 Check
PLAT242_ALERT_2_C	Low 'MainMol' Ueq as Compared to Neighbors of	C2 Check
PLAT242_ALERT_2_C	Low 'MainMol' Ueq as Compared to Neighbors of	C5 Check
PLAT242_ALERT_2_C	Low 'MainMol' Ueq as Compared to Neighbors of	C24 Check
PLAT242_ALERT_2_C	Low 'MainMol' Ueq as Compared to Neighbors of	C37 Check
PLAT242_ALERT_2_C	Low 'MainMol' Ueq as Compared to Neighbors of	C60 Check
PLAT242_ALERT_2_C	Low 'MainMol' Ueq as Compared to Neighbors of	C63 Check
PLAT342_ALERT_3_C	Low Bond Precision on C-C Bonds	0.01589 Ang.
PLAT911_ALERT_3_C	Missing FCF Refl Between Thmin & STh/L= 0.600	5 Report
PLAT918_ALERT_3_C	Reflection(s) with I(obs) much Smaller I(calc) .	1 Check

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Alert level G

PLAT002_ALERT_2_G	Number of Distance or Angle Restraints on AtSite	46 Note
PLAT003_ALERT_2_G	Number of Uiso or Uij Restrained non-H Atoms ...	38 Report
PLAT083_ALERT_2_G	SHELXL Second Parameter in WGHT Unusually Large	6.82 Why?
PLAT171_ALERT_4_G	The CIF-Embedded .res File Contains EADP Records	3 Report

PLAT175_ALERT_4_G	The CIF-Embedded .res File Contains SAME Records		5	Report
PLAT178_ALERT_4_G	The CIF-Embedded .res File Contains SIMU Records		8	Report
PLAT187_ALERT_4_G	The CIF-Embedded .res File Contains RIGU Records		4	Report
PLAT301_ALERT_3_G	Main Residue Disorder .....(Resd 1)		23%	Note
PLAT301_ALERT_3_G	Main Residue Disorder .....(Resd 2)		6%	Note
PLAT395_ALERT_2_G	Deviating X-O-Y Angle From 120 for O2		135.3	Degree
PLAT395_ALERT_2_G	Deviating X-O-Y Angle From 120 for O20		130.2	Degree
PLAT720_ALERT_4_G	Number of Unusual/Non-Standard Labels		3	Note
PLAT794_ALERT_5_G	Tentative Bond Valency for Nd1 (II) .		2.20	Info
PLAT794_ALERT_5_G	Tentative Bond Valency for Nd2 (II) .		2.18	Info
PLAT860_ALERT_3_G	Number of Least-Squares Restraints		329	Note
PLAT883_ALERT_1_G	No Info/Value for _atom_sites_solution_primary .		Please	Do !
PLAT912_ALERT_4_G	Missing # of FCF Reflections Above STh/L=	0.600	96	Note
PLAT933_ALERT_2_G	Number of OMIT Records in Embedded .res File ...		8	Note
PLAT941_ALERT_3_G	Average HKL Measurement Multiplicity		3.6	Low
PLAT978_ALERT_2_G	Number C-C Bonds with Positive Residual Density.		0	Info

2.4 Complex 3

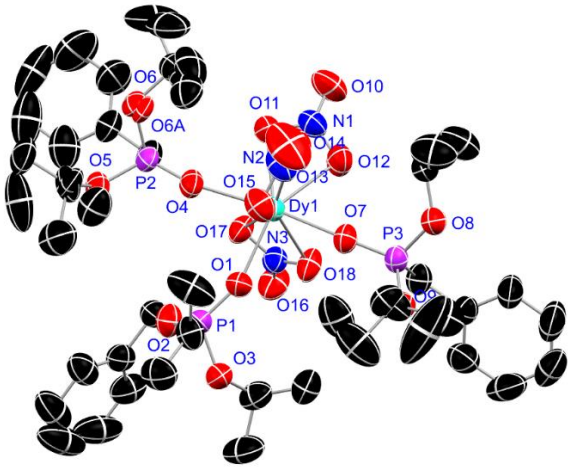

Figure 43: Asymmetric unit of **3**.

3 crystalizes in the space group $P2_1/n$. Three of the six *i*Pr residues are disordered about two positions. One disorder also includes the bridging O-atom. They are refined with distance restraints and partially with restraints for the anisotropic displacement parameters. The occupancy of the minor positions refined to 0.422(11), 0.414(29) and 0.441(32), respectively.

Bond lengths [Å] and angles [°] of **3**.

C(1)–C(2)	1.502(4)	C(24)–C(26)	1.489(12)
C(1)–P(1)	1.785(3)	O(6A)–C(24A)	1.468(14)
C(2)–C(3)	1.371(5)	O(6A)–P(2)	1.60(3)
C(2)–C(7)	1.383(5)	C(24A)–C(25A)	1.489(16)
C(3)–C(4)	1.380(6)	C(24A)–C(26A)	1.489(15)
C(4)–C(5)	1.355(9)	C(27)–C(28)	1.507(4)
C(5)–C(6)	1.346(9)	C(27)–P(3)	1.778(3)
C(6)–C(7)	1.384(7)	C(28)–C(29)	1.356(5)
C(8)–C(9)	1.469(6)	C(28)–C(33)	1.361(5)
C(8)–C(10)	1.487(5)	C(29)–C(30)	1.403(6)
C(8)–O(2)	1.463(4)	C(30)–C(31)	1.367(7)
C(11)–C(12)	1.496(6)	C(31)–C(32)	1.312(7)
C(11)–C(13)	1.466(6)	C(32)–C(33)	1.379(6)
C(11)–O(3)	1.444(4)	C(34)–C(35)	1.492(6)
C(14)–C(15)	1.491(4)	C(34)–C(36)	1.457(6)
C(14)–P(2)	1.778(3)	C(34)–O(8)	1.466(4)
O(6)–C(24)	1.465(10)	O(9)–C(37)	1.458(8)
O(6)–P(2)	1.54(2)	O(9)–C(37A)	1.466(12)
C(24)–C(25)	1.486(12)	O(9)–P(3)	1.552(2)

C(37)–C(38)	1.486(12)	C(7)–C(2)–C(1)	119.8(4)
C(37)–C(39)	1.473(12)	C(2)–C(3)–C(4)	120.8(5)
C(37A)–C(38A)	1.485(15)	C(5)–C(4)–C(3)	120.4(6)
C(37A)–C(39A)	1.468(16)	C(6)–C(5)–C(4)	119.7(5)
Dy(1)–O(1)	2.286(2)	C(5)–C(6)–C(7)	121.1(5)
Dy(1)–O(4)	2.318(2)	C(2)–C(7)–C(6)	119.7(5)
Dy(1)–O(7)	2.2856(19)	C(9)–C(8)–C(10)	112.2(3)
Dy(1)–O(11)	2.465(2)	O(2)–C(8)–C(9)	109.7(3)
Dy(1)–O(12)	2.458(2)	O(2)–C(8)–C(10)	107.5(3)
Dy(1)–O(14)	2.438(2)	C(13)–C(11)–C(12)	113.4(4)
Dy(1)–O(15)	2.462(2)	O(3)–C(11)–C(12)	105.9(4)
Dy(1)–O(17)	2.442(2)	O(3)–C(11)–C(13)	111.4(3)
Dy(1)–O(18)	2.466(2)	C(15)–C(14)–P(2)	115.7(2)
N(1)–O(10)	1.211(4)	C(20)–C(15)–C(14)	120.1(2)
N(1)–O(11)	1.256(3)	C(16)–C(15)–C(14)	119.9(2)
N(1)–O(12)	1.258(4)	C(24)–O(6)–P(2)	123.9(15)
N(2)–O(13)	1.212(4)	O(6)–C(24)–C(25)	109.0(10)
N(2)–O(14)	1.259(4)	O(6)–C(24)–C(26)	107.8(10)
N(2)–O(15)	1.257(4)	C(25)–C(24)–C(26)	111.4(9)
N(3)–O(16)	1.213(3)	C(24A)–O(6A)–P(2)	123(2)
N(3)–O(17)	1.264(3)	O(6A)–C(24A)–C(25A)	106.0(15)
N(3)–O(18)	1.253(3)	O(6A)–C(24A)–C(26A)	105.8(15)
O(1)–P(1)	1.470(2)	C(26A)–C(24A)–C(25A)	111.7(12)
O(2)–P(1)	1.561(2)	C(28)–C(27)–P(3)	116.7(2)
O(3)–P(1)	1.547(2)	C(29)–C(28)–C(27)	119.7(3)
O(4)–P(2)	1.479(2)	C(29)–C(28)–C(33)	117.9(4)
O(7)–P(3)	1.479(2)	C(33)–C(28)–C(27)	122.3(3)
O(8)–P(3)	1.560(2)	C(28)–C(29)–C(30)	120.4(4)
P(2)–O(5)	1.555(2)	C(31)–C(30)–C(29)	120.1(4)
O(5)–C(21)	1.476(9)	C(32)–C(31)–C(30)	118.8(4)
O(5)–C(21A)	1.457(11)	C(31)–C(32)–C(33)	122.1(5)
C(21)–C(22)	1.518(15)	C(28)–C(33)–C(32)	120.8(4)
C(21)–C(23)	1.45(2)	C(36)–C(34)–C(35)	113.3(4)
C(21A)–C(22A)	1.518(17)	C(36)–C(34)–O(8)	107.9(4)
C(21A)–C(23A)	1.42(2)	O(8)–C(34)–C(35)	107.6(4)
		C(37)–O(9)–P(3)	122.6(7)
C(2)–C(1)–P(1)	114.5(2)	C(37A)–O(9)–P(3)	119.0(11)
C(3)–C(2)–C(1)	121.8(3)	O(9)–C(37)–C(38)	106.3(9)
C(3)–C(2)–C(7)	118.3(4)	O(9)–C(37)–C(39)	106.2(8)

C(39)–C(37)–C(38)	111.9(11)	O(11)–Dy(1)–N(3)	89.09(8)
O(9)–C(37A)–C(38A)	110.1(14)	O(11)–Dy(1)–O(18)	102.88(8)
O(9)–C(37A)–C(39A)	110.2(16)	O(12)–Dy(1)–N(1)	25.67(8)
C(39A)–C(37A)–C(38A)	111.7(19)	O(12)–Dy(1)–N(2)	97.34(9)
N(2)–Dy(1)–N(1)	94.86(9)	O(12)–Dy(1)–N(3)	81.73(8)
N(2)–Dy(1)–N(3)	173.83(9)	O(12)–Dy(1)–O(11)	51.16(8)
N(3)–Dy(1)–N(1)	86.92(8)	O(12)–Dy(1)–O(15)	121.80(8)
O(1)–Dy(1)–N(1)	162.43(8)	O(12)–Dy(1)–O(18)	74.93(9)
O(1)–Dy(1)–N(2)	102.63(9)	O(14)–Dy(1)–N(1)	69.04(8)
O(1)–Dy(1)–N(3)	75.86(8)	O(14)–Dy(1)–N(2)	25.84(9)
O(1)–Dy(1)–O(4)	81.84(8)	O(14)–Dy(1)–N(3)	155.02(8)
O(1)–Dy(1)–O(11)	147.48(8)	O(14)–Dy(1)–O(11)	72.31(9)
O(1)–Dy(1)–O(12)	148.91(8)	O(14)–Dy(1)–O(12)	73.65(9)
O(1)–Dy(1)–O(14)	128.47(8)	O(14)–Dy(1)–O(15)	51.63(8)
O(1)–Dy(1)–O(15)	76.92(8)	O(14)–Dy(1)–O(17)	146.12(8)
O(1)–Dy(1)–O(17)	80.49(8)	O(14)–Dy(1)–O(18)	142.33(8)
O(1)–Dy(1)–O(18)	75.53(8)	O(15)–Dy(1)–N(1)	120.44(8)
O(4)–Dy(1)–N(1)	98.57(8)	O(15)–Dy(1)–N(2)	25.85(9)
O(4)–Dy(1)–N(2)	83.78(9)	O(15)–Dy(1)–N(3)	152.57(8)
O(4)–Dy(1)–N(3)	101.82(7)	O(15)–Dy(1)–O(11)	116.19(9)
O(4)–Dy(1)–O(11)	73.13(8)	O(15)–Dy(1)–O(18)	139.55(9)
O(4)–Dy(1)–O(12)	124.23(8)	O(17)–Dy(1)–N(1)	82.47(8)
O(4)–Dy(1)–O(14)	88.86(8)	O(17)–Dy(1)–N(2)	160.25(9)
O(4)–Dy(1)–O(15)	77.45(8)	O(17)–Dy(1)–N(3)	25.88(7)
O(4)–Dy(1)–O(17)	77.32(7)	O(17)–Dy(1)–O(11)	74.10(8)
O(4)–Dy(1)–O(18)	126.38(7)	O(17)–Dy(1)–O(12)	88.70(8)
O(7)–Dy(1)–N(1)	99.61(8)	O(17)–Dy(1)–O(15)	148.12(8)
O(7)–Dy(1)–N(2)	74.03(9)	O(17)–Dy(1)–O(18)	51.56(7)
O(7)–Dy(1)–N(3)	99.86(7)	O(18)–Dy(1)–N(1)	90.51(8)
O(7)–Dy(1)–O(1)	86.99(8)	O(18)–Dy(1)–N(2)	148.20(9)
O(7)–Dy(1)–O(4)	152.30(8)	O(18)–Dy(1)–N(3)	25.70(7)
O(7)–Dy(1)–O(11)	124.39(8)	O(10)–N(1)–Dy(1)	173.1(2)
O(7)–Dy(1)–O(12)	75.83(8)	O(10)–N(1)–O(11)	121.4(3)
O(7)–Dy(1)–O(14)	78.41(8)	O(10)–N(1)–O(12)	123.1(3)
O(7)–Dy(1)–O(15)	75.35(8)	O(11)–N(1)–Dy(1)	58.16(15)
O(7)–Dy(1)–O(17)	125.73(7)	O(11)–N(1)–O(12)	115.5(3)
O(7)–Dy(1)–O(18)	74.17(7)	O(12)–N(1)–Dy(1)	57.86(15)
O(11)–Dy(1)–N(1)	25.64(8)	O(13)–N(2)–Dy(1)	174.8(3)
O(11)–Dy(1)–N(2)	95.08(10)	O(13)–N(2)–O(14)	121.9(4)

O(13)–N(2)–O(15)	122.2(4)	O(3)–P(1)–O(2)	102.45(13)
O(14)–N(2)–Dy(1)	57.56(16)	O(6)–P(2)–C(14)	106.8(8)
O(15)–N(2)–Dy(1)	58.62(16)	O(6)–P(2)–O(5)	102.5(7)
O(15)–N(2)–O(14)	116.0(3)	O(6A)–P(2)–C(14)	114.1(13)
O(16)–N(3)–Dy(1)	176.8(2)	O(4)–P(2)–C(14)	112.10(14)
O(16)–N(3)–O(17)	121.9(2)	O(4)–P(2)–O(6)	116.3(6)
O(16)–N(3)–O(18)	122.1(3)	O(4)–P(2)–O(6A)	108.3(10)
O(17)–N(3)–Dy(1)	57.53(14)	O(4)–P(2)–O(5)	114.08(13)
O(18)–N(3)–Dy(1)	58.58(14)	O(5)–P(2)–C(14)	103.86(15)
O(18)–N(3)–O(17)	116.0(2)	O(5)–P(2)–O(6A)	104.2(11)
P(1)–O(1)–Dy(1)	156.34(14)	O(9)–P(3)–C(27)	103.89(15)
C(8)–O(2)–P(1)	122.8(2)	O(9)–P(3)–O(8)	104.50(13)
C(11)–O(3)–P(1)	125.6(2)	O(7)–P(3)–C(27)	112.74(13)
P(2)–O(4)–Dy(1)	151.40(14)	O(7)–P(3)–O(9)	113.72(13)
P(3)–O(7)–Dy(1)	159.09(14)	O(7)–P(3)–O(8)	112.00(13)
C(34)–O(8)–P(3)	124.3(2)	O(8)–P(3)–C(27)	109.38(15)
N(1)–O(11)–Dy(1)	96.20(18)	C(21)–O(5)–P(2)	126.8(7)
N(1)–O(12)–Dy(1)	96.48(17)	C(21A)–O(5)–P(2)	122.9(7)
N(2)–O(14)–Dy(1)	96.60(19)	O(5)–C(21)–C(22)	107.5(9)
N(2)–O(15)–Dy(1)	95.5(2)	C(23)–C(21)–O(5)	109.3(12)
N(3)–O(17)–Dy(1)	96.59(16)	C(23)–C(21)–C(22)	113.7(12)
N(3)–O(18)–Dy(1)	95.72(16)	O(5)–C(21A)–C(22A)	103.6(11)
O(1)–P(1)–C(1)	112.31(14)	C(23A)–C(21A)–O(5)	112.6(11)
O(1)–P(1)–O(2)	113.83(14)	C(23A)–C(21A)–C(22A)	115.3(13)
O(1)–P(1)–O(3)	113.81(13)		
O(2)–P(1)–C(1)	103.80(15)		
O(3)–P(1)–C(1)	109.78(14)		

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) i2894

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: i2894

Bond precision: C-C = 0.0061 A Wavelength=0.71073

Cell:	a=15.0470(5)	b=21.2523(7)	c=16.3650(6)
	alpha=90	beta=97.899(3)	gamma=90
Temperature:	100 K		

	Calculated	Reported
Volume	5183.6(3)	5183.6(3)
Space group	P 21/n	P 1 21/n 1
Hall group	-P 2yn	-P 2yn
Moiety formula	C39 H63 Dy N3 O18 P3	C39 H63 Dy N3 O18 P3
Sum formula	C39 H63 Dy N3 O18 P3	C39 H63 Dy N3 O18 P3
Mr	1117.34	1117.33
Dx,g cm-3	1.432	1.432
Z	4	4
Mu (mm-1)	1.602	1.602
F000	2292.0	2292.0
F000'	2293.52	
h,k,lmax	18,25,19	18,25,19
Nref	9870	9767
Tmin,Tmax	0.726,0.907	0.678,1.000
Tmin'	0.685	

Correction method= # Reported T Limits: Tmin=0.678 Tmax=1.000 AbsCorr = MULTI-SCAN

Data completeness= 0.990 Theta(max)= 25.719

R(reflections)= 0.0286(8484) wR2(reflections)= 0.0751(9767)

S = 1.037 Npar= 676

The following ALERTS were generated. Each ALERT has the format

***test-name_ALERT_alert-type_alert-level**.*

Click on the hyperlinks for more details of the test.

Alert level C

PLAT220_ALERT_2_C NonSolvent Resd 1	*C*	*Ueq(max) / Ueq(min) Range*	*3.9 Ratio*
PLAT222_ALERT_3_C NonSolvent Resd 1	*H*	*Uiso(max)/Uiso(min) Range*	*4.7 Ratio*
PLAT230_ALERT_2_C Hirshfeld Test Diff for	*C30*	*--C31 .*	*6.0 s.u.*
PLAT234_ALERT_4_C Large Hirshfeld Difference	*C21*	*--C23 .*	*0.19 Ang.*
PLAT234_ALERT_4_C Large Hirshfeld Difference	*C24*	*--C25 .*	*0.18 Ang.*
PLAT234_ALERT_4_C Large Hirshfeld Difference	*C37*	*--C39 .*	*0.18 Ang.*

PLAT241_ALERT_2_C High 'MainMol' Ueq as Compared to Neighbors of O12 Check

PLAT241_ALERT_2_C	High	'MainMol' Ueq as	Compared to	Neighbors of				O18	Check
PLAT241_ALERT_2_C	High	'MainMol' Ueq as	Compared to	Neighbors of				C5	Check
PLAT241_ALERT_2_C	High	'MainMol' Ueq as	Compared to	Neighbors of				C17	Check
PLAT241_ALERT_2_C	High	'MainMol' Ueq as	Compared to	Neighbors of				C19	Check
PLAT241_ALERT_2_C	High	'MainMol' Ueq as	Compared to	Neighbors of				C30	Check
PLAT241_ALERT_2_C	High	'MainMol' Ueq as	Compared to	Neighbors of				C32	Check
PLAT242_ALERT_2_C	Low	'MainMol' Ueq as	Compared to	Neighbors of				Dy1	Check
PLAT242_ALERT_2_C	Low	'MainMol' Ueq as	Compared to	Neighbors of				C8	Check
PLAT242_ALERT_2_C	Low	'MainMol' Ueq as	Compared to	Neighbors of				C11	Check
PLAT242_ALERT_2_C	Low	'MainMol' Ueq as	Compared to	Neighbors of				C15	Check
PLAT242_ALERT_2_C	Low	'MainMol' Ueq as	Compared to	Neighbors of				C28	Check
PLAT242_ALERT_2_C	Low	'MainMol' Ueq as	Compared to	Neighbors of				C34	Check
PLAT331_ALERT_2_C	*Small Aver Phenyl C-C Dist C28*			*--C33*		*.*		*1.36 Ang.*	
PLAT911_ALERT_3_C	*Missing FCF Refl Between Thmin & STh/L=*			*0.600*				*6 Report*	
PLAT913_ALERT_3_C	Missing # of Very Strong Reflections in FCF							4	Note

Alert level G

PLAT002_ALERT_2_G	Number of Distance or Angle Restraints on AtSite							22	Note
PLAT003_ALERT_2_G	Number of Uiso or Uij Restrained non-H Atoms ...							11	Report
PLAT175_ALERT_4_G	The CIF-Embedded .res File Contains SAME Records							3	Report
PLAT187_ALERT_4_G	The CIF-Embedded .res File Contains RIGU Records							3	Report
PLAT232_ALERT_2_G	Hirshfeld Test Diff (M-X)	Dy1	--O11	.				6.8	s.u.
PLAT232_ALERT_2_G	Hirshfeld Test Diff (M-X)	Dy1	--O12	.				6.9	s.u.
PLAT232_ALERT_2_G	Hirshfeld Test Diff (M-X)	Dy1	--O17	.				5.7	s.u.
PLAT301_ALERT_3_G	Main Residue Disorder .....(Resd				1	)		16%	Note
PLAT793_ALERT_4_G	Model has Chirality at P3		(Centro SPGR)					S	Verify
PLAT794_ALERT_5_G	Tentative Bond Valency for Dy1		(III)	.				3.11	Info
PLAT860_ALERT_3_G	Number of Least-Squares Restraints							42	Note
PLAT883_ALERT_1_G	No Info/Value for _atom_sites_solution_primary .							Please	Do !
PLAT912_ALERT_4_G	Missing # of FCF Reflections Above STh/L=				0.600			98	Note
PLAT933_ALERT_2_G	Number of OMIT Records in Embedded .res File ...							8	Note
PLAT941_ALERT_3_G	Average HKL Measurement Multiplicity							3.0	Low
PLAT978_ALERT_2_G	Number C-C Bonds with Positive Residual Density.							3	Info
PLAT992_ALERT_5_G	Repd & Actual _reflns_number_gt Values Differ by							1	Check

2.5 Complex 5

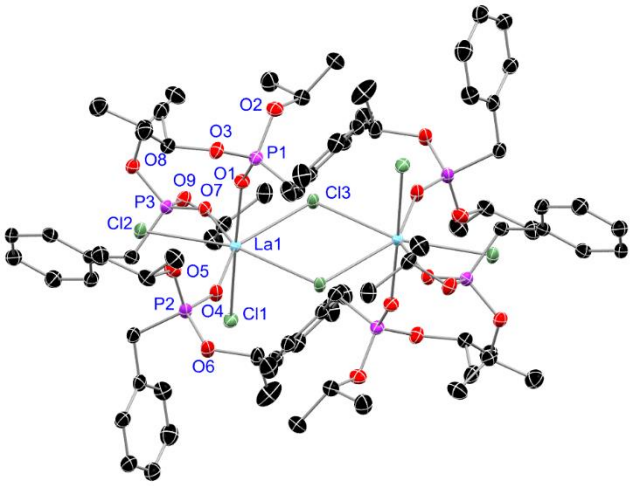

Figure 44: Asymmetric unit of **5**.

5 crystalizes in the space group $P\bar{1}$. The asymmetric unit contains half of the dimer. The other half is generated by the inversion center.

Bond length [Å] and angles [°] of **5**.

C(1)–C(2)	1.511(4)	C(18)–C(19)	1.391(5)
C(1)–P(1)	1.790(3)	C(19)–C(20)	1.386(4)
C(2)–C(3)	1.389(4)	C(21)–C(22)	1.520(4)
C(2)–C(7)	1.384(4)	C(21)–C(23)	1.503(4)
C(3)–C(4)	1.391(4)	C(21)–O(5)	1.475(3)
C(4)–C(5)	1.382(4)	C(24)–C(25)	1.501(5)
C(5)–C(6)	1.385(4)	C(24)–C(26)	1.505(4)
C(6)–C(7)	1.392(4)	C(24)–O(6)	1.479(3)
C(8)–C(9)	1.511(4)	C(27)–C(28)	1.518(3)
C(8)–C(10)	1.513(4)	C(27)–P(3)	1.792(3)
C(8)–O(2)	1.483(3)	C(28)–C(29)	1.396(4)
C(11)–C(12)	1.509(4)	C(28)–C(33)	1.385(4)
C(11)–C(13)	1.513(4)	C(29)–C(30)	1.391(4)
C(11)–O(3)	1.481(3)	C(30)–C(31)	1.380(4)
C(14)–C(15)	1.518(4)	C(31)–C(32)	1.383(4)
C(14)–P(2)	1.789(3)	C(32)–C(33)	1.388(4)
C(15)–C(16)	1.386(4)	C(34)–C(35)	1.504(4)
C(15)–C(20)	1.388(4)	C(34)–C(36)	1.506(4)
C(16)–C(17)	1.392(4)	C(34)–O(8)	1.470(3)
C(17)–C(18)	1.380(5)	C(37)–C(38)	1.510(4)

C(37)–C(39)	1.514(4)
C(37)–O(9)	1.475(3)
Cl(1)–La(1)	2.7807(6)
Cl(2)–La(1)	2.8596(6)
Cl(3)–La(1)	2.9034(6)
Cl(3)–La(1)#1	2.9450(5)
La(1)–O(1)	2.4061(17)
La(1)–O(4)	2.4374(17)
La(1)–O(7)	2.4574(16)
O(1)–P(1)	1.4859(18)
O(2)–P(1)	1.5678(19)
O(3)–P(1)	1.5621(19)
O(4)–P(2)	1.4873(18)
O(5)–P(2)	1.573(2)
O(6)–P(2)	1.5639(18)
O(7)–P(3)	1.4860(17)
O(8)–P(3)	1.5677(18)
O(9)–P(3)	1.5681(19)

C(2)–C(1)–P(1)	115.89(18)
C(3)–C(2)–C(1)	120.1(2)
C(7)–C(2)–C(1)	120.8(2)
C(7)–C(2)–C(3)	119.0(2)
C(2)–C(3)–C(4)	120.3(3)
C(5)–C(4)–C(3)	120.5(3)
C(4)–C(5)–C(6)	119.4(3)
C(5)–C(6)–C(7)	120.1(3)
C(2)–C(7)–C(6)	120.7(3)
C(9)–C(8)–C(10)	113.3(2)
O(2)–C(8)–C(9)	108.7(2)
O(2)–C(8)–C(10)	105.8(2)
C(12)–C(11)–C(13)	111.8(2)
O(3)–C(11)–C(12)	111.5(2)
O(3)–C(11)–C(13)	104.8(2)
C(15)–C(14)–P(2)	113.29(18)
C(16)–C(15)–C(14)	122.0(2)
C(16)–C(15)–C(20)	119.1(3)
C(20)–C(15)–C(14)	118.9(2)
C(15)–C(16)–C(17)	120.2(3)
C(18)–C(17)–C(16)	120.7(3)
C(17)–C(18)–C(19)	119.2(3)
C(20)–C(19)–C(18)	120.2(3)
C(19)–C(20)–C(15)	120.6(3)
C(23)–C(21)–C(22)	113.6(2)
O(5)–C(21)–C(22)	108.1(2)
O(5)–C(21)–C(23)	107.6(2)
C(25)–C(24)–C(26)	114.1(3)
O(6)–C(24)–C(25)	107.8(3)
O(6)–C(24)–C(26)	106.1(2)
C(28)–C(27)–P(3)	116.21(18)
C(29)–C(28)–C(27)	120.8(2)
C(33)–C(28)–C(27)	120.3(2)
C(33)–C(28)–C(29)	118.9(2)
C(30)–C(29)–C(28)	120.3(3)
C(31)–C(30)–C(29)	120.1(3)
C(30)–C(31)–C(32)	119.9(3)
C(31)–C(32)–C(33)	120.1(3)
C(28)–C(33)–C(32)	120.7(3)
C(35)–C(34)–C(36)	113.9(2)
O(8)–C(34)–C(35)	105.3(2)
O(8)–C(34)–C(36)	109.5(2)
C(38)–C(37)–C(39)	113.3(2)
O(9)–C(37)–C(38)	107.8(2)
O(9)–C(37)–C(39)	106.8(2)
La(1)–Cl(3)–La(1)#1	108.870(17)
Cl(1)–La(1)–Cl(2)	100.441(18)
Cl(1)–La(1)–Cl(3)	97.659(18)
Cl(1)–La(1)–Cl(3)#1	89.263(17)
Cl(2)–La(1)–Cl(3)#1	144.525(16)
Cl(2)–La(1)–Cl(3)	139.395(16)
Cl(3)–La(1)–Cl(3)#1	71.128(17)
P(1)–O(1)–La(1)	153.76(11)
C(8)–O(2)–P(1)	122.37(16)
C(11)–O(3)–P(1)	123.98(17)
P(2)–O(4)–La(1)	154.78(10)
C(21)–O(5)–P(2)	121.30(16)
C(24)–O(6)–P(2)	122.63(16)
P(3)–O(7)–La(1)	145.16(10)

C(34)–O(8)–P(3)	122.33(15)	O(5)–P(2)–C(14)	108.20(11)
C(37)–O(9)–P(3)	119.44(16)	O(6)–P(2)–C(14)	104.21(11)
O(1)–P(1)–C(1)	110.56(11)	O(6)–P(2)–O(5)	107.68(11)
O(1)–P(1)–O(2)	112.27(10)	O(7)–P(3)–C(27)	113.30(11)
O(1)–P(1)–O(3)	115.00(10)	O(7)–P(3)–O(8)	114.32(10)
O(2)–P(1)–C(1)	110.07(12)	O(7)–P(3)–O(9)	112.02(10)
O(3)–P(1)–C(1)	104.81(12)	O(8)–P(3)–C(27)	105.19(11)
O(3)–P(1)–O(2)	103.68(10)	O(8)–P(3)–O(9)	103.24(10)
O(4)–P(2)–C(14)	114.44(12)	O(9)–P(3)–C(27)	108.02(12)
O(4)–P(2)–O(5)	108.77(10)		
O(4)–P(2)–O(6)	113.23(10)		

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) sv0743

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No syntax errors found. CIF dictionary Interpreting this report

Datablock: sv0743

```
Bond precision:  C-C = 0.0045 A          Wavelength=1.54186

Cell:          a=13.9287(4)      b=13.9984(4)      c=14.8205(4)
               alpha=91.100(2)   beta=104.058(2)   gamma=119.094(2)
Temperature:   100 K


                 Calculated                 Reported
Volume           2417.11(13)               2417.10(13)
Space group      P -1                       P -1
Hall group       -P 1                       -P 1
Moiety formula   C78 H126 Cl6 La2 O18 P6    C78 H126
Cl6 La2 O18 P6
Sum formula      C78 H126 Cl6 La2 O18 P6    C78 H126
Cl6 La2 O18 P6
Mr               2028.13                    2028.12
Dx,g cm-3        1.393                      1.393
Z                1                          1
Mu (mm-1)        9.676                      9.676
F000             1044.0                     1044.0
F000'            1047.65
h,k,lmax         17,17,18                   16,17,17
Nref             9409                       9025
```

Tmin,Tmax 0.373,0.679 0.177,0.547
Tmin' 0.202

Correction method= # Reported T Limits: Tmin=0.177 Tmax=0.547
AbsCorr = MULTI-SCAN

Data completeness= 0.959 Theta(max)= 71.350

R(reflections)= 0.0272(8807) wR2(reflections)= 0.0755(9025)

S = 1.078 Npar= 508

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level C

PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L= 0.600 138 Report
PLAT918_ALERT_3_C Reflection(s) with I(obs) much Smaller I(calc) . 1 Check
PLAT978_ALERT_2_C Number C-C Bonds with Positive Residual Density. 0 Info

---

Alert level G

PLAT154_ALERT_1_G The s.u.'s on the Cell Angles are Equal ..(Note) 0.002 Degree
PLAT232_ALERT_2_G Hirshfeld Test Diff (M-X) La1 --Cl1 . 5.3 s.u.
PLAT232_ALERT_2_G Hirshfeld Test Diff (M-X) La1 --Cl2 . 5.3 s.u.
PLAT232_ALERT_2_G Hirshfeld Test Diff (M-X) La1 --Cl3 . 5.7 s.u.
PLAT794_ALERT_5_G Tentative Bond Valency for La1 (III) . 3.16 Info
PLAT883_ALERT_1_G No Info/Value for _atom_sites_solution_primary . Please Do!
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L= 0.600 217 Note
PLAT933_ALERT_2_G Number of OMIT Records in Embedded .res File ... 12 Note

2.6 Complex 10

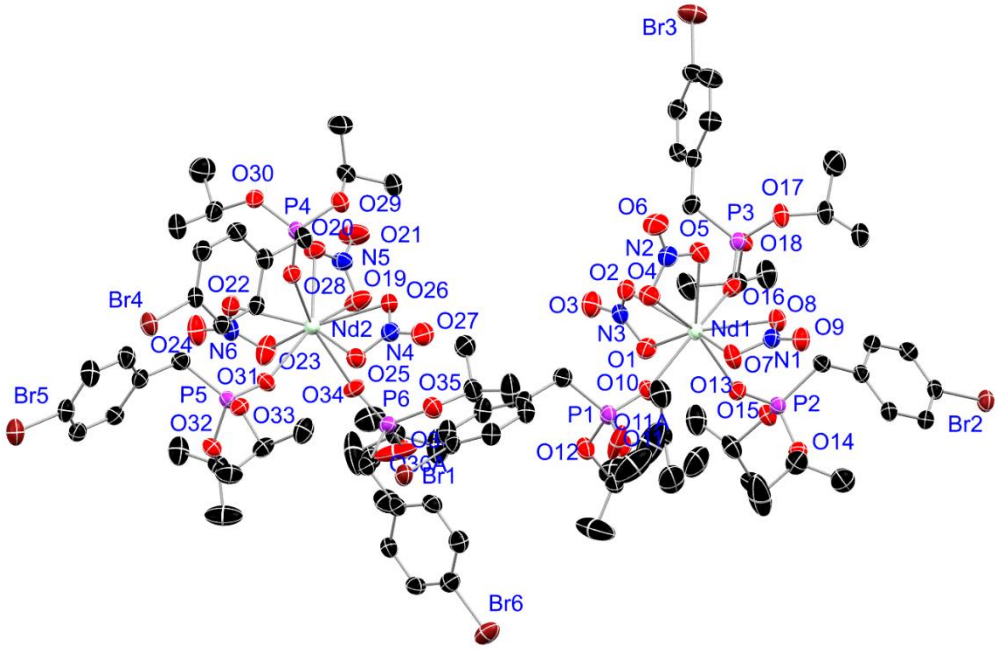

Figure 45: Asymmetric unit of **10**.

10 crystalizes in the space group $P\bar{1}$ and contains two molecules in the asymmetric unit. Two of the twelve *i*Pr residues are disordered about two positions also including their bridging O-atom. They are refined with distance restraints and partially with restraints for the anisotropic displacement parameters. The occupancy of the minor positions refined to 0.487(10) and 0.424(9), respectively. Just like in **L6**, halogen bonds are formed. In this case between the bromide Br2 and the outwards titled N=O (O9) oxygen atoms of a NO_3^- anion of adjacent molecules. But in contrast to **L6**, there is less lack of electron density at the bromides. Thus, the O···Br distance of 3.06 Å is longer, but still shorter than the sum of the van der Waals radii of both atoms (3.35 Å), still causing a B-Alert upon cif-file check.

Bond lengths [Å] and angles [°] of **10.**

C(1)–C(2)	1.379(5)	C(11)–O(12)	1.479(5)
C(1)–C(6)	1.389(5)	C(14)–C(15)	1.383(5)
C(2)–C(3)	1.387(5)	C(14)–C(19)	1.401(5)
C(3)–C(4)	1.379(5)	C(15)–C(16)	1.386(5)
C(3)–Br(1)	1.892(4)	C(16)–C(17)	1.377(5)
C(4)–C(5)	1.388(5)	C(16)–Br(2)	1.901(3)
C(5)–C(6)	1.384(5)	C(17)–C(18)	1.387(5)
C(6)–C(7)	1.514(5)	C(18)–C(19)	1.391(5)
C(7)–P(1)	1.787(4)	C(19)–C(20)	1.512(5)
C(11)–C(12)	1.490(7)	C(20)–P(2)	1.793(3)
C(11)–C(13)	1.489(7)	C(21)–C(22)	1.497(6)

C(21)–C(23)	1.488(7)	C(55)–Br(5)	1.900(4)
C(21)–O(14)	1.476(4)	C(56)–C(57)	1.384(6)
C(24)–C(25)	1.502(6)	C(57)–C(58)	1.384(5)
C(24)–C(26)	1.496(7)	C(58)–C(59)	1.508(5)
C(24)–O(15)	1.476(4)	C(59)–P(5)	1.786(4)
C(27)–C(28)	1.384(6)	C(60)–C(61)	1.504(6)
C(27)–C(32)	1.385(5)	C(60)–C(62)	1.507(5)
C(28)–C(29)	1.382(6)	C(60)–O(33)	1.468(4)
C(29)–C(30)	1.374(5)	C(63)–C(64)	1.502(5)
C(29)–Br(3)	1.901(4)	C(63)–C(65)	1.501(6)
C(30)–C(31)	1.387(5)	C(63)–O(32)	1.474(4)
C(31)–C(32)	1.391(5)	C(66)–C(67)	1.387(5)
C(32)–C(33)	1.506(5)	C(66)–C(71)	1.384(5)
C(33)–P(3)	1.787(4)	C(67)–C(68)	1.379(5)
C(34)–C(35)	1.500(5)	C(68)–C(69)	1.381(6)
C(34)–C(36)	1.503(5)	C(68)–Br(6)	1.894(4)
C(34)–O(18)	1.465(4)	C(69)–C(70)	1.386(5)
C(37)–C(38)	1.505(5)	C(70)–C(71)	1.385(5)
C(37)–C(39)	1.500(6)	C(71)–C(72)	1.505(5)
C(37)–O(17)	1.478(4)	C(72)–P(6)	1.786(4)
C(40)–C(41)	1.388(5)	C(73)–C(74)	1.487(6)
C(40)–C(45)	1.392(5)	C(73)–C(75)	1.475(6)
C(41)–C(42)	1.381(5)	C(73)–O(35)	1.465(5)
C(42)–C(43)	1.386(5)	N(1)–O(7)	1.271(4)
C(42)–Br(4)	1.899(3)	N(1)–O(8)	1.268(4)
C(43)–C(44)	1.383(5)	N(1)–O(9)	1.212(4)
C(44)–C(45)	1.394(5)	N(2)–O(4)	1.268(4)
C(45)–C(46)	1.502(5)	N(2)–O(5)	1.281(4)
C(46)–P(4)	1.791(3)	N(2)–O(6)	1.213(4)
C(47)–C(48)	1.504(6)	N(3)–O(1)	1.273(4)
C(47)–C(49)	1.502(6)	N(3)–O(2)	1.272(4)
C(47)–O(30)	1.479(4)	N(3)–O(3)	1.216(4)
C(50)–C(51)	1.501(5)	N(4)–O(25)	1.264(4)
C(50)–C(52)	1.513(5)	N(4)–O(26)	1.277(4)
C(50)–O(29)	1.475(4)	N(4)–O(27)	1.218(4)
C(53)–C(54)	1.390(5)	N(5)–O(19)	1.258(4)
C(53)–C(58)	1.393(5)	N(5)–O(20)	1.262(4)
C(54)–C(55)	1.387(5)	N(5)–O(21)	1.223(4)
C(55)–C(56)	1.367(6)	N(6)–O(22)	1.261(4)

N(6)–O(23)	1.266(4)	C(8)–C(9)	1.541(12)
N(6)–O(24)	1.218(4)	C(8)–C(10)	1.487(12)
Nd(1)–O(1)	2.577(2)	O(11A)–C(8A)	1.383(13)
Nd(1)–O(2)	2.521(2)	C(8A)–C(9A)	1.490(17)
Nd(1)–O(4)	2.566(2)	C(8A)–C(10A)	1.496(16)
Nd(1)–O(5)	2.490(2)	P(6)–O(36)	1.559(6)
Nd(1)–O(7)	2.511(2)	P(6)–O(36A)	1.529(10)
Nd(1)–O(8)	2.556(2)	O(36)–C(76)	1.418(9)
Nd(1)–O(10)	2.373(2)	C(76)–C(77)	1.487(9)
Nd(1)–O(13)	2.384(2)	C(76)–C(78)	1.469(11)
Nd(1)–O(16)	2.424(2)	O(36A)–C(76A)	1.383(14)
Nd(2)–O(19)	2.548(3)	C(76A)–C(77A)	1.473(14)
Nd(2)–O(20)	2.536(3)	C(76A)–C(78A)	1.485(12)
Nd(2)–O(22)	2.554(2)		
Nd(2)–O(23)	2.516(3)	C(2)–C(1)–C(6)	121.3(3)
Nd(2)–O(25)	2.542(2)	C(1)–C(2)–C(3)	118.9(3)
Nd(2)–O(26)	2.536(2)	C(2)–C(3)–Br(1)	119.0(3)
Nd(2)–O(28)	2.396(2)	C(4)–C(3)–C(2)	121.1(3)
Nd(2)–O(31)	2.381(2)	C(4)–C(3)–Br(1)	119.9(3)
Nd(2)–O(34)	2.375(2)	C(3)–C(4)–C(5)	119.0(3)
O(10)–P(1)	1.477(2)	C(6)–C(5)–C(4)	121.0(3)
O(12)–P(1)	1.569(3)	C(1)–C(6)–C(7)	120.3(3)
O(13)–P(2)	1.487(2)	C(5)–C(6)–C(1)	118.6(3)
O(14)–P(2)	1.565(3)	C(5)–C(6)–C(7)	121.1(3)
O(15)–P(2)	1.558(2)	C(6)–C(7)–P(1)	114.5(3)
O(16)–P(3)	1.485(3)	C(13)–C(11)–C(12)	113.9(5)
O(17)–P(3)	1.558(2)	O(12)–C(11)–C(12)	106.7(4)
O(18)–P(3)	1.570(2)	O(12)–C(11)–C(13)	108.7(4)
O(28)–P(4)	1.487(2)	C(15)–C(14)–C(19)	121.5(3)
O(29)–P(4)	1.561(2)	C(14)–C(15)–C(16)	118.6(3)
O(30)–P(4)	1.562(3)	C(15)–C(16)–Br(2)	119.4(3)
O(31)–P(5)	1.483(2)	C(17)–C(16)–C(15)	121.3(3)
O(32)–P(5)	1.562(2)	C(17)–C(16)–Br(2)	119.2(3)
O(33)–P(5)	1.563(2)	C(16)–C(17)–C(18)	119.5(3)
O(34)–P(6)	1.476(3)	C(17)–C(18)–C(19)	120.9(3)
O(35)–P(6)	1.570(3)	C(14)–C(19)–C(20)	119.8(3)
P(1)–O(11)	1.557(8)	C(18)–C(19)–C(14)	118.2(3)
P(1)–O(11A)	1.549(10)	C(18)–C(19)–C(20)	121.9(3)
O(11)–C(8)	1.458(9)	C(19)–C(20)–P(2)	115.6(2)

C(23)–C(21)–C(22)	113.2(4)	O(29)–C(50)–C(52)	106.2(3)
O(14)–C(21)–C(22)	108.1(3)	C(54)–C(53)–C(58)	121.3(3)
O(14)–C(21)–C(23)	107.6(4)	C(55)–C(54)–C(53)	118.3(3)
C(26)–C(24)–C(25)	114.1(4)	C(54)–C(55)–Br(5)	119.1(3)
O(15)–C(24)–C(25)	106.6(3)	C(56)–C(55)–C(54)	121.5(4)
O(15)–C(24)–C(26)	107.6(3)	C(56)–C(55)–Br(5)	119.4(3)
C(28)–C(27)–C(32)	120.7(4)	C(55)–C(56)–C(57)	119.3(4)
C(29)–C(28)–C(27)	118.9(4)	C(58)–C(57)–C(56)	121.3(4)
C(28)–C(29)–Br(3)	119.2(3)	C(53)–C(58)–C(59)	121.2(3)
C(30)–C(29)–C(28)	122.1(4)	C(57)–C(58)–C(53)	118.2(3)
C(30)–C(29)–Br(3)	118.8(3)	C(57)–C(58)–C(59)	120.6(3)
C(29)–C(30)–C(31)	118.1(3)	C(58)–C(59)–P(5)	114.5(2)
C(30)–C(31)–C(32)	121.4(3)	C(61)–C(60)–C(62)	114.5(3)
C(27)–C(32)–C(31)	118.7(3)	O(33)–C(60)–C(61)	106.2(3)
C(27)–C(32)–C(33)	121.1(3)	O(33)–C(60)–C(62)	107.0(3)
C(31)–C(32)–C(33)	120.2(3)	C(65)–C(63)–C(64)	114.4(4)
C(32)–C(33)–P(3)	114.8(2)	O(32)–C(63)–C(64)	109.8(3)
C(35)–C(34)–C(36)	112.9(3)	O(32)–C(63)–C(65)	104.9(3)
O(18)–C(34)–C(35)	108.7(3)	C(71)–C(66)–C(67)	121.2(3)
O(18)–C(34)–C(36)	105.6(3)	C(68)–C(67)–C(66)	118.7(3)
C(39)–C(37)–C(38)	113.5(3)	C(67)–C(68)–C(69)	121.2(3)
O(17)–C(37)–C(38)	107.8(3)	C(67)–C(68)–Br(6)	120.0(3)
O(17)–C(37)–C(39)	107.0(3)	C(69)–C(68)–Br(6)	118.8(3)
C(41)–C(40)–C(45)	120.9(3)	C(68)–C(69)–C(70)	119.3(3)
C(42)–C(41)–C(40)	118.7(3)	C(71)–C(70)–C(69)	120.6(3)
C(41)–C(42)–C(43)	121.5(3)	C(66)–C(71)–C(70)	119.0(3)
C(41)–C(42)–Br(4)	119.1(3)	C(66)–C(71)–C(72)	120.8(3)
C(43)–C(42)–Br(4)	119.4(3)	C(70)–C(71)–C(72)	120.2(3)
C(44)–C(43)–C(42)	119.2(3)	C(71)–C(72)–P(6)	116.0(2)
C(43)–C(44)–C(45)	120.5(3)	C(75)–C(73)–C(74)	114.2(4)
C(40)–C(45)–C(44)	119.1(3)	O(35)–C(73)–C(74)	106.4(3)
C(40)–C(45)–C(46)	120.0(3)	O(35)–C(73)–C(75)	112.4(3)
C(44)–C(45)–C(46)	120.9(3)	O(7)–N(1)–Nd(1)	56.88(16)
C(45)–C(46)–P(4)	116.6(2)	O(8)–N(1)–Nd(1)	58.90(15)
C(49)–C(47)–C(48)	113.6(4)	O(8)–N(1)–O(7)	115.5(3)
O(30)–C(47)–C(48)	106.0(3)	O(9)–N(1)–Nd(1)	175.3(2)
O(30)–C(47)–C(49)	108.3(3)	O(9)–N(1)–O(7)	122.1(3)
C(51)–C(50)–C(52)	113.7(3)	O(9)–N(1)–O(8)	122.3(3)
O(29)–C(50)–C(51)	108.2(3)	O(4)–N(2)–Nd(1)	59.60(16)

O(4)–N(2)–O(5)	115.7(3)	O(2)–Nd(1)–O(4)	69.75(8)
O(5)–N(2)–Nd(1)	56.23(16)	O(2)–Nd(1)–O(8)	144.38(8)
O(6)–N(2)–Nd(1)	175.9(3)	O(4)–Nd(1)–N(1)	86.32(8)
O(6)–N(2)–O(4)	122.9(3)	O(4)–Nd(1)–N(2)	25.24(8)
O(6)–N(2)–O(5)	121.4(3)	O(4)–Nd(1)–N(3)	91.87(8)
O(1)–N(3)–Nd(1)	59.23(16)	O(4)–Nd(1)–O(1)	112.93(8)
O(2)–N(3)–Nd(1)	56.71(16)	O(5)–Nd(1)–N(1)	77.22(8)
O(2)–N(3)–O(1)	115.8(3)	O(5)–Nd(1)–N(2)	25.32(8)
O(3)–N(3)–Nd(1)	174.4(2)	O(5)–Nd(1)–N(3)	98.11(9)
O(3)–N(3)–O(1)	122.6(3)	O(5)–Nd(1)–O(1)	121.77(9)
O(3)–N(3)–O(2)	121.6(3)	O(5)–Nd(1)–O(2)	75.21(8)
O(25)–N(4)–Nd(2)	58.35(17)	O(5)–Nd(1)–O(4)	50.52(8)
O(25)–N(4)–O(26)	116.5(3)	O(5)–Nd(1)–O(7)	85.60(9)
O(26)–N(4)–Nd(2)	58.13(16)	O(5)–Nd(1)–O(8)	73.52(9)
O(27)–N(4)–Nd(2)	179.6(3)	O(7)–Nd(1)–N(1)	25.08(8)
O(27)–N(4)–O(25)	122.1(4)	O(7)–Nd(1)–N(2)	79.27(9)
O(27)–N(4)–O(26)	121.4(3)	O(7)–Nd(1)–N(3)	157.45(8)
O(19)–N(5)–Nd(2)	58.66(17)	O(7)–Nd(1)–O(1)	149.92(9)
O(19)–N(5)–O(20)	116.7(3)	O(7)–Nd(1)–O(2)	142.62(8)
O(20)–N(5)–Nd(2)	58.11(17)	O(7)–Nd(1)–O(4)	73.26(9)
O(21)–N(5)–Nd(2)	178.6(3)	O(7)–Nd(1)–O(8)	50.15(8)
O(21)–N(5)–O(19)	121.4(3)	O(8)–Nd(1)–N(1)	25.14(8)
O(21)–N(5)–O(20)	121.9(3)	O(8)–Nd(1)–N(2)	88.36(8)
O(22)–N(6)–Nd(2)	58.83(16)	O(8)–Nd(1)–N(3)	152.11(8)
O(22)–N(6)–O(23)	116.0(3)	O(8)–Nd(1)–O(1)	143.70(8)
O(23)–N(6)–Nd(2)	57.13(17)	O(8)–Nd(1)–O(4)	101.94(8)
O(24)–N(6)–Nd(2)	178.6(3)	O(10)–Nd(1)–N(1)	100.23(8)
O(24)–N(6)–O(22)	122.4(3)	O(10)–Nd(1)–N(2)	100.25(9)
O(24)–N(6)–O(23)	121.6(3)	O(10)–Nd(1)–N(3)	83.69(8)
N(1)–Nd(1)–N(3)	175.09(8)	O(10)–Nd(1)–O(1)	77.27(9)
N(2)–Nd(1)–N(1)	81.86(8)	O(10)–Nd(1)–O(2)	89.13(9)
N(2)–Nd(1)–N(3)	94.57(8)	O(10)–Nd(1)–O(4)	75.01(8)
O(1)–Nd(1)–N(1)	158.64(8)	O(10)–Nd(1)–O(5)	125.51(8)
O(1)–Nd(1)–N(2)	119.49(8)	O(10)–Nd(1)–O(7)	76.22(8)
O(1)–Nd(1)–N(3)	25.12(8)	O(10)–Nd(1)–O(8)	123.12(8)
O(2)–Nd(1)–N(1)	151.20(8)	O(10)–Nd(1)–O(13)	84.41(8)
O(2)–Nd(1)–N(2)	69.64(8)	O(10)–Nd(1)–O(16)	152.15(8)
O(2)–Nd(1)–N(3)	24.94(8)	O(13)–Nd(1)–N(1)	80.79(8)
O(2)–Nd(1)–O(1)	50.02(8)	O(13)–Nd(1)–N(2)	162.58(8)

O(13)–Nd(1)–N(3)	102.65(8)	O(23)–Nd(2)–O(25)	142.17(10)
O(13)–Nd(1)–O(1)	77.86(8)	O(23)–Nd(2)–O(26)	147.32(9)
O(13)–Nd(1)–O(2)	127.51(8)	O(25)–Nd(2)–N(4)	25.03(9)
O(13)–Nd(1)–O(4)	153.35(9)	O(25)–Nd(2)–N(5)	129.15(9)
O(13)–Nd(1)–O(5)	145.39(8)	O(25)–Nd(2)–N(6)	149.08(8)
O(13)–Nd(1)–O(7)	85.65(9)	O(25)–Nd(2)–O(19)	117.19(10)
O(13)–Nd(1)–O(8)	75.20(8)	O(25)–Nd(2)–O(22)	139.98(8)
O(13)–Nd(1)–O(16)	81.00(8)	O(26)–Nd(2)–N(4)	25.32(9)
O(16)–Nd(1)–N(1)	100.67(8)	O(26)–Nd(2)–N(5)	79.27(9)
O(16)–Nd(1)–N(2)	100.75(8)	O(26)–Nd(2)–N(6)	160.55(9)
O(16)–Nd(1)–N(3)	76.60(8)	O(26)–Nd(2)–O(19)	74.71(11)
O(16)–Nd(1)–O(1)	76.55(8)	O(26)–Nd(2)–O(20)	84.47(9)
O(16)–Nd(1)–O(2)	81.27(8)	O(26)–Nd(2)–O(22)	150.13(8)
O(16)–Nd(1)–O(4)	124.53(8)	O(26)–Nd(2)–O(25)	50.35(8)
O(16)–Nd(1)–O(5)	77.25(8)	O(28)–Nd(2)–N(4)	76.51(8)
O(16)–Nd(1)–O(7)	125.74(8)	O(28)–Nd(2)–N(5)	99.86(8)
O(16)–Nd(1)–O(8)	75.60(8)	O(28)–Nd(2)–N(6)	103.85(9)
N(4)–Nd(2)–N(6)	174.11(9)	O(28)–Nd(2)–O(19)	121.47(9)
N(5)–Nd(2)–N(4)	104.39(9)	O(28)–Nd(2)–O(20)	77.12(8)
N(5)–Nd(2)–N(6)	81.39(9)	O(28)–Nd(2)–O(22)	78.89(8)
O(19)–Nd(2)–N(4)	96.30(11)	O(28)–Nd(2)–O(23)	128.81(9)
O(19)–Nd(2)–N(5)	24.93(9)	O(28)–Nd(2)–O(25)	78.17(8)
O(19)–Nd(2)–N(6)	88.52(10)	O(28)–Nd(2)–O(26)	77.61(8)
O(19)–Nd(2)–O(22)	102.73(10)	O(31)–Nd(2)–N(4)	96.52(9)
O(20)–Nd(2)–N(4)	108.61(9)	O(31)–Nd(2)–N(5)	158.78(9)
O(20)–Nd(2)–N(5)	25.00(8)	O(31)–Nd(2)–N(6)	77.64(8)
O(20)–Nd(2)–N(6)	77.10(9)	O(31)–Nd(2)–O(19)	149.56(10)
O(20)–Nd(2)–O(19)	49.91(8)	O(31)–Nd(2)–O(20)	146.77(9)
O(20)–Nd(2)–O(22)	72.49(9)	O(31)–Nd(2)–O(22)	75.47(9)
O(20)–Nd(2)–O(25)	132.11(8)	O(31)–Nd(2)–O(23)	82.19(10)
O(22)–Nd(2)–N(4)	154.33(9)	O(31)–Nd(2)–O(25)	71.55(8)
O(22)–Nd(2)–N(5)	86.89(9)	O(31)–Nd(2)–O(26)	121.78(9)
O(22)–Nd(2)–N(6)	25.00(9)	O(31)–Nd(2)–O(28)	88.39(8)
O(23)–Nd(2)–N(4)	154.40(9)	O(34)–Nd(2)–N(4)	78.20(9)
O(23)–Nd(2)–N(5)	77.40(10)	O(34)–Nd(2)–N(5)	94.23(9)
O(23)–Nd(2)–N(6)	25.00(9)	O(34)–Nd(2)–N(6)	100.46(9)
O(23)–Nd(2)–O(19)	74.45(12)	O(34)–Nd(2)–O(19)	69.41(9)
O(23)–Nd(2)–O(20)	83.98(10)	O(34)–Nd(2)–O(20)	119.21(8)
O(23)–Nd(2)–O(22)	50.00(9)	O(34)–Nd(2)–O(22)	124.60(9)

O(34)–Nd(2)–O(23)	76.20(9)	O(11)–P(1)–O(12)	103.5(4)
O(34)–Nd(2)–O(25)	75.45(9)	O(11A)–P(1)–C(7)	103.3(7)
O(34)–Nd(2)–O(26)	83.06(9)	O(11A)–P(1)–O(12)	106.9(6)
O(34)–Nd(2)–O(28)	153.41(8)	C(8)–O(11)–P(1)	122.1(8)
O(34)–Nd(2)–O(31)	86.42(9)	O(11)–C(8)–C(9)	115.9(8)
N(3)–O(1)–Nd(1)	95.64(18)	O(11)–C(8)–C(10)	103.2(8)
N(3)–O(2)–Nd(1)	98.35(19)	C(10)–C(8)–C(9)	113.4(8)
N(2)–O(4)–Nd(1)	95.16(18)	C(8A)–O(11A)–P(1)	136.1(13)
N(2)–O(5)–Nd(1)	98.5(2)	O(11A)–C(8A)–C(9A)	116.3(15)
N(1)–O(7)–Nd(1)	98.04(19)	O(11A)–C(8A)–C(10A)	108.9(13)
N(1)–O(8)–Nd(1)	95.97(18)	C(9A)–C(8A)–C(10A)	114.9(13)
P(1)–O(10)–Nd(1)	161.58(16)	O(13)–P(2)–C(20)	112.73(15)
C(11)–O(12)–P(1)	118.9(2)	O(13)–P(2)–O(14)	111.43(14)
P(2)–O(13)–Nd(1)	148.99(15)	O(13)–P(2)–O(15)	114.65(14)
C(21)–O(14)–P(2)	121.9(2)	O(14)–P(2)–C(20)	108.96(15)
C(24)–O(15)–P(2)	121.4(2)	O(15)–P(2)–C(20)	103.40(15)
P(3)–O(16)–Nd(1)	145.06(14)	O(15)–P(2)–O(14)	105.07(14)
C(37)–O(17)–P(3)	126.2(2)	O(16)–P(3)–C(33)	111.58(15)
C(34)–O(18)–P(3)	125.8(2)	O(16)–P(3)–O(17)	114.17(15)
N(5)–O(19)–Nd(2)	96.4(2)	O(16)–P(3)–O(18)	113.15(13)
N(5)–O(20)–Nd(2)	96.89(19)	O(17)–P(3)–C(33)	104.24(16)
N(6)–O(22)–Nd(2)	96.2(2)	O(17)–P(3)–O(18)	104.82(13)
N(6)–O(23)–Nd(2)	97.9(2)	O(18)–P(3)–C(33)	108.24(16)
N(4)–O(25)–Nd(2)	96.6(2)	O(28)–P(4)–C(46)	114.08(16)
N(4)–O(26)–Nd(2)	96.55(19)	O(28)–P(4)–O(29)	115.55(14)
P(4)–O(28)–Nd(2)	153.70(15)	O(28)–P(4)–O(30)	111.56(13)
C(50)–O(29)–P(4)	124.7(2)	O(29)–P(4)–C(46)	99.71(15)
C(47)–O(30)–P(4)	124.1(2)	O(29)–P(4)–O(30)	106.75(14)
P(5)–O(31)–Nd(2)	156.74(16)	O(30)–P(4)–C(46)	108.28(16)
C(63)–O(32)–P(5)	124.1(2)	O(31)–P(5)–C(59)	112.61(16)
C(60)–O(33)–P(5)	123.2(2)	O(31)–P(5)–O(32)	111.60(14)
P(6)–O(34)–Nd(2)	148.02(16)	O(31)–P(5)–O(33)	115.00(14)
C(73)–O(35)–P(6)	125.9(3)	O(32)–P(5)–C(59)	110.02(15)
O(10)–P(1)–C(7)	112.74(17)	O(32)–P(5)–O(33)	103.79(13)
O(10)–P(1)–O(12)	112.72(15)	O(33)–P(5)–C(59)	103.17(15)
O(10)–P(1)–O(11)	110.7(5)	O(34)–P(6)–C(72)	110.86(16)
O(10)–P(1)–O(11A)	116.5(6)	O(34)–P(6)–O(35)	113.24(16)
O(12)–P(1)–C(7)	103.41(16)	O(34)–P(6)–O(36)	114.3(3)
O(11)–P(1)–C(7)	113.2(5)	O(34)–P(6)–O(36A)	110.7(6)

O(35)–P(6)–C(72)	103.25(16)	O(36)–C(76)–C(78)	115.1(9)
O(36)–P(6)–C(72)	109.5(4)	C(78)–C(76)–C(77)	112.5(7)
O(36)–P(6)–O(35)	105.0(2)	C(76A)–O(36A)–P(6)	134.2(12)
O(36A)–P(6)–C(72)	104.1(7)	O(36A)–C(76A)–C(77A)	116.9(12)
O(36A)–P(6)–O(35)	114.0(7)	O(36A)–C(76A)–C(78A)	107.5(10)
C(76)–O(36)–P(6)	125.7(7)	C(77A)–C(76A)–C(78A)	113.4(9)
O(36)–C(76)–C(77)	103.8(6)		

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) sv0709

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: sv0709

Bond precision: C-C = 0.0062 A Wavelength=1.54186

Cell:	a=16.4380(3)	b=20.2226(3)	c=20.3926(3)
	alpha=61.933(1)	beta=70.494(1)	gamma=67.900(1)
Temperature:	100 K		

	Calculated	Reported
Volume	5439.19(16)	5439.20(16)
Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	C39 H60 Br3 N3 Nd O18 P3	0.5(C78 H120 Br6 N6 Nd2 O36 P6)
Sum formula	C39 H60 Br3 N3 Nd O18 P3	C39 H60 Br3 N3 Nd O18 P3
Mr	1335.75	1335.78
Dx,g cm-3	1.631	1.631
Z	4	4
Mu (mm-1)	11.279	11.279
F000	2676.0	2676.0
F000’	2665.90	
h,k,lmax	19,24,24	19,24,24
Nref	20414	19710
Tmin,Tmax	0.075,0.133	0.043,0.217
Tmin’	0.005	

Correction method= # Reported T Limits: Tmin=0.043 Tmax=0.217 AbsCorr = MULTI-SCAN

Data completeness= 0.966 *Theta(max)= 69.474*

R(reflections)= 0.0341(18960) *wR2(reflections)= 0.0902(19710)*

S = 1.039 *Npar= 1309*

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.

Click on the hyperlinks for more details of the test.

Alert level B

PLAT431_ALERT_2_B Short Inter HL..A Contact Br2 ..O9 . 3.06 Ang.
2-x,-y,2-z = 2_757 Check

Alert level C

PLAT220_ALERT_2_C	NonSolvent	Resd	1	C	Ueq(max) / Ueq(min)	Range	4.0	Ratio
PLAT220_ALERT_2_C	NonSolvent	Resd	2	C	Ueq(max) / Ueq(min)	Range	3.5	Ratio
PLAT220_ALERT_2_C	NonSolvent	Resd	2	O	Ueq(max) / Ueq(min)	Range	3.1	Ratio
PLAT222_ALERT_3_C	NonSolvent	Resd	1	H	Uiso(max)/Uiso(min)	Range	4.4	Ratio
PLAT222_ALERT_3_C	NonSolvent	Resd	2	H	Uiso(max)/Uiso(min)	Range	4.4	Ratio

PLAT241_ALERT_2_C High 'MainMol' Ueq as Compared to Neighbors of O19 Check
PLAT241_ALERT_2_C High 'MainMol' Ueq as Compared to Neighbors of O23 Check
PLAT242_ALERT_2_C Low 'MainMol' Ueq as Compared to Neighbors of C11 Check
PLAT242_ALERT_2_C Low 'MainMol' Ueq as Compared to Neighbors of C21 Check
PLAT242_ALERT_2_C Low 'MainMol' Ueq as Compared to Neighbors of C63 Check
PLAT431_ALERT_2_C Short Inter HL..A Contact Br5 ..O24 . 3.10 Ang.
1-x,3-y,1-z = 2_686 Check
PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L= 0.600 369 Report

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Alert level G

PLAT002_ALERT_2_G Number of Distance or Angle Restraints on AtSite 18 Note
PLAT003_ALERT_2_G Number of Uiso or Uij Restrained non-H Atoms ... 16 Report
PLAT042_ALERT_1_G Calc. and Reported MoietyFormula Strings Differ Please Check
PLAT083_ALERT_2_G SHELXL Second Parameter in WGHT Unusually Large 7.28 Why ?
PLAT154_ALERT_1_G The s.u.'s on the Cell Angles are Equal ..(Note) 0.001 Degree
PLAT175_ALERT_4_G The CIF-Embedded .res File Contains SAME Records 2 Report
PLAT178_ALERT_4_G The CIF-Embedded .res File Contains SIMU Records 4 Report
PLAT187_ALERT_4_G The CIF-Embedded .res File Contains RIGU Records 2 Report
PLAT232_ALERT_2_G Hirshfeld Test Diff (M-X) Nd1 --O1 . 5.7 s.u.
PLAT301_ALERT_3_G Main Residue Disorder .....(Resd 1) 6% Note
PLAT301_ALERT_3_G Main Residue Disorder .....(Resd 2) 6% Note
PLAT395_ALERT_2_G Deviating X-O-Y Angle From 120 for O11A 136.1 Degree
PLAT395_ALERT_2_G Deviating X-O-Y Angle From 120 for O36A 134.2 Degree
PLAT432_ALERT_2_G Short Inter X...Y Contact Br1 ..C71 3.30 Ang.
2-x,2-y,1-z = 2_776 Check
PLAT720_ALERT_4_G Number of Unusual/Non-Standard Labels ..... 3 Note
PLAT790_ALERT_4_G Centre of Gravity not Within Unit Cell: Resd. # 2 Note
C39 H60 Br3 N3 Nd O18 P3
PLAT794_ALERT_5_G Tentative Bond Valency for Nd1 (II) . 2.14 Info
PLAT794_ALERT_5_G Tentative Bond Valency for Nd2 (II) . 2.15 Info
PLAT860_ALERT_3_G Number of Least-Squares Restraints ..... 106 Note
PLAT883_ALERT_1_G No Info/Value for _atom_sites_solution_primary . Please Do!
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L= 0.600 335 Note
PLAT941_ALERT_3_G Average HKL Measurement Multiplicity ..... 3.1 Low
PLAT960_ALERT_3_G Number of Intensities with I < - 2*sig(I) ... 1 Check
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density. 0 Info
PLAT992_ALERT_5_G Repd & Actual _reflns_number_gt Values Differ by 1 Check

2.7 Complex 13

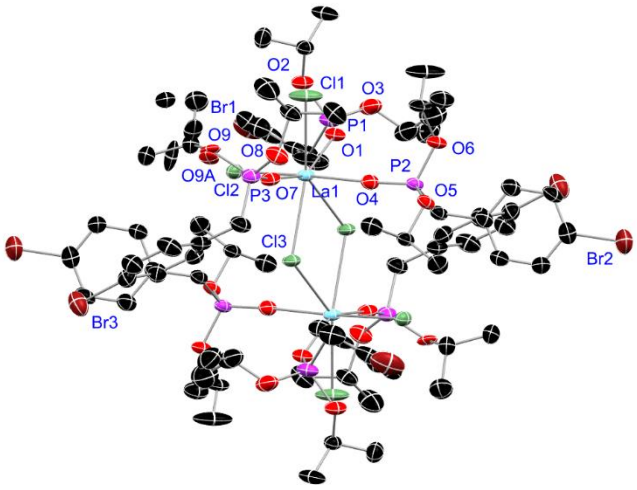

Figure 46: Asymmetric unit of **13**.

13 crystalizes in the space group $P2_1/c$. The asymmetric unit contains half of the dimer. The other half is generated by a twofold rotation axis. One of the *i*Pr groups and its bridging O-atom are disordered about two positions. They are refined with distance restraints and restraints for the anisotropic displacement parameters. The occupancy of the minor position refined to 0.375(13).

Bond lengths [Å] and angles [°] of **13**.

O(9)–C(34)	1.460(15)	P(1)–O(3)	1.534(5)
O(9)–P(3)	1.585(12)	P(1)–O(1)	1.459(5)
C(34)–C(35)	1.501(15)	P(2)–O(5)	1.545(4)
C(34)–C(36)	1.468(16)	P(2)–O(6)	1.537(4)
O(9A)–C(34A)	1.464(16)	P(2)–C(14)	1.757(6)
O(9A)–P(3)	1.51(2)	P(2)–O(4)	1.470(4)
C(34A)–C(35A)	1.51(2)	O(2)–C(11)	1.447(7)
C(34A)–C(36A)	1.48(2)	Br(2)–C(18)	1.867(7)
La(1)–Cl(1)	2.7583(15)	C(2)–C(3)	1.361(9)
La(1)–Cl(2)	2.7425(14)	C(2)–C(7)	1.372(11)
La(1)–Cl(3)#1	2.8952(13)	Br(3)–C(31)	1.860(7)
La(1)–Cl(3)	2.9317(14)	O(3)–C(8)	1.437(9)
La(1)–O(7)	2.425(4)	P(3)–O(7)	1.459(4)
La(1)–O(4)	2.366(4)	P(3)–O(8)	1.531(5)
La(1)–O(1)	2.436(4)	P(3)–C(27)	1.775(6)
Br(1)–C(5)	1.884(7)	C(3)–C(4)	1.364(10)
C(1)–P(1)	1.768(6)	C(5)–C(6)	1.340(11)
C(1)–C(2)	1.479(9)	C(5)–C(4)	1.360(12)
P(1)–O(2)	1.539(4)	O(5)–C(21)	1.460(6)

C(6)–C(7)	1.354(11)
O(6)–C(24)	1.460(7)
C(8)–C(10)	1.492(14)
C(8)–C(9)	1.480(11)
O(8)–C(37)	1.474(7)
C(11)–C(12)	1.487(8)
C(11)–C(13)	1.479(9)
C(14)–C(15)	1.494(8)
C(15)–C(16)	1.369(9)
C(15)–C(20)	1.370(8)
C(16)–C(17)	1.369(9)
C(17)–C(18)	1.364(9)
C(18)–C(19)	1.362(9)
C(19)–C(20)	1.356(9)
C(21)–C(22)	1.481(9)
C(21)–C(23)	1.484(8)
C(24)–C(25)	1.486(10)
C(24)–C(26)	1.465(9)
C(27)–C(28)	1.489(9)
C(28)–C(29)	1.369(9)
C(28)–C(33)	1.366(9)
C(29)–C(30)	1.382(9)
C(30)–C(31)	1.361(9)
C(31)–C(32)	1.369(9)
C(32)–C(33)	1.375(10)
C(37)–C(38)	1.466(11)
C(37)–C(39)	1.486(10)

C(34)–O(9)–P(3)	122.3(10)
O(9)–C(34)–C(35)	105.9(11)
O(9)–C(34)–C(36)	108.6(12)
C(36)–C(34)–C(35)	113.8(10)
C(34A)–O(9A)–P(3)	118.3(15)
O(9A)–C(34A)–C(35A)	106.8(16)
O(9A)–C(34A)–C(36A)	107.5(18)
C(36A)–C(34A)–C(35A)	112.4(16)
Cl(1)–La(1)–Cl(3)#1	146.10(5)
Cl(1)–La(1)–Cl(3)	142.00(6)
Cl(2)–La(1)–Cl(1)	97.70(6)
Cl(2)–La(1)–Cl(3)	93.11(4)
Cl(2)–La(1)–Cl(3)#1	91.76(4)
Cl(3)#1–La(1)–Cl(3)	69.09(5)
O(7)–La(1)–Cl(1)	75.68(10)
O(7)–La(1)–Cl(2)	94.09(10)
O(7)–La(1)–Cl(3)#1	71.21(9)
O(7)–La(1)–Cl(3)	139.81(9)
O(7)–La(1)–O(1)	149.58(13)
O(4)–La(1)–Cl(1)	86.37(10)
O(4)–La(1)–Cl(2)	174.27(11)
O(4)–La(1)–Cl(3)#1	87.02(10)
O(4)–La(1)–Cl(3)	81.22(10)
O(4)–La(1)–O(7)	90.82(15)
O(4)–La(1)–O(1)	92.23(15)
O(1)–La(1)–Cl(1)	74.33(10)
O(1)–La(1)–Cl(2)	85.03(11)
O(1)–La(1)–Cl(3)#1	139.17(10)
O(1)–La(1)–Cl(3)	70.46(9)
C(2)–C(1)–P(1)	118.8(4)
O(2)–P(1)–C(1)	104.7(3)
O(3)–P(1)–C(1)	109.6(3)
O(3)–P(1)–O(2)	103.3(3)
O(1)–P(1)–C(1)	111.1(3)
O(1)–P(1)–O(2)	115.3(3)
O(1)–P(1)–O(3)	112.2(3)
O(5)–P(2)–C(14)	109.7(3)
O(6)–P(2)–O(5)	104.9(2)
O(6)–P(2)–C(14)	104.1(3)
O(4)–P(2)–O(5)	111.7(2)
O(4)–P(2)–O(6)	114.2(2)
O(4)–P(2)–C(14)	111.7(3)
C(11)–O(2)–P(1)	121.5(4)
C(3)–C(2)–C(1)	121.8(7)
C(3)–C(2)–C(7)	118.4(7)
C(7)–C(2)–C(1)	119.6(6)
C(8)–O(3)–P(1)	120.3(5)
O(9)–P(3)–C(27)	111.7(5)
O(9A)–P(3)–O(8)	103.5(9)
O(9A)–P(3)–C(27)	97.1(7)

O(7)–P(3)–O(9)	106.9(5)	C(19)–C(18)–Br(2)	120.1(5)
O(7)–P(3)–O(9A)	122.5(7)	C(19)–C(18)–C(17)	120.0(6)
O(7)–P(3)–O(8)	115.6(3)	C(20)–C(19)–C(18)	120.5(6)
O(7)–P(3)–C(27)	113.7(3)	C(19)–C(20)–C(15)	120.8(6)
O(8)–P(3)–O(9)	107.9(5)	O(5)–C(21)–C(22)	108.1(5)
O(8)–P(3)–C(27)	101.0(3)	O(5)–C(21)–C(23)	106.5(5)
La(1)#1–Cl(3)–La(1)	110.91(5)	C(22)–C(21)–C(23)	113.3(6)
C(2)–C(3)–C(4)	121.7(7)	O(6)–C(24)–C(25)	106.3(6)
C(6)–C(5)–Br(1)	119.6(7)	O(6)–C(24)–C(26)	108.4(5)
C(6)–C(5)–C(4)	121.6(7)	C(26)–C(24)–C(25)	113.5(6)
C(4)–C(5)–Br(1)	118.8(6)	C(28)–C(27)–P(3)	114.2(5)
C(21)–O(5)–P(2)	120.0(3)	C(29)–C(28)–C(27)	120.8(6)
C(5)–C(6)–C(7)	120.1(8)	C(33)–C(28)–C(27)	120.2(6)
C(24)–O(6)–P(2)	122.6(4)	C(33)–C(28)–C(29)	119.0(6)
C(6)–C(7)–C(2)	120.3(7)	C(28)–C(29)–C(30)	120.2(6)
P(3)–O(7)–La(1)	172.9(3)	C(31)–C(30)–C(29)	120.2(6)
O(3)–C(8)–C(10)	109.6(7)	C(30)–C(31)–Br(3)	119.4(5)
O(3)–C(8)–C(9)	107.1(7)	C(30)–C(31)–C(32)	120.0(6)
C(9)–C(8)–C(10)	111.9(8)	C(32)–C(31)–Br(3)	120.6(5)
C(37)–O(8)–P(3)	122.3(4)	C(31)–C(32)–C(33)	119.4(6)
O(2)–C(11)–C(12)	108.3(5)	C(28)–C(33)–C(32)	121.2(6)
O(2)–C(11)–C(13)	107.6(5)	O(8)–C(37)–C(39)	107.2(6)
C(13)–C(11)–C(12)	113.6(6)	C(38)–C(37)–O(8)	106.2(6)
C(15)–C(14)–P(2)	116.6(4)	C(38)–C(37)–C(39)	113.4(6)
C(16)–C(15)–C(14)	120.4(5)	C(5)–C(4)–C(3)	118.0(7)
C(16)–C(15)–C(20)	118.1(6)	P(2)–O(4)–La(1)	160.2(3)
C(20)–C(15)–C(14)	121.4(6)	P(1)–O(1)–La(1)	145.1(3)
C(17)–C(16)–C(15)	121.6(6)		
C(18)–C(17)–C(16)	119.0(6)		
C(17)–C(18)–Br(2)	119.9(5)		

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) i2939

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: i2939

```
Bond precision:  C-C = 0.0106 A          Wavelength=0.71073

Cell:           a=14.0316(6)    b=28.3123(14)       c=14.2123(6)
                alpha=90        beta=116.555(3)     gamma=90
Temperature:    100 K

                Calculated                  Reported
Volume          5050.5(4)                   5050.4(4)
Space group     P 21/c                      P 1 21/c 1
Hall group      -P 2ybc                     -P 2ybc
Moiety formula  C78 H120 Br6 Cl6 La2 O18    C78 H120 Br6 Cl6 La2 O18
                P6                          P6

Sum formula     C78 H120 Br6 Cl6 La2 O18    C78 H120 Br6 Cl6 La2 O18
                P6                          P6
Mr              2501.49                     2501.53
Dx,g cm-3       1.645                       1.645
Z               2                           2
Mu (mm-1)       3.520                       3.520
F000            2496.0                      2496.0
F000'           2495.34
h,k,lmax        17,35,18                    17,35,18
Nref            10812                       10707
Tmin,Tmax       0.520,0.869                 0.251,0.748
Tmin'           0.321

Correction method= # Reported T Limits: Tmin=0.251 Tmax=0.748
AbsCorr = MULTI-SCAN

Data completeness= 0.990          Theta(max)= 26.823

R(reflections)= 0.0531( 8482)     wR2(reflections)= 0.1346( 10707)

S = 1.110                  Npar= 574
```

The following ALERTS were generated. Each ALERT has the format

test-name_ALERT_alert-type_alert-level.

Click on the hyperlinks for more details of the test.

Alert level C

```
PLAT213_ALERT_2_C Atom O9A            has ADP max/min Ratio .....       4.0 oblate
PLAT213_ALERT_2_C Atom C34A           has ADP max/min Ratio .....       3.1 prolat
PLAT334_ALERT_2_C Small Aver. Benzene C-C Dist C2       -C7             1.36 Ang.
PLAT334_ALERT_2_C Small Aver. Benzene C-C Dist C15      -C20            1.37 Ang.
PLAT334_ALERT_2_C Small Aver. Benzene C-C Dist C28      -C33            1.37 Ang.
PLAT342_ALERT_3_C Low Bond Precision on  C-C Bonds ..............    0.01065 Ang.
PLAT434_ALERT_2_C Short Inter HL..HL Contact Br1      ..Br2             3.31 Ang.
                                          1+x,3/2-y,-1/2+z  =      4_675 Check
PLAT906_ALERT_3_C Large K Value in the Analysis of Variance ......     4.176 Check
PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L=    0.600      10 Report
```

PLAT977_ALERT_2_C Check Negative Difference Density on H34A -0.36 eA-3
PLAT978_ALERT_2_C Number C-C Bonds with Positive Residual Density. 0 Info

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Alert level G

PLAT002_ALERT_2_G Number of Distance or Angle Restraints on AtSite 8 Note
PLAT003_ALERT_2_G Number of Uiso or Uij Restrained non-H Atoms ... 7 Report
PLAT083_ALERT_2_G SHELXL Second Parameter in WGHT Unusually Large 32.98 Why?
PLAT175_ALERT_4_G The CIF-Embedded .res File Contains SAME Records 1 Report
PLAT178_ALERT_4_G The CIF-Embedded .res File Contains SIMU Records 1 Report
PLAT187_ALERT_4_G The CIF-Embedded .res File Contains RIGU Records 1 Report
PLAT301_ALERT_3_G Main Residue Disorder .....(Resd 1) 7% Note
PLAT413_ALERT_2_G Short Inter XH3 .. XHn H33 ..H36D . 2.10 Ang.
x,1/2-y,1/2+z = 4_566 Check
PLAT432_ALERT_2_G Short Inter X...Y Contact C9 ..C35 3.09 Ang.
1-x,1/2+y,3/2-z = 2_656 Check
PLAT794_ALERT_5_G Tentative Bond Valency for La1 (III) . 3.47 Info
PLAT860_ALERT_3_G Number of Least-Squares Restraints ..... 105 Note
PLAT883_ALERT_1_G No Info/Value for _atom_sites_solution_primary . Please Do!
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L= 0.600 96 Note
PLAT933_ALERT_2_G Number of OMIT Records in Embedded .res File ... 12 Note

2.8 Complex 14

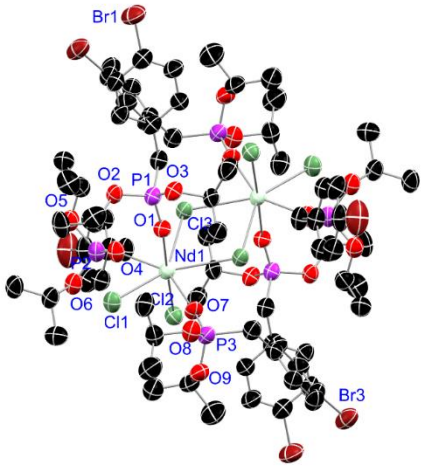

Figure 47: Asymmetric unit of **14**.

14 crystalizes in the space group $P2_1/c$. The asymmetric unit contains half of the dimer. The other half is generated by a twofold rotation axis. The B-Alert on low bond precision is due to bad crystal quality - no hints for twinning are found.

Bond lengths [Å] and angles [°] of **14.**

C(1)–C(2)	1.521(17)	C(17)–C(18)	1.36(2)
C(1)–P(1)	1.766(13)	C(18)–C(19)	1.36(2)
C(2)–C(3)	1.40(2)	C(18)–Br(2)	1.917(15)
C(2)–C(7)	1.386(18)	C(19)–C(20)	1.36(2)
C(3)–C(4)	1.38(2)	C(21)–C(22)	1.47(2)
C(4)–C(5)	1.33(2)	C(21)–C(23)	1.52(2)
C(5)–C(6)	1.37(2)	C(21)–O(5)	1.445(19)
C(5)–Br(1)	1.903(15)	C(24)–C(25)	1.488(19)
C(6)–C(7)	1.39(2)	C(24)–C(26)	1.49(2)
C(8)–C(9)	1.479(19)	C(24)–O(6)	1.500(14)
C(8)–C(10)	1.49(2)	C(27)–C(28)	1.504(19)
C(8)–O(3)	1.477(14)	C(27)–P(3)	1.804(14)
C(11)–C(12)	1.48(2)	C(28)–C(29)	1.403(18)
C(11)–C(13)	1.492(19)	C(28)–C(33)	1.396(19)
C(11)–O(2)	1.496(16)	C(29)–C(30)	1.37(2)
C(14)–C(15)	1.508(18)	C(30)–C(31)	1.38(2)
C(14)–P(2)	1.797(13)	C(31)–C(32)	1.372(19)
C(15)–C(16)	1.36(2)	C(31)–Br(3)	1.932(14)
C(15)–C(20)	1.39(2)	C(32)–C(33)	1.41(2)
C(16)–C(17)	1.40(2)	C(34)–C(35)	1.51(2)

C(34)–C(36)	1.49(2)
C(34)–O(8)	1.470(17)
C(37)–C(38)	1.44(2)
C(37)–C(39)	1.51(2)
C(37)–O(9)	1.52(2)
Cl(1)–Nd(1)	2.747(3)
Cl(2)–Nd(1)	2.703(3)
Cl(3)–Nd(1)	2.892(3)
Cl(3)–Nd(1)#1	2.927(3)
Nd(1)–O(1)	2.333(7)
Nd(1)–O(4)	2.398(9)
Nd(1)–O(7)	2.403(8)
O(1)–P(1)	1.485(8)
O(2)–P(1)	1.556(8)
O(3)–P(1)	1.557(9)
O(4)–P(2)	1.479(9)
O(5)–P(2)	1.571(10)
O(6)–P(2)	1.557(11)
O(7)–P(3)	1.476(8)
O(8)–P(3)	1.568(9)
O(9)–P(3)	1.542(10)

C(2)–C(1)–P(1)	116.8(8)
C(3)–C(2)–C(1)	119.7(11)
C(7)–C(2)–C(1)	122.3(13)
C(7)–C(2)–C(3)	118.0(13)
C(4)–C(3)–C(2)	120.3(14)
C(5)–C(4)–C(3)	121.3(16)
C(4)–C(5)–C(6)	120.2(15)
C(4)–C(5)–Br(1)	122.3(13)
C(6)–C(5)–Br(1)	117.4(11)
C(5)–C(6)–C(7)	120.2(13)
C(2)–C(7)–C(6)	120.0(14)
C(9)–C(8)–C(10)	113.7(13)
O(3)–C(8)–C(9)	108.6(10)
O(3)–C(8)–C(10)	107.7(10)
C(12)–C(11)–C(13)	116.1(13)
C(12)–C(11)–O(2)	106.8(11)
C(13)–C(11)–O(2)	108.0(12)
C(15)–C(14)–P(2)	116.8(10)
C(16)–C(15)–C(14)	120.8(15)
C(16)–C(15)–C(20)	119.5(13)
C(20)–C(15)–C(14)	119.5(13)
C(15)–C(16)–C(17)	120.7(15)
C(17)–C(18)–Br(2)	118.1(13)
C(19)–C(18)–C(17)	123.0(15)
C(19)–C(18)–Br(2)	118.9(15)
C(20)–C(19)–C(18)	119.1(18)
C(19)–C(20)–C(15)	120.4(15)
C(22)–C(21)–C(23)	113.4(15)
O(5)–C(21)–C(22)	111.4(14)
O(5)–C(21)–C(23)	105.8(12)
C(25)–C(24)–C(26)	115.6(14)
C(25)–C(24)–O(6)	107.9(10)
C(26)–C(24)–O(6)	106.6(10)
C(28)–C(27)–P(3)	113.6(10)
C(29)–C(28)–C(27)	121.2(12)
C(33)–C(28)–C(27)	122.3(11)
C(33)–C(28)–C(29)	116.6(13)
C(30)–C(29)–C(28)	122.8(13)
C(29)–C(30)–C(31)	117.3(13)
C(30)–C(31)–Br(3)	119.4(11)
C(32)–C(31)–C(30)	124.7(14)
C(32)–C(31)–Br(3)	115.9(11)
C(31)–C(32)–C(33)	115.9(13)
C(28)–C(33)–C(32)	122.7(12)
C(36)–C(34)–C(35)	111.8(14)
O(8)–C(34)–C(35)	105.4(12)
O(8)–C(34)–C(36)	106.8(12)
C(38)–C(37)–C(39)	115.8(16)
C(38)–C(37)–O(9)	109.7(15)
C(39)–C(37)–O(9)	105.9(14)
Nd(1)–Cl(3)–Nd(1)#1	111.13(9)
Cl(1)–Nd(1)–Cl(3)	146.34(9)
Cl(1)–Nd(1)–Cl(3)#1	141.51(9)
Cl(2)–Nd(1)–Cl(1)	98.09(10)
Cl(2)–Nd(1)–Cl(3)	91.58(9)
Cl(2)–Nd(1)–Cl(3)#1	94.10(9)

Cl(3)–Nd(1)–Cl(3)#1	68.87(9)	P(3)–O(7)–Nd(1)	173.5(6)
O(1)–Nd(1)–Cl(1)	85.4(2)	C(34)–O(8)–P(3)	122.3(9)
O(1)–Nd(1)–Cl(2)	175.9(2)	C(37)–O(9)–P(3)	122.4(9)
O(1)–Nd(1)–Cl(3)#1	81.8(2)	O(1)–P(1)–C(1)	111.0(5)
O(1)–Nd(1)–Cl(3)	86.7(2)	O(1)–P(1)–O(2)	114.4(5)
O(1)–Nd(1)–O(4)	93.6(3)	O(1)–P(1)–O(3)	112.0(5)
O(1)–Nd(1)–O(7)	91.4(3)	O(2)–P(1)–C(1)	104.4(5)
O(4)–Nd(1)–Cl(1)	74.0(2)	O(2)–P(1)–O(3)	104.8(5)
O(4)–Nd(1)–Cl(2)	85.3(2)	O(3)–P(1)–C(1)	109.7(6)
O(4)–Nd(1)–Cl(3)	139.2(2)	O(4)–P(2)–C(14)	110.8(6)
O(4)–Nd(1)–Cl(3)#1	70.8(2)	O(4)–P(2)–O(5)	111.4(6)
O(4)–Nd(1)–O(7)	149.1(3)	O(4)–P(2)–O(6)	115.8(5)
O(7)–Nd(1)–Cl(1)	76.1(2)	O(5)–P(2)–C(14)	110.2(6)
O(7)–Nd(1)–Cl(2)	91.5(2)	O(6)–P(2)–C(14)	104.8(7)
O(7)–Nd(1)–Cl(3)#1	140.0(2)	O(6)–P(2)–O(5)	103.4(5)
O(7)–Nd(1)–Cl(3)	71.4(2)	O(7)–P(3)–C(27)	112.4(6)
P(1)–O(1)–Nd(1)	160.6(5)	O(7)–P(3)–O(8)	116.7(5)
C(11)–O(2)–P(1)	123.7(8)	O(7)–P(3)–O(9)	111.8(6)
C(8)–O(3)–P(1)	120.2(7)	O(8)–P(3)–C(27)	100.9(6)
P(2)–O(4)–Nd(1)	146.6(6)	O(9)–P(3)–C(27)	108.8(6)
C(21)–O(5)–P(2)	120.4(9)	O(9)–P(3)–O(8)	105.3(6)
C(24)–O(6)–P(2)	121.1(9)		

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) sv0766

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. *CIF dictionary* *Interpreting this report*

Datablock: sv0766

```
Bond precision:  C-C = 0.0223 A          Wavelength=1.54186

Cell:            a=14.0648(5)     b=29.1214(12)       c=14.2850(5)
                 alpha=90         beta=116.184(2)     gamma=90
Temperature:     100 K

                 Calculated                Reported
Volume           5250.5(4)                 5250.5(3)
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Space group	P 21/c	P 1 21/c 1
Hall group	-P 2ybc	-P 2ybc
Moiety formula	C78 H120 Br6 Cl6 Nd2 O18	C78 H120 Br6 Cl6 Nd2 O18
	P6	P6
Sum formula	C78 H120 Br6 Cl6 Nd2 O18	C78 H120 Br6 Cl6 Nd2 O18
	P6	P6
Mr	2512.15	2512.19
Dx,g cm-3	1.589	1.589
Z	2	2
Mu (mm-1)	12.863	12.863
F000	2508.0	2508.0
F000'	2500.48	
h,k,lmax	17,35,17	16,34,17
Nref	9853	9520
Tmin,Tmax	0.127,0.314	0.040,0.345
Tmin'	0.045	

Correction method= # Reported T Limits: Tmin=0.040 Tmax=0.345
AbsCorr = MULTI-SCAN

Data completeness= 0.966 Theta(max)= 69.496

R(reflections)= 0.0965(7701) wR2(reflections)= 0.2872(9520)

S = 1.053 Npar= 535

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level B

PLAT342_ALERT_3_B Low Bond Precision on C-C Bonds ..... 0.0223 Ang.

Alert level C

PLAT029_ALERT_3_C _diffrn_measured_fraction_theta_full value Low . 0.977 Why?
PLAT084_ALERT_3_C High wR2 Value (i.e. > 0.25) ..... 0.29 Report
PLAT094_ALERT_2_C Ratio of Maximum / Minimum Residual Density .... 2.09 Report
PLAT242_ALERT_2_C Low MainMol Ueq as Compared to Neighbors of C8 Check
PLAT334_ALERT_2_C Small Aver. Benzene C-C Dist C15 -C20 1.37 Ang.
PLAT434_ALERT_2_C Short Inter HL..HL Contact Br1 ..Br2 3.29 Ang.
-1+x,3/2-y,1/2+z = 4_476 Check
PLAT906_ALERT_3_C Large K Value in the Analysis of Variance ..... 4.331 Check
PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L= 0.600 214 Report
PLAT971_ALERT_2_C Check Calcd Resid. Dens. 1.07A From Br1 2.40 eA-3
PLAT971_ALERT_2_C Check Calcd Resid. Dens. 1.13A From Nd1 1.86 eA-3
PLAT971_ALERT_2_C Check Calcd Resid. Dens. 1.10A From Nd1 1.83 eA-3
PLAT971_ALERT_2_C Check Calcd Resid. Dens. 1.16A From Nd1 1.69 eA-3
PLAT971_ALERT_2_C Check Calcd Resid. Dens. 1.12A From Nd1 1.66 eA-3
PLAT973_ALERT_2_C Check Calcd Positive Resid. Density on Nd1 1.46 eA-3
PLAT977_ALERT_2_C Check Negative Difference Density on H32 -0.42 eA-3
PLAT978_ALERT_2_C Number C-C Bonds with Positive Residual Density. 0 Info

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Alert level G

PLAT072_ALERT_2_G SHELXL First Parameter in WGHT Unusually Large 0.15 Report
PLAT083_ALERT_2_G SHELXL Second Parameter in WGHT Unusually Large 66.81 Why ?

PLAT794_ALERT_5_G Tentative Bond Valency for Nd1 (III) . 3.16 Info
PLAT883_ALERT_1_G No Info/Value for _atom_sites_solution_primary . Please Do !
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L= 0.600 102 Note
PLAT933_ALERT_2_G Number of OMIT Records in Embedded .res File ... 14 Note
PLAT960_ALERT_3_G Number of Intensities with I < - 2*sig(I) ... 34 Check

2.9 Complex 17

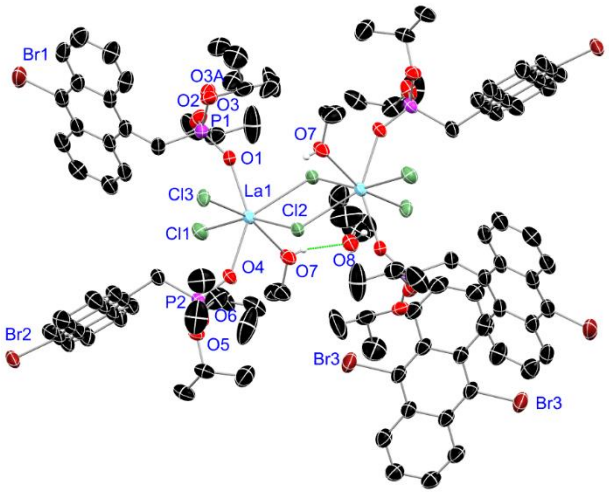

Figure 48: Asymmetric unit of **17**.

17 crystalizes as $C_{88}H_{108}Br_4Cl_6La_2O_{14}P_4 \cdot C_{14}H_8Br_2 \cdot 2EtOH$ in the space group $P\bar{1}$. The asymmetric unit contains half of the dimer as well as a co-crystallized EtOH and half of a 9,10-dibromoanthracene molecule. The other half is generated by an inversion center. One of the three anthracene-phosphonate ligands is missing and substituted by a coordinating EtOH molecule. Two *i*Pr groups and the co-crystalized EtOH molecule are disordered about two positions. In one of the *i*Pr group disorders, the bridging O-atom is involved as well. They are refined with distance restraints and partially with restraints for the anisotropic displacement parameters. The occupancy of the minor positions refined to 0.436(41), 0.476(19) and 0.462(20), respectively. The protons of the EtOH hydroxy groups are refined freely.

Bond lengths [Å] and angles [°] of **17**

La(1)–O(1)	2.417(3)	O(3)–C(19)	1.421(13)
La(1)–Cl(1)	2.7684(13)	C(19)–C(20)	1.447(19)
La(1)–Cl(2)	2.8653(11)	C(19)–C(21)	1.518(15)
La(1)–Cl(2)#1	2.8796(12)	O(3A)–C(19A)	1.392(16)
La(1)–Cl(3)	2.8347(12)	C(19A)–C(20A)	1.42(2)
La(1)–O(4)	2.425(3)	C(19A)–C(21A)	1.501(17)
La(1)–O(7)	2.501(4)	Br(2)–C(30)	1.904(5)
Br(1)–C(9)	1.892(5)	C(2)–C(3)	1.412(8)
C(1)–P(1)	1.792(5)	C(2)–C(15)	1.394(7)
C(1)–C(2)	1.508(7)	O(2)–C(16)	1.454(7)
O(1)–P(1)	1.478(3)	P(2)–O(4)	1.487(3)
P(1)–O(3)	1.572(8)	P(2)–O(5)	1.546(3)
P(1)–O(3A)	1.571(11)	P(2)–C(22)	1.780(5)
P(1)–O(2)	1.545(4)	P(2)–O(6)	1.562(4)

C(3)–C(4)	1.423(8)	O(6)–C(40)	1.460(13)
C(3)–C(8)	1.440(7)	O(6)–C(40A)	1.470(13)
Br(3)–C(46)	1.899(5)	C(40)–C(41)	1.52(2)
C(4)–C(5)	1.364(8)	C(40)–C(42)	1.434(18)
O(5)–C(37)	1.471(6)	C(40A)–C(41A)	1.53(2)
C(5)–C(6)	1.397(8)	C(40A)–C(42A)	1.44(2)
C(6)–C(7)	1.336(9)	C(43)–C(44)	1.470(9)
C(9)–C(8)	1.392(8)	C(45)–C(46)	1.390(7)
C(9)–C(10)	1.402(8)	C(45)–C(47)#2	1.433(7)
C(8)–C(7)	1.428(8)	C(45)–C(51)#2	1.430(7)
O(7)–H(7)	0.70(7)	C(46)–C(47)	1.396(7)
O(7)–C(43)	1.444(6)	C(47)–C(48)	1.420(7)
C(10)–C(11)	1.422(8)	C(48)–C(49)	1.361(8)
C(10)–C(15)	1.432(7)	C(49)–C(50)	1.387(8)
C(12)–C(11)	1.344(9)	C(50)–C(51)	1.355(8)
C(12)–C(13)	1.411(8)	O(8)–C(52)	1.409(12)
C(13)–C(14)	1.344(8)	O(8)–H(8)	0.79(5)
C(14)–C(15)	1.429(8)	O(8)–C(52A)	1.440(15)
C(16)–C(17)	1.459(9)	C(52)–C(53)	1.410(16)
C(16)–C(18)	1.475(9)	C(52A)–C(53A)	1.428(16)
C(22)–C(23)	1.515(6)		
C(23)–C(24)	1.404(7)	O(1)–La(1)–Cl(1)	76.81(8)
C(23)–C(36)	1.408(7)	O(1)–La(1)–Cl(2)	72.85(8)
C(24)–C(25)	1.412(7)	O(1)–La(1)–Cl(2)#1	123.12(8)
C(24)–C(29)	1.442(7)	O(1)–La(1)–Cl(3)	78.70(9)
C(25)–C(26)	1.352(7)	O(1)–La(1)–O(4)	145.48(10)
C(26)–C(27)	1.411(8)	O(1)–La(1)–O(7)	124.15(13)
C(27)–C(28)	1.351(8)	Cl(1)–La(1)–Cl(2)	118.14(4)
C(28)–C(29)	1.429(7)	Cl(1)–La(1)–Cl(2)#1	159.74(4)
C(29)–C(30)	1.392(8)	Cl(1)–La(1)–Cl(3)	102.18(4)
C(30)–C(31)	1.392(7)	Cl(2)–La(1)–Cl(2)#1	75.19(4)
C(31)–C(32)	1.428(8)	Cl(3)–La(1)–Cl(2)#1	80.34(3)
C(31)–C(36)	1.439(6)	Cl(3)–La(1)–Cl(2)	122.19(3)
C(32)–C(33)	1.351(8)	O(4)–La(1)–Cl(1)	80.51(8)
C(33)–C(34)	1.409(7)	O(4)–La(1)–Cl(2)#1	80.04(8)
C(34)–C(35)	1.349(7)	O(4)–La(1)–Cl(2)	141.52(8)
C(35)–C(36)	1.429(7)	O(4)–La(1)–Cl(3)	81.09(8)
C(37)–C(38)	1.498(7)	O(4)–La(1)–O(7)	77.36(11)
C(37)–C(39)	1.517(8)	O(7)–La(1)–Cl(1)	82.33(10)

O(7)–La(1)–Cl(2)	72.81(9)	C(4)–C(5)–C(6)	120.2(6)
O(7)–La(1)–Cl(2)#1	87.92(10)	C(7)–C(6)–C(5)	120.6(6)
O(7)–La(1)–Cl(3)	156.95(10)	C(8)–C(9)–Br(1)	119.0(4)
C(2)–C(1)–P(1)	118.7(4)	C(8)–C(9)–C(10)	122.6(5)
P(1)–O(1)–La(1)	152.06(19)	C(10)–C(9)–Br(1)	118.4(4)
O(1)–P(1)–C(1)	111.7(2)	C(9)–C(8)–C(3)	118.7(5)
O(1)–P(1)–O(3)	107.9(6)	C(9)–C(8)–C(7)	123.8(5)
O(1)–P(1)–O(3A)	119.3(7)	C(7)–C(8)–C(3)	117.5(6)
O(1)–P(1)–O(2)	114.3(2)	La(1)–O(7)–H(7)	119(6)
O(3)–P(1)–C(1)	104.0(6)	C(43)–O(7)–La(1)	129.4(3)
O(3A)–P(1)–C(1)	110.2(8)	C(43)–O(7)–H(7)	111(6)
O(2)–P(1)–C(1)	108.0(2)	C(6)–C(7)–C(8)	122.4(6)
O(2)–P(1)–O(3)	110.4(7)	C(9)–C(10)–C(11)	122.7(5)
O(2)–P(1)–O(3A)	91.5(9)	C(9)–C(10)–C(15)	117.7(5)
C(19)–O(3)–P(1)	125.9(11)	C(11)–C(10)–C(15)	119.6(5)
O(3)–C(19)–C(20)	115.4(13)	C(11)–C(12)–C(13)	119.8(6)
O(3)–C(19)–C(21)	104.7(12)	C(12)–C(11)–C(10)	121.4(5)
C(20)–C(19)–C(21)	112.8(13)	C(14)–C(13)–C(12)	120.7(6)
C(19A)–O(3A)–P(1)	125.2(13)	C(13)–C(14)–C(15)	122.4(5)
O(3A)–C(19A)–C(20A)	122(2)	C(2)–C(15)–C(10)	121.3(5)
O(3A)–C(19A)–C(21A)	109.5(17)	C(2)–C(15)–C(14)	122.6(5)
C(20A)–C(19A)–C(21A)	119.8(16)	C(14)–C(15)–C(10)	116.2(5)
C(3)–C(2)–C(1)	119.0(5)	O(2)–C(16)–C(17)	110.8(6)
C(15)–C(2)–C(1)	121.1(5)	O(2)–C(16)–C(18)	106.8(6)
C(15)–C(2)–C(3)	119.9(5)	C(17)–C(16)–C(18)	113.1(6)
C(16)–O(2)–P(1)	126.7(4)	C(23)–C(22)–P(2)	117.6(4)
O(4)–P(2)–O(5)	113.45(18)	C(24)–C(23)–C(22)	120.5(5)
O(4)–P(2)–C(22)	110.4(2)	C(24)–C(23)–C(36)	120.6(4)
O(4)–P(2)–O(6)	112.06(19)	C(36)–C(23)–C(22)	118.8(4)
O(5)–P(2)–C(22)	104.8(2)	C(23)–C(24)–C(25)	122.2(4)
O(5)–P(2)–O(6)	105.5(2)	C(23)–C(24)–C(29)	119.7(5)
O(6)–P(2)–C(22)	110.3(2)	C(25)–C(24)–C(29)	118.1(5)
La(1)–Cl(2)–La(1)#1	104.81(4)	C(26)–C(25)–C(24)	122.0(5)
C(2)–C(3)–C(4)	122.5(5)	C(25)–C(26)–C(27)	120.2(6)
C(2)–C(3)–C(8)	119.7(5)	C(28)–C(27)–C(26)	120.3(5)
C(4)–C(3)–C(8)	117.8(5)	C(27)–C(28)–C(29)	121.5(5)
C(5)–C(4)–C(3)	121.6(5)	C(28)–C(29)–C(24)	117.8(5)
P(2)–O(4)–La(1)	146.3(2)	C(30)–C(29)–C(24)	118.6(5)
C(37)–O(5)–P(2)	128.9(3)	C(30)–C(29)–C(28)	123.6(5)

C(29)–C(30)–Br(2)	118.3(4)	C(42A)–C(40A)–O(6)	108.2(16)
C(29)–C(30)–C(31)	122.8(5)	C(42A)–C(40A)–C(41A)	110.6(15)
C(31)–C(30)–Br(2)	118.8(4)	O(7)–C(43)–C(44)	111.4(5)
C(30)–C(31)–C(32)	123.2(5)	C(46)–C(45)–C(47)#2	118.5(5)
C(30)–C(31)–C(36)	118.6(5)	C(46)–C(45)–C(51)#2	123.7(5)
C(32)–C(31)–C(36)	118.2(5)	C(51)#2–C(45)–C(47)#2	117.8(5)
C(33)–C(32)–C(31)	121.6(5)	C(45)–C(46)–Br(3)	118.6(4)
C(32)–C(33)–C(34)	120.3(5)	C(45)–C(46)–C(47)	123.4(5)
C(35)–C(34)–C(33)	120.6(5)	C(47)–C(46)–Br(3)	118.0(4)
C(34)–C(35)–C(36)	121.8(5)	C(46)–C(47)–C(45)#2	118.1(5)
C(23)–C(36)–C(31)	119.7(4)	C(46)–C(47)–C(48)	123.9(5)
C(23)–C(36)–C(35)	122.7(4)	C(48)–C(47)–C(45)#2	118.0(5)
C(35)–C(36)–C(31)	117.6(5)	C(49)–C(48)–C(47)	121.9(5)
O(5)–C(37)–C(38)	105.8(4)	C(48)–C(49)–C(50)	119.9(6)
O(5)–C(37)–C(39)	108.0(4)	C(51)–C(50)–C(49)	121.2(5)
C(38)–C(37)–C(39)	113.6(5)	C(50)–C(51)–C(45)#2	121.2(5)
C(40)–O(6)–P(2)	127.8(11)	C(52)–O(8)–H(8)	108(4)
C(40A)–O(6)–P(2)	119.1(9)	C(52A)–O(8)–H(8)	101(4)
O(6)–C(40)–C(41)	104.8(14)	O(8)–C(52)–C(53)	113.9(13)
C(42)–C(40)–O(6)	108.0(13)	C(53A)–C(52A)–O(8)	113.5(15)
C(42)–C(40)–C(41)	114.1(16)		
O(6)–C(40A)–C(41A)	110.3(13)		

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) i2974

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. *CIF dictionary* *Interpreting this report*

Datablock: i2974

```
Bond precision:  C-C = 0.0080 A          Wavelength=0.71073

Cell:          a=13.4455(5)      b=13.7347(6)      c=15.6918(7)
               alpha=97.274(3)   beta=98.726(3)    gamma=93.626(3)
Temperature:   100 K

                Calculated                Reported
Volume          2831.2(2)                 2831.1(2)
```

Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	C88 H108 Br4 Cl6 La2 O14	C88 H108 Br4 Cl6 La2 O14
	P4, C14 H8 Br2, 2(C2 H6 O)	P4, C14 H8 Br2, 2(C2 H6 O)
Sum formula	C106 H128 Br6 Cl6 La2 O16	C106 H128 Br6 Cl6 La2 O16
	P4	P4
Mr	2751.89	2751.94
Dx,g cm-3	1.614	1.614
Z	1	1
Mu (mm-1)	3.120	3.120
F000	1378.0	1378.0
F000'	1377.49	
h,k,lmax	17,17,19	17,17,19
Nref	12141	11968
Tmin,Tmax	0.721,0.856	0.581,0.840
Tmin'	0.660	

Correction method= # Reported T Limits: Tmin=0.581 Tmax=0.840
AbsCorr = MULTI-SCAN

Data completeness= 0.986 Theta(max)= 26.834

R(reflections)= 0.0437(8675) wR2(reflections)= 0.1035(11968)

S = 1.079 Npar= 738

The following ALERTS were generated. Each ALERT has the format

test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level C

PLAT213_ALERT_2_C Atom C20A has ADP max/min Ratio ..... 3.6 prolat
PLAT220_ALERT_2_C NonSolvent Resd 1 C Ueq(max) / Ueq(min) Range 3.3 Ratio
PLAT223_ALERT_4_C Solv./Anion Resd 3 H Ueq(max)/Ueq(min) Range 4.3 Ratio
PLAT242_ALERT_2_C Low MainMol Ueq as Compared to Neighbors of C16 Check
PLAT244_ALERT_4_C Low Solvent Ueq as Compared to Neighbors of O8 Check
PLAT245_ALERT_2_C U(iso) H8 Smaller than U(eq) O8 by 0.017 Ang**2
PLAT250_ALERT_2_C Large U3/U1 Ratio for Average U(i,j) Tensor .... 2.1 Note
PLAT354_ALERT_3_C Short O-H (X0.82,N0.98A) O7 - H7 . 0.70 Ang.
PLAT410_ALERT_2_C Short Intra H...H Contact H1A ..H4 . 1.99 Ang.
x,y,z = 1_555 Check
PLAT906_ALERT_3_C Large K Value in the Analysis of Variance ..... 2.719 Check
PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L= 0.600 33 Report
PLAT978_ALERT_2_C Number C-C Bonds with Positive Residual Density. 0 Info

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Alert level G

PLAT002_ALERT_2_G Number of Distance or Angle Restraints on AtSite 21 Note
PLAT003_ALERT_2_G Number of Uiso or Uij Restrained non-H Atoms ... 14 Report
PLAT083_ALERT_2_G SHELXL Second Parameter in WGHT Unusually Large 9.82 Why ?
PLAT154_ALERT_1_G The s.u.'s on the Cell Angles are Equal ..(Note) 0.003 Degree
PLAT175_ALERT_4_G The CIF-Embedded .res File Contains SAME Records 3 Report
PLAT178_ALERT_4_G The CIF-Embedded .res File Contains SIMU Records 4 Report
PLAT187_ALERT_4_G The CIF-Embedded .res File Contains RIGU Records 1 Report
PLAT301_ALERT_3_G Main Residue Disorder .....(Resd 1) 12% Note
PLAT302_ALERT_4_G Anion/Solvent/Minor-Residue Disorder (Resd 3) 67% Note

```
PLAT398_ALERT_2_G Deviating  C-O-C    Angle From 120 for O8              27.4 Degree
PLAT414_ALERT_2_G Short Intra D-H..H-X       H8       ..H52B            2.06 Ang.
                                                      x,y,z  =      1_555 Check
PLAT414_ALERT_2_G Short Intra D-H..H-X       H8       ..H52D            1.92 Ang.
                                                      x,y,z  =      1_555 Check
PLAT793_ALERT_4_G Model has Chirality at P2          (Centro SPGR)          S Verify
PLAT794_ALERT_5_G Tentative Bond Valency for La1       (III)     .       3.26 Info
PLAT860_ALERT_3_G Number of Least-Squares Restraints .............         95 Note
PLAT883_ALERT_1_G No Info/Value for _atom_sites_solution_primary .     Please Do!
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L=  0.600       142 Note
PLAT933_ALERT_2_G Number of OMIT Records in Embedded .res File ...        12 Note
```

2.10 Complex 18

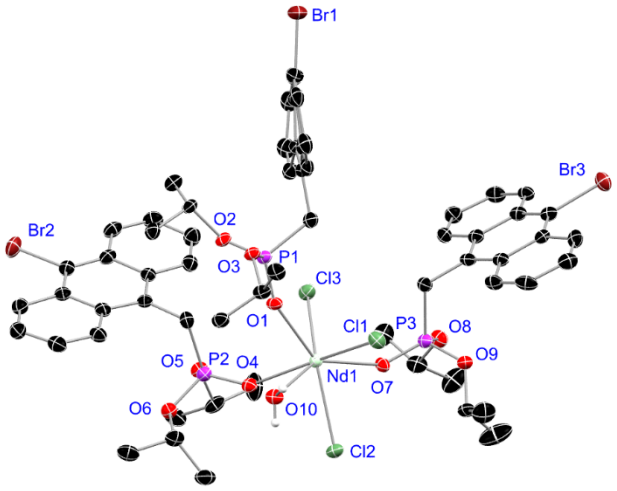

Figure 49: Asymmetric unit of **18**.

18 crystalizes as $C_{63}H_{74}Br_3Cl_3NdO_{10}P_3$ in the space group $P\bar{1}$. The asymmetric unit contains one monomeric molecule. The H_2O molecule coordinates to the Nd ion to complete its coordination sphere and its protons are refined freely.

Bond lengths [Å] and angles [°] of **18**

Nd(1)–O(1)	2.388(3)	C(2)–C(15)	1.409(8)
Nd(1)–Cl(1)	2.7405(14)	O(3)–C(19)	1.490(6)
Nd(1)–Cl(2)	2.7535(13)	P(3)–O(9)	1.553(4)
Nd(1)–Cl(3)	2.7987(14)	P(3)–O(8)	1.568(4)
Nd(1)–O(4)	2.357(4)	P(3)–O(7)	1.477(4)
Nd(1)–O(7)	2.388(4)	P(3)–C(43)	1.778(5)
Nd(1)–O(10)	2.551(4)	Br(3)–C(51)	1.899(6)
C(1)–P(1)	1.795(6)	C(3)–C(4)	1.417(8)
C(1)–C(2)	1.524(7)	C(3)–C(8)	1.451(8)
Br(1)–C(9)	1.909(5)	C(4)–C(5)	1.365(8)
O(1)–P(1)	1.483(3)	O(9)–C(61)	1.466(6)
P(1)–O(2)	1.548(4)	C(9)–C(8)	1.383(8)
P(1)–O(3)	1.565(4)	C(9)–C(10)	1.400(8)
O(2)–C(16)	1.484(6)	O(8)–C(58)	1.478(6)
P(2)–O(4)	1.488(4)	C(8)–C(7)	1.447(8)
P(2)–O(6)	1.564(4)	C(7)–C(6)	1.339(9)
P(2)–O(5)	1.550(4)	O(6)–C(40)	1.474(7)
P(2)–C(22)	1.793(5)	C(6)–C(5)	1.409(9)
Br(2)–C(30)	1.913(6)	O(5)–C(37)	1.471(6)
C(2)–C(3)	1.395(8)	C(21)–C(19)	1.503(8)

C(20)–C(19)	1.506(8)	C(56)–C(57)	1.442(8)
C(22)–C(23)	1.511(8)	C(57)–C(44)	1.410(8)
C(23)–C(24)	1.398(8)	C(58)–C(59)	1.493(9)
C(23)–C(36)	1.402(8)	C(58)–C(60)	1.511(9)
C(10)–C(11)	1.422(9)	C(61)–C(62)	1.488(9)
C(10)–C(15)	1.437(7)	C(61)–C(63)	1.484(8)
O(10)–H(10A)	0.89(6)	C(50)–C(49)	1.417(8)
O(10)–H(10B)	0.77(9)	C(50)–C(45)	1.444(8)
C(11)–C(12)	1.364(9)	C(49)–C(48)	1.348(8)
C(12)–C(13)	1.409(9)	C(48)–C(47)	1.420(9)
C(13)–C(14)	1.366(9)	C(47)–C(46)	1.357(8)
C(14)–C(15)	1.431(8)	C(46)–C(45)	1.425(8)
C(18)–C(16)	1.503(8)	C(45)–C(44)	1.415(8)
C(17)–C(16)	1.506(8)	C(44)–C(43)	1.509(8)
C(24)–C(25)	1.432(8)		
C(24)–C(29)	1.454(8)	O(1)–Nd(1)–Cl(1)	81.32(10)
C(25)–C(26)	1.358(8)	O(1)–Nd(1)–Cl(2)	144.86(9)
C(31)–C(30)	1.384(8)	O(1)–Nd(1)–Cl(3)	75.16(9)
C(31)–C(32)	1.437(8)	O(1)–Nd(1)–O(10)	72.08(13)
C(31)–C(36)	1.439(8)	Cl(1)–Nd(1)–Cl(2)	95.81(4)
C(30)–C(29)	1.384(8)	Cl(1)–Nd(1)–Cl(3)	112.42(4)
C(29)–C(28)	1.430(8)	Cl(2)–Nd(1)–Cl(3)	135.92(4)
C(26)–C(27)	1.399(9)	O(4)–Nd(1)–O(1)	89.61(12)
C(32)–C(33)	1.358(9)	O(4)–Nd(1)–Cl(1)	160.38(9)
C(33)–C(34)	1.413(9)	O(4)–Nd(1)–Cl(2)	81.62(9)
C(27)–C(28)	1.342(9)	O(4)–Nd(1)–Cl(3)	81.57(9)
C(34)–C(35)	1.354(8)	O(4)–Nd(1)–O(7)	116.14(12)
C(41)–C(40)	1.514(8)	O(4)–Nd(1)–O(10)	78.21(13)
C(40)–C(42)	1.509(9)	O(7)–Nd(1)–O(1)	136.67(12)
C(39)–C(37)	1.502(9)	O(7)–Nd(1)–Cl(1)	81.73(9)
C(38)–C(37)	1.484(9)	O(7)–Nd(1)–Cl(2)	76.43(9)
C(36)–C(35)	1.442(8)	O(7)–Nd(1)–Cl(3)	75.10(9)
C(51)–C(52)	1.393(8)	O(7)–Nd(1)–O(10)	143.59(13)
C(51)–C(50)	1.398(8)	O(10)–Nd(1)–Cl(1)	82.44(10)
C(52)–C(53)	1.435(8)	O(10)–Nd(1)–Cl(2)	72.82(10)
C(52)–C(57)	1.423(8)	O(10)–Nd(1)–Cl(3)	141.31(10)
C(53)–C(54)	1.351(8)	C(2)–C(1)–P(1)	118.0(4)
C(54)–C(55)	1.402(9)	P(1)–O(1)–Nd(1)	155.9(2)
C(55)–C(56)	1.346(8)	O(1)–P(1)–C(1)	112.0(2)

O(1)–P(1)–O(2)	109.3(2)	C(37)–O(5)–P(2)	122.4(3)
O(1)–P(1)–O(3)	113.3(2)	C(4)–C(5)–C(6)	119.7(6)
O(2)–P(1)–C(1)	109.1(2)	C(23)–C(22)–P(2)	116.1(4)
O(2)–P(1)–O(3)	106.7(2)	C(24)–C(23)–C(22)	119.2(5)
O(3)–P(1)–C(1)	106.2(2)	C(24)–C(23)–C(36)	120.2(5)
C(16)–O(2)–P(1)	127.0(3)	C(36)–C(23)–C(22)	120.6(5)
O(4)–P(2)–O(6)	111.8(2)	C(9)–C(10)–C(11)	123.0(5)
O(4)–P(2)–O(5)	115.9(2)	C(9)–C(10)–C(15)	118.0(5)
O(4)–P(2)–C(22)	110.5(2)	C(11)–C(10)–C(15)	119.0(5)
O(6)–P(2)–C(22)	110.5(2)	H(10A)–O(10)–H(10B)	108(8)
O(5)–P(2)–O(6)	103.8(2)	C(12)–C(11)–C(10)	121.1(5)
O(5)–P(2)–C(22)	103.9(2)	C(11)–C(12)–C(13)	119.9(6)
C(3)–C(2)–C(1)	120.4(5)	C(14)–C(13)–C(12)	121.2(6)
C(3)–C(2)–C(15)	120.7(5)	C(13)–C(14)–C(15)	120.7(5)
C(15)–C(2)–C(1)	118.9(5)	O(3)–C(19)–C(21)	107.2(5)
C(19)–O(3)–P(1)	120.5(3)	O(3)–C(19)–C(20)	106.8(4)
O(9)–P(3)–O(8)	106.9(2)	C(21)–C(19)–C(20)	114.7(5)
O(9)–P(3)–C(43)	104.1(2)	O(2)–C(16)–C(18)	105.8(4)
O(8)–P(3)–C(43)	105.5(2)	O(2)–C(16)–C(17)	108.3(4)
O(7)–P(3)–O(9)	113.2(2)	C(18)–C(16)–C(17)	111.8(5)
O(7)–P(3)–O(8)	112.2(2)	C(2)–C(15)–C(10)	119.8(5)
O(7)–P(3)–C(43)	114.2(2)	C(2)–C(15)–C(14)	122.5(5)
C(2)–C(3)–C(4)	123.0(5)	C(14)–C(15)–C(10)	117.7(5)
C(2)–C(3)–C(8)	119.6(5)	C(23)–C(24)–C(25)	123.1(5)
C(4)–C(3)–C(8)	117.5(5)	C(23)–C(24)–C(29)	119.6(5)
P(2)–O(4)–Nd(1)	147.7(2)	C(25)–C(24)–C(29)	117.3(5)
C(5)–C(4)–C(3)	122.3(5)	C(26)–C(25)–C(24)	121.6(6)
C(61)–O(9)–P(3)	123.6(3)	C(30)–C(31)–C(32)	123.3(5)
C(8)–C(9)–Br(1)	117.9(4)	C(30)–C(31)–C(36)	117.8(5)
C(8)–C(9)–C(10)	123.0(5)	C(32)–C(31)–C(36)	118.8(5)
C(10)–C(9)–Br(1)	119.0(4)	C(31)–C(30)–Br(2)	118.0(4)
C(58)–O(8)–P(3)	123.8(4)	C(29)–C(30)–Br(2)	118.5(4)
C(9)–C(8)–C(3)	118.4(5)	C(29)–C(30)–C(31)	123.5(5)
C(9)–C(8)–C(7)	123.8(5)	C(30)–C(29)–C(24)	118.2(5)
C(7)–C(8)–C(3)	117.9(5)	C(30)–C(29)–C(28)	124.1(5)
P(3)–O(7)–Nd(1)	140.2(2)	C(28)–C(29)–C(24)	117.6(5)
C(6)–C(7)–C(8)	121.1(5)	C(25)–C(26)–C(27)	120.8(6)
C(40)–O(6)–P(2)	121.3(3)	C(33)–C(32)–C(31)	120.8(6)
C(7)–C(6)–C(5)	121.3(5)	C(32)–C(33)–C(34)	120.5(6)

C(28)–C(27)–C(26)	120.4(6)	C(52)–C(57)–C(56)	117.6(5)
C(27)–C(28)–C(29)	122.2(6)	C(44)–C(57)–C(52)	120.4(5)
C(35)–C(34)–C(33)	120.9(6)	C(44)–C(57)–C(56)	122.0(5)
O(6)–C(40)–C(41)	110.2(5)	O(8)–C(58)–C(59)	108.5(5)
O(6)–C(40)–C(42)	106.5(5)	O(8)–C(58)–C(60)	106.0(5)
C(42)–C(40)–C(41)	112.2(5)	C(59)–C(58)–C(60)	113.9(5)
O(5)–C(37)–C(39)	106.2(5)	O(9)–C(61)–C(62)	109.6(5)
O(5)–C(37)–C(38)	108.1(5)	O(9)–C(61)–C(63)	106.6(5)
C(38)–C(37)–C(39)	115.4(5)	C(63)–C(61)–C(62)	113.0(6)
C(23)–C(36)–C(31)	120.7(5)	C(51)–C(50)–C(49)	124.0(5)
C(23)–C(36)–C(35)	122.0(5)	C(51)–C(50)–C(45)	117.6(5)
C(31)–C(36)–C(35)	117.4(5)	C(49)–C(50)–C(45)	118.4(5)
C(34)–C(35)–C(36)	121.4(6)	C(48)–C(49)–C(50)	122.0(5)
C(52)–C(51)–Br(3)	118.5(4)	C(49)–C(48)–C(47)	120.1(5)
C(52)–C(51)–C(50)	123.7(5)	C(46)–C(47)–C(48)	120.1(6)
C(50)–C(51)–Br(3)	117.8(4)	C(47)–C(46)–C(45)	121.8(5)
C(51)–C(52)–C(53)	122.8(5)	C(46)–C(45)–C(50)	117.5(5)
C(51)–C(52)–C(57)	118.2(5)	C(44)–C(45)–C(50)	119.8(5)
C(57)–C(52)–C(53)	118.9(5)	C(44)–C(45)–C(46)	122.7(5)
C(54)–C(53)–C(52)	121.2(6)	C(57)–C(44)–C(45)	120.3(5)
C(53)–C(54)–C(55)	119.5(6)	C(57)–C(44)–C(43)	120.4(5)
C(56)–C(55)–C(54)	122.3(6)	C(45)–C(44)–C(43)	119.3(5)
C(55)–C(56)–C(57)	120.4(6)	C(44)–C(43)–P(3)	115.3(4)

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) i2983

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. *CIF dictionary* *Interpreting this report*

Datablock: i2983

```
Bond precision:  C-C = 0.0094 A          Wavelength=0.71073

Cell:          a=13.6169(7)       b=15.8635(9)      c=17.6449(8)
               alpha=103.591(4)   beta=100.785(4)   gamma=111.726(4)
Temperature:   100 K
```

	Calculated	Reported
Volume	3279.2(3)	3279.2(3)
Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	C63 H74 Br3 Cl3 Nd O10 P3	C63 H74 Br3 Cl3 Nd O10 P3
Sum formula	C63 H74 Br3 Cl3 Nd O10 P3	C63 H74 Br3 Cl3 Nd O10 P3
Mr	1574.43	1574.45
Dx,g cm-3	1.594	1.595
Z	2	2
Mu (mm-1)	2.870	2.870
F000	1586.0	1586.0
F000'	1585.96	
h,k,lmax	17,20,22	17,20,22
Nref	14104	13831
Tmin,Tmax	0.733,0.944	0.490,0.936
Tmin'	0.512	

Correction method= # Reported T Limits: Tmin=0.490 Tmax=0.936
AbsCorr = MULTI-SCAN

Data completeness= 0.981 Theta(max)= 26.851

R(reflections)= 0.0477(9355) wR2(reflections)= 0.1118(13831)

S = 1.057 Npar= 768

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level C

PLAT220_ALERT_2_C NonSolvent Resd 1 C Ueq(max) / Ueq(min) Range 3.4 Ratio
PLAT242_ALERT_2_C Low MainMol Ueq as Compared to Neighbors of C61 Check
PLAT342_ALERT_3_C Low Bond Precision on C-C Bonds ..... 0.00944 Ang.
PLAT410_ALERT_2_C Short Intra H...H Contact H22A ..H35 . 1.97 Ang.
x,y,z = 1_555 Check
PLAT410_ALERT_2_C Short Intra H...H Contact H22B ..H25 . 1.99 Ang.
x,y,z = 1_555 Check
PLAT410_ALERT_2_C Short Intra H...H Contact H43A ..H46 . 1.99 Ang.
x,y,z = 1_555 Check
PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L= 0.600 64 Report
PLAT976_ALERT_2_C Check Calcd Resid. Dens. 1.04A From O10 -0.42 eA-3
PLAT978_ALERT_2_C Number C-C Bonds with Positive Residual Density. 0 Info

---

Alert level G

PLAT002_ALERT_2_G Number of Distance or Angle Restraints on AtSite 6 Note
PLAT083_ALERT_2_G SHELXL Second Parameter in WGHT Unusually Large 12.26 Why ?
PLAT154_ALERT_1_G The s.u.'s on the Cell Angles are Equal ..(Note) 0.004 Degree
PLAT175_ALERT_4_G The CIF-Embedded .res File Contains SAME Records 1 Report
PLAT794_ALERT_5_G Tentative Bond Valency for Nd1 (III) . 3.18 Info
PLAT883_ALERT_1_G No Info/Value for _atom_sites_solution_primary . Please Do !
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L= 0.600 209 Note
PLAT933_ALERT_2_G Number of OMIT Records in Embedded .res File ... 10 Note

2.11 Complex 19

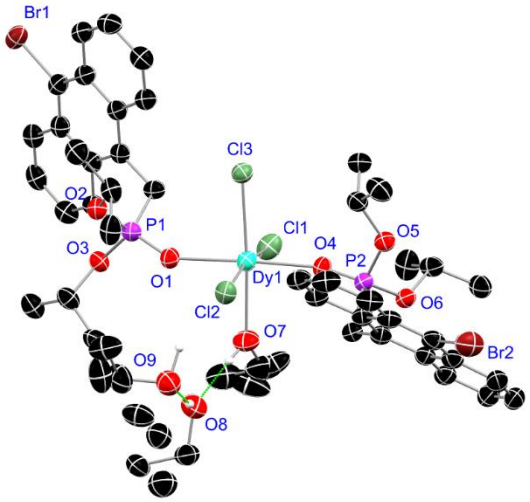

Figure 50: Asymmetric unit of **19**.

19 crystalizes as $C_{44}H_{54}Br_2Cl_3DyO_7P_2 \cdot 2EtOH$ in the space group $P\bar{1}$. The asymmetric unit contains one monomeric molecule. One of the three anthracene-phosphonate ligands is missing and replaced by a coordinating EtOH molecule. The other two EtOH molecules are co-crystalized and connected via a two-dimensional hydrogen-bond network. The protons of the EtOH hydroxy groups are refined freely. All three ethyl residues are disordered about two positions. They are partially refined with distance restraints and restraints for the anisotropic displacement parameters. The minor positions refined to 0.345(53), 0.468(14) and 0.306(11), respectively.

Bond lengths [Å] and angles [°] of **19**

Dy(1)–O(1)	2.255(3)	Br(2)–C(30)	1.909(4)
Dy(1)–Cl(1)	2.6030(10)	C(2)–C(3)	1.405(5)
Dy(1)–Cl(2)	2.6434(9)	C(2)–C(15)	1.414(5)
Dy(1)–Cl(3)	2.5885(10)	O(2)–C(16)	1.479(5)
Dy(1)–O(4)	2.251(3)	O(3)–C(19)	1.472(5)
Dy(1)–O(7)	2.354(3)	C(3)–C(4)	1.439(6)
C(1)–P(1)	1.791(4)	C(3)–C(8)	1.443(6)
C(1)–C(2)	1.515(6)	O(5)–C(37)	1.476(5)
Br(1)–C(9)	1.913(4)	C(5)–C(4)	1.353(6)
P(1)–O(1)	1.487(3)	C(5)–C(6)	1.419(7)
P(1)–O(2)	1.562(3)	C(7)–C(6)	1.366(7)
P(1)–O(3)	1.555(3)	C(7)–C(8)	1.426(6)
P(2)–O(5)	1.567(3)	O(6)–C(40)	1.487(4)
P(2)–O(4)	1.490(3)	C(9)–C(8)	1.394(6)
P(2)–O(6)	1.558(3)	C(9)–C(10)	1.391(6)
P(2)–C(22)	1.798(4)	C(10)–C(11)	1.427(6)

C(10)–C(15)	1.447(6)	O(9)–C(47)	1.489(9)
C(11)–C(12)	1.356(7)	C(48)–C(47)	1.445(11)
C(12)–C(13)	1.410(7)		
C(13)–C(14)	1.359(6)	O(1)–Dy(1)–Cl(1)	92.18(7)
C(14)–C(15)	1.431(6)	O(1)–Dy(1)–Cl(2)	88.94(7)
C(16)–C(17)	1.497(7)	O(1)–Dy(1)–Cl(3)	87.41(8)
C(16)–C(18)	1.506(7)	O(1)–Dy(1)–O(7)	88.97(12)
C(19)–C(20)	1.500(6)	Cl(1)–Dy(1)–Cl(2)	169.12(4)
C(19)–C(21)	1.492(7)	Cl(3)–Dy(1)–Cl(1)	93.28(3)
C(22)–C(23)	1.511(5)	Cl(3)–Dy(1)–Cl(2)	97.57(3)
C(23)–C(24)	1.406(6)	O(4)–Dy(1)–O(1)	175.51(9)
C(23)–C(36)	1.411(5)	O(4)–Dy(1)–Cl(1)	92.03(7)
C(24)–C(25)	1.438(6)	O(4)–Dy(1)–Cl(2)	86.63(7)
C(24)–C(29)	1.447(6)	O(4)–Dy(1)–Cl(3)	93.91(8)
C(25)–C(26)	1.363(6)	O(4)–Dy(1)–O(7)	89.77(12)
C(26)–C(27)	1.408(7)	O(7)–Dy(1)–Cl(1)	85.86(10)
C(27)–C(28)	1.349(7)	O(7)–Dy(1)–Cl(2)	83.35(9)
C(28)–C(29)	1.432(6)	O(7)–Dy(1)–Cl(3)	176.25(9)
C(29)–C(30)	1.396(6)	C(2)–C(1)–P(1)	116.1(3)
C(30)–C(31)	1.404(6)	O(1)–P(1)–C(1)	111.02(18)
C(31)–C(32)	1.428(6)	O(1)–P(1)–O(2)	112.88(16)
C(31)–C(36)	1.441(6)	O(1)–P(1)–O(3)	114.44(16)
C(32)–C(33)	1.354(7)	O(2)–P(1)–C(1)	111.80(18)
C(33)–C(34)	1.422(7)	O(3)–P(1)–C(1)	102.65(17)
C(34)–C(35)	1.355(6)	O(3)–P(1)–O(2)	103.41(15)
C(35)–C(36)	1.439(6)	P(1)–O(1)–Dy(1)	146.40(17)
C(37)–C(38)	1.501(7)	O(5)–P(2)–C(22)	110.26(17)
C(37)–C(39)	1.499(7)	O(4)–P(2)–O(5)	112.36(16)
C(40)–C(41)	1.499(6)	O(4)–P(2)–O(6)	114.16(15)
C(40)–C(42)	1.491(7)	O(4)–P(2)–C(22)	112.01(18)
O(7)–C(43)	1.458(18)	O(6)–P(2)–O(5)	104.18(15)
O(7)–H(7A)	0.88(6)	O(6)–P(2)–C(22)	103.26(16)
O(7)–C(43A)	1.449(8)	C(3)–C(2)–C(1)	119.1(3)
C(43)–C(44)	1.48(2)	C(3)–C(2)–C(15)	120.1(4)
C(43A)–C(44A)	1.478(13)	C(15)–C(2)–C(1)	120.8(3)
O(8)–C(45)	1.422(9)	C(16)–O(2)–P(1)	124.6(3)
O(8)–C(45A)	1.496(10)	C(19)–O(3)–P(1)	124.2(2)
C(45)–C(46)	1.478(12)	C(2)–C(3)–C(4)	122.2(4)
C(45A)–C(46A)	1.490(14)	C(2)–C(3)–C(8)	120.0(4)

C(4)–C(3)–C(8)	117.8(4)	C(25)–C(24)–C(29)	116.9(4)
C(37)–O(5)–P(2)	124.1(3)	C(26)–C(25)–C(24)	121.5(4)
C(4)–C(5)–C(6)	120.9(4)	C(25)–C(26)–C(27)	120.7(4)
P(2)–O(4)–Dy(1)	159.65(17)	C(28)–C(27)–C(26)	120.9(4)
C(5)–C(4)–C(3)	121.5(4)	C(27)–C(28)–C(29)	121.0(4)
C(6)–C(7)–C(8)	122.0(4)	C(28)–C(29)–C(24)	119.1(4)
C(40)–O(6)–P(2)	123.6(2)	C(30)–C(29)–C(24)	117.4(4)
C(7)–C(6)–C(5)	119.6(4)	C(30)–C(29)–C(28)	123.5(4)
C(8)–C(9)–Br(1)	118.4(3)	C(29)–C(30)–Br(2)	117.8(3)
C(10)–C(9)–Br(1)	118.5(3)	C(29)–C(30)–C(31)	123.7(4)
C(10)–C(9)–C(8)	123.1(4)	C(31)–C(30)–Br(2)	118.5(3)
C(7)–C(8)–C(3)	118.1(4)	C(30)–C(31)–C(32)	123.3(4)
C(9)–C(8)–C(3)	118.6(4)	C(30)–C(31)–C(36)	117.7(4)
C(9)–C(8)–C(7)	123.3(4)	C(32)–C(31)–C(36)	119.1(4)
C(9)–C(10)–C(11)	123.2(4)	C(33)–C(32)–C(31)	121.1(4)
C(9)–C(10)–C(15)	118.1(4)	C(32)–C(33)–C(34)	120.6(4)
C(11)–C(10)–C(15)	118.7(4)	C(35)–C(34)–C(33)	120.2(4)
C(12)–C(11)–C(10)	121.2(4)	C(34)–C(35)–C(36)	121.8(4)
C(11)–C(12)–C(13)	120.5(4)	C(23)–C(36)–C(31)	120.5(4)
C(14)–C(13)–C(12)	120.6(4)	C(23)–C(36)–C(35)	122.2(4)
C(13)–C(14)–C(15)	121.6(4)	C(35)–C(36)–C(31)	117.2(4)
O(2)–C(16)–C(17)	105.9(4)	O(5)–C(37)–C(38)	105.2(4)
O(2)–C(16)–C(18)	108.8(4)	O(5)–C(37)–C(39)	108.7(3)
C(17)–C(16)–C(18)	113.5(4)	C(39)–C(37)–C(38)	113.5(4)
C(2)–C(15)–C(10)	120.2(4)	O(6)–C(40)–C(41)	105.5(3)
C(2)–C(15)–C(14)	122.5(4)	O(6)–C(40)–C(42)	109.1(4)
C(14)–C(15)–C(10)	117.3(4)	C(42)–C(40)–C(41)	114.1(4)
O(3)–C(19)–C(20)	105.0(3)	Dy(1)–O(7)–H(7A)	120(4)
O(3)–C(19)–C(21)	109.1(4)	C(43)–O(7)–Dy(1)	134(2)
C(21)–C(19)–C(20)	113.3(4)	C(43)–O(7)–H(7A)	104(4)
C(23)–C(22)–P(2)	115.9(3)	C(43A)–O(7)–Dy(1)	126.1(9)
C(24)–C(23)–C(22)	120.4(3)	O(7)–C(43)–C(44)	107(2)
C(24)–C(23)–C(36)	119.9(4)	O(7)–C(43A)–C(44A)	112.3(10)
C(36)–C(23)–C(22)	119.8(4)	O(8)–C(45)–C(46)	112.5(8)
C(23)–C(24)–C(25)	122.2(4)	C(46A)–C(45A)–O(8)	107.7(9)
C(23)–C(24)–C(29)	120.8(4)	C(48)–C(47)–O(9)	111.3(7)

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) sv0796

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No syntax errors found. CIF dictionary Interpreting this report

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Datablock: sv0796

Bond precision: C-C = 0.0066 A Wavelength=1.54186

Cell: a=11.2192(4) b=13.3757(4) c=20.6992(7)
alpha=101.357(3) beta=98.671(3) gamma=111.903(2)
Temperature: 100 K

	Calculated	Reported
Volume	2738.39(18)	2738.39(17)
Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	C44 H54 Br2 Cl3 Dy O7 P2, 2(C2 H6 O) [+ solvent]	C44 H54 Br2 Cl3 Dy O7 P2, 2(C2 H6 O)
Sum formula	C48 H66 Br2 Cl3 Dy O9 P2 [+ solvent]	C48 H66 Br2 Cl3 Dy O9 P2
Mr	1277.60	1277.61
Dx,g cm-3	1.549	1.549
Z	2	2
Mu (mm-1)	11.311	11.311
F000	1286.0	1286.0
F000'	1269.90	
h,k,lmax	13,16,25	13,16,25
Nref	10614	10129
Tmin,Tmax	0.090,0.093	0.038,0.237
Tmin'	0.025	

Correction method= # Reported T Limits: Tmin=0.038 Tmax=0.237
AbsCorr = MULTI-SCAN

Data completeness= 0.954 Theta(max)= 71.152

R(reflections)= 0.0470(9981) wR2(reflections)= 0.1317(10129)

S = 1.038 Npar= 627

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level C

PLAT202_ALERT_3_C Isotropic non-H Atoms in Anion/Solvent ..... 3 Check
O9 C47 C48
PLAT234_ALERT_4_C Large Hirshfeld Difference O7 --C43 . 0.24 Ang.
PLAT243_ALERT_4_C High Solvent Ueq as Compared to Neighbors of O8 Check
PLAT410_ALERT_2_C Short Intra H...H Contact H1A ..H4 . 1.93 Ang.
x,y,z = 1_555 Check
PLAT410_ALERT_2_C Short Intra H...H Contact H22A ..H35 . 1.95 Ang.
x,y,z = 1_555 Check
PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L= 0.600 176 Report
PLAT918_ALERT_3_C Reflection(s) with I(obs) much Smaller I(calc) . 23 Check
PLAT975_ALERT_2_C Check Calcd Resid. Dens. 0.80A From O1 0.46 eA-3
PLAT976_ALERT_2_C Check Calcd Resid. Dens. 0.89A From O1 -0.50 eA-3
PLAT976_ALERT_2_C Check Calcd Resid. Dens. 0.89A From O1 -0.49 eA-3
PLAT977_ALERT_2_C Check Negative Difference Density on H9 -0.63 eA-3
PLAT978_ALERT_2_C Number C-C Bonds with Positive Residual Density. 0 Info

Alert level G

PLAT002_ALERT_2_G Number of Distance or Angle Restraints on AtSite 12 Note
PLAT003_ALERT_2_G Number of Uiso or Uij Restrained non-H Atoms ... 4 Report
PLAT007_ALERT_5_G Number of Unrefined Donor-H Atoms ..... 3 Report
PLAT172_ALERT_4_G The CIF-Embedded .res File Contains DFIX Records 1 Report
PLAT175_ALERT_4_G The CIF-Embedded .res File Contains SAME Records 4 Report
PLAT178_ALERT_4_G The CIF-Embedded .res File Contains SIMU Records 1 Report
PLAT187_ALERT_4_G The CIF-Embedded .res File Contains RIGU Records 1 Report
PLAT232_ALERT_2_G Hirshfeld Test Diff (M-X) Dy1 --O4 . 5.0 s.u.
PLAT301_ALERT_3_G Main Residue Disorder .....(Resd 1) 3% Note
PLAT302_ALERT_4_G Anion/Solvent/Minor-Residue Disorder (Resd 2) 67% Note
PLAT398_ALERT_2_G Deviating C-O-C Angle From 120 for O8 43.7 Degree
PLAT417_ALERT_2_G Short Inter D-H..H-D H8A ..H9 . 1.71 Ang.
x,y,z = 1_555 Check
PLAT417_ALERT_2_G Short Inter D-H..H-D H9 ..H8B . 1.72 Ang.
x,y,z = 1_555 Check
PLAT605_ALERT_4_G Largest Solvent Accessible VOID in the Structure 35 A**3
PLAT722_ALERT_1_G Angle Calc 111.00, Rep 109.50 Dev... 1.50 Degree
H44A -C44 -H44C 1.555 1.555 1.555 # 217 Check
PLAT860_ALERT_3_G Number of Least-Squares Restraints ..... 49 Note
PLAT868_ALERT_4_G ALERTS Due to the Use of _smtbx_masks Suppressed ! Info
PLAT883_ALERT_1_G No Info/Value for _atom_sites_solution_primary . Please Do!
PLAT910_ALERT_3_G Missing # of FCF Reflection(s) Below Theta(Min). 1 Note
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L= 0.600 308 Note

2.12 Complex 20

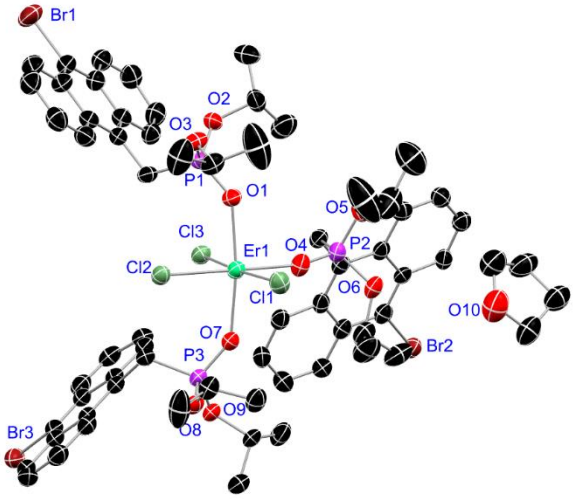

Figure 51: Asymmetric unit of **20**.

20 crystalizes as $C_{63}H_{72}Br_3Cl_3ErO_9P_3 \cdot$ THF in the space group $P2_1/c$. The asymmetric unit contains one monomer and a co-crystalized THF lattice molecule. High residual electron densities located close to very heavy atoms (here Er or Br) can be expected due to minor crystal quality, therefore causing a B-Alert upon cif-file check.

Bond lengths [Å] and angles [°] of **20**

Er(1)–O(1)	2.234(4)	O(2)–C(16)	1.468(7)
Er(1)–Cl(1)	2.5746(14)	Br(3)–C(51)	1.913(6)
Er(1)–Cl(2)	2.6270(12)	P(3)–O(7)	1.504(4)
Er(1)–Cl(3)	2.6472(13)	P(3)–O(9)	1.561(4)
Er(1)–O(4)	2.220(4)	P(3)–C(43)	1.798(6)
Er(1)–O(7)	2.235(4)	P(3)–O(8)	1.562(4)
P(1)–C(1)	1.792(6)	C(3)–C(4)	1.420(9)
P(1)–O(1)	1.493(4)	C(3)–C(8)	1.440(8)
P(1)–O(2)	1.551(4)	O(3)–C(19)	1.484(7)
P(1)–O(3)	1.559(4)	C(4)–C(5)	1.359(9)
C(1)–C(2)	1.520(7)	C(7)–C(6)	1.350(11)
Br(1)–C(9)	1.914(6)	C(7)–C(8)	1.429(9)
Br(2)–C(30)	1.902(5)	O(6)–C(40)	1.465(9)
P(2)–O(4)	1.482(4)	C(6)–C(5)	1.420(11)
P(2)–O(6)	1.557(4)	O(5)–C(37)	1.460(9)
P(2)–O(5)	1.563(5)	C(9)–C(10)	1.392(9)
P(2)–C(22)	1.791(6)	C(9)–C(8)	1.405(9)
C(2)–C(3)	1.408(8)	O(9)–C(58)	1.486(6)
C(2)–C(15)	1.409(8)	O(10)–C(64)	1.457(12)

O(10)–C(67)	1.420(10)	C(48)–C(49)	1.363(10)
C(10)–C(11)	1.437(9)	C(49)–C(50)	1.436(9)
C(10)–C(15)	1.424(8)	C(50)–C(51)	1.396(9)
C(11)–C(12)	1.362(10)	C(51)–C(52)	1.370(10)
C(12)–C(13)	1.415(9)	C(52)–C(53)	1.427(9)
C(13)–C(14)	1.348(9)	C(52)–C(57)	1.467(9)
C(14)–C(15)	1.435(8)	C(53)–C(54)	1.354(11)
C(16)–C(17)	1.508(9)	C(54)–C(55)	1.409(11)
C(16)–C(18)	1.515(11)	C(55)–C(56)	1.373(9)
C(19)–C(20)	1.490(12)	C(56)–C(57)	1.419(9)
C(19)–C(21)	1.476(10)	C(58)–C(59)	1.501(9)
C(22)–C(23)	1.519(7)	C(58)–C(60)	1.497(9)
C(24)–C(23)	1.397(8)	C(61)–C(62)	1.495(10)
C(24)–C(25)	1.438(8)	C(61)–C(63)	1.498(9)
C(24)–C(29)	1.459(8)	C(61)–O(8)	1.481(8)
C(23)–C(36)	1.413(7)	C(64)–C(65)	1.484(14)
C(25)–C(26)	1.369(9)	C(65)–C(66)	1.512(11)
C(26)–C(27)	1.403(9)	C(66)–C(67)	1.494(13)
C(27)–C(28)	1.351(9)		
C(28)–C(29)	1.414(8)	O(1)–Er(1)–Cl(1)	94.49(10)
C(29)–C(30)	1.417(8)	O(1)–Er(1)–Cl(2)	87.25(10)
C(30)–C(31)	1.383(8)	O(1)–Er(1)–Cl(3)	84.99(10)
C(31)–C(32)	1.442(7)	O(1)–Er(1)–O(7)	170.83(14)
C(31)–C(36)	1.433(7)	Cl(1)–Er(1)–Cl(2)	92.22(4)
C(32)–C(33)	1.358(8)	Cl(1)–Er(1)–Cl(3)	172.79(4)
C(33)–C(34)	1.413(8)	Cl(2)–Er(1)–Cl(3)	94.93(4)
C(34)–C(35)	1.353(8)	O(4)–Er(1)–O(1)	92.13(15)
C(35)–C(36)	1.439(8)	O(4)–Er(1)–Cl(1)	87.00(11)
C(37)–C(38)	1.546(16)	O(4)–Er(1)–Cl(2)	178.96(11)
C(37)–C(39)	1.396(16)	O(4)–Er(1)–Cl(3)	85.84(10)
C(40)–C(41)	1.518(10)	O(4)–Er(1)–O(7)	92.21(15)
C(40)–C(42)	1.501(10)	O(7)–Er(1)–Cl(1)	93.80(10)
C(43)–C(44)	1.522(8)	O(7)–Er(1)–Cl(2)	88.53(10)
C(44)–C(45)	1.403(8)	O(7)–Er(1)–Cl(3)	87.28(10)
C(44)–C(57)	1.405(8)	O(1)–P(1)–C(1)	113.4(2)
C(45)–C(46)	1.423(8)	O(1)–P(1)–O(2)	114.4(2)
C(45)–C(50)	1.448(8)	O(1)–P(1)–O(3)	111.1(2)
C(46)–C(47)	1.378(9)	O(2)–P(1)–C(1)	101.7(2)
C(47)–C(48)	1.393(10)	O(2)–P(1)–O(3)	105.9(2)

O(3)–P(1)–C(1)	109.7(3)	C(12)–C(11)–C(10)	121.1(6)
C(2)–C(1)–P(1)	115.7(4)	C(11)–C(12)–C(13)	119.8(6)
P(1)–O(1)–Er(1)	142.5(2)	C(14)–C(13)–C(12)	120.7(6)
O(4)–P(2)–O(6)	112.6(2)	C(13)–C(14)–C(15)	122.1(6)
O(4)–P(2)–O(5)	114.8(2)	O(2)–C(16)–C(17)	105.4(5)
O(4)–P(2)–C(22)	111.3(2)	O(2)–C(16)–C(18)	108.8(6)
O(6)–P(2)–O(5)	104.3(2)	C(17)–C(16)–C(18)	113.3(5)
O(6)–P(2)–C(22)	110.6(2)	C(2)–C(15)–C(10)	120.1(5)
O(5)–P(2)–C(22)	102.7(3)	C(2)–C(15)–C(14)	122.6(5)
C(3)–C(2)–C(1)	119.3(5)	C(10)–C(15)–C(14)	117.3(5)
C(3)–C(2)–C(15)	120.7(5)	O(3)–C(19)–C(20)	108.6(6)
C(15)–C(2)–C(1)	120.0(5)	C(21)–C(19)–O(3)	107.7(6)
C(16)–O(2)–P(1)	127.4(4)	C(21)–C(19)–C(20)	114.4(8)
O(7)–P(3)–O(9)	113.4(2)	C(23)–C(22)–P(2)	114.5(4)
O(7)–P(3)–C(43)	110.8(2)	C(23)–C(24)–C(25)	123.4(5)
O(7)–P(3)–O(8)	111.3(2)	C(23)–C(24)–C(29)	120.0(5)
O(9)–P(3)–C(43)	106.9(2)	C(25)–C(24)–C(29)	116.6(5)
O(9)–P(3)–O(8)	104.4(2)	C(24)–C(23)–C(22)	119.7(5)
O(8)–P(3)–C(43)	109.8(2)	C(24)–C(23)–C(36)	120.7(5)
C(2)–C(3)–C(4)	123.0(6)	C(36)–C(23)–C(22)	119.6(5)
C(2)–C(3)–C(8)	119.6(5)	C(26)–C(25)–C(24)	121.9(5)
C(4)–C(3)–C(8)	117.4(5)	C(25)–C(26)–C(27)	120.4(5)
C(19)–O(3)–P(1)	123.7(4)	C(28)–C(27)–C(26)	120.3(6)
P(2)–O(4)–Er(1)	165.0(3)	C(27)–C(28)–C(29)	122.3(6)
C(5)–C(4)–C(3)	122.4(6)	C(28)–C(29)–C(24)	118.5(5)
C(6)–C(7)–C(8)	121.5(6)	C(28)–C(29)–C(30)	124.3(5)
P(3)–O(7)–Er(1)	142.9(2)	C(30)–C(29)–C(24)	117.2(5)
C(40)–O(6)–P(2)	122.2(4)	C(29)–C(30)–Br(2)	117.3(4)
C(7)–C(6)–C(5)	120.3(6)	C(31)–C(30)–Br(2)	119.3(4)
C(37)–O(5)–P(2)	127.3(6)	C(31)–C(30)–C(29)	123.3(5)
C(4)–C(5)–C(6)	119.8(6)	C(30)–C(31)–C(32)	123.0(5)
C(10)–C(9)–Br(1)	119.1(5)	C(30)–C(31)–C(36)	118.5(5)
C(10)–C(9)–C(8)	122.9(6)	C(36)–C(31)–C(32)	118.4(5)
C(8)–C(9)–Br(1)	118.0(4)	C(33)–C(32)–C(31)	121.4(5)
C(58)–O(9)–P(3)	117.3(3)	C(32)–C(33)–C(34)	120.2(5)
C(67)–O(10)–C(64)	109.6(7)	C(35)–C(34)–C(33)	120.4(6)
C(9)–C(10)–C(11)	122.3(6)	C(34)–C(35)–C(36)	122.0(5)
C(9)–C(10)–C(15)	118.6(6)	C(23)–C(36)–C(31)	120.2(5)
C(15)–C(10)–C(11)	119.0(6)	C(23)–C(36)–C(35)	122.3(5)

C(31)–C(36)–C(35)	117.5(5)	C(51)–C(52)–C(57)	118.4(5)
O(5)–C(37)–C(38)	102.9(9)	C(53)–C(52)–C(57)	117.5(6)
C(39)–C(37)–O(5)	113.0(8)	C(54)–C(53)–C(52)	122.2(7)
C(39)–C(37)–C(38)	108.1(10)	C(53)–C(54)–C(55)	120.3(6)
O(6)–C(40)–C(41)	104.7(6)	C(56)–C(55)–C(54)	120.6(7)
O(6)–C(40)–C(42)	109.3(6)	C(55)–C(56)–C(57)	121.4(6)
C(42)–C(40)–C(41)	113.6(6)	C(44)–C(57)–C(52)	119.4(6)
C(44)–C(43)–P(3)	119.8(4)	C(44)–C(57)–C(56)	122.6(6)
C(45)–C(44)–C(43)	118.6(5)	C(56)–C(57)–C(52)	118.0(5)
C(45)–C(44)–C(57)	120.6(5)	O(9)–C(58)–C(59)	107.4(5)
C(57)–C(44)–C(43)	120.5(5)	O(9)–C(58)–C(60)	108.0(5)
C(44)–C(45)–C(46)	122.2(5)	C(60)–C(58)–C(59)	114.2(5)
C(44)–C(45)–C(50)	119.8(5)	C(62)–C(61)–C(63)	112.7(6)
C(46)–C(45)–C(50)	118.0(5)	O(8)–C(61)–C(62)	105.7(6)
C(47)–C(46)–C(45)	121.6(6)	O(8)–C(61)–C(63)	108.7(5)
C(46)–C(47)–C(48)	120.5(6)	O(10)–C(64)–C(65)	104.7(7)
C(49)–C(48)–C(47)	120.1(6)	C(64)–C(65)–C(66)	103.4(7)
C(48)–C(49)–C(50)	122.3(6)	C(67)–C(66)–C(65)	100.9(7)
C(49)–C(50)–C(45)	117.3(5)	O(10)–C(67)–C(66)	105.4(7)
C(51)–C(50)–C(45)	118.3(6)	C(61)–O(8)–P(3)	123.5(4)
C(51)–C(50)–C(49)	124.4(6)	C(7)–C(8)–C(3)	118.6(6)
C(50)–C(51)–Br(3)	117.3(5)	C(9)–C(8)–C(3)	118.1(5)
C(52)–C(51)–Br(3)	119.1(5)	C(9)–C(8)–C(7)	123.3(6)
C(52)–C(51)–C(50)	123.5(6)		
C(51)–C(52)–C(53)	124.1(6)		

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) sv0773

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. *CIF dictionary* *Interpreting this report*

Datablock: sv0773

```
Bond precision:  C-C = 0.0100 A          Wavelength=1.54186

Cell:            a=21.3578(6)     b=16.3493(6)        c=22.3921(6)
                 alpha=90         beta=115.243(2)     gamma=90
Temperature:     100 K
```

	Calculated	Reported
Volume	7072.3(4)	7072.3(4)
Space group	P 21/c	P 1 21/c 1
Hall group	-P 2ybc	-P 2ybc
Moiety formula	C63 H72 Br3 Cl3 Er O9 P3, C4 H8 O	C63 H72 Br3 Cl3 Er O9 P3, C4 H8 O
Sum formula	C67 H80 Br3 Cl3 Er O10 P3	C67 H80 Br3 Cl3 Er O10 P3
Mr	1651.53	1651.56
Dx,g cm-3	1.551	1.551
Z	4	4
Mu (mm-1)	6.311	6.311
F000	3324.0	3324.0
F000'	3292.75	
h,k,lmax	26,19,27	25,19,26
Nref	13399	12928
Tmin,Tmax	0.361,0.881	0.105,0.743
Tmin'	0.108	

Correction method= # Reported T Limits: Tmin=0.105 Tmax=0.743
AbsCorr = MULTI-SCAN

Data completeness= 0.965 Theta(max)= 69.984

R(reflections)= 0.0661(10927) wR2(reflections)= 0.1915(12928)

S = 1.038 Npar= 796

The following ALERTS were generated. Each ALERT has the format

test-name_ALERT_alert-type_alert-level.

Click on the hyperlinks for more details of the test.

Alert level B

PLAT971_ALERT_2_B Check Calcd Resid. Dens. 1.11A From Er1 3.22 eA-3
PLAT971_ALERT_2_B Check Calcd Resid. Dens. 1.06A From Er1 3.02 eA-3

Alert level C

PLAT029_ALERT_3_C _diffrn_measured_fraction_theta_full value Low . 0.976 Why?
PLAT094_ALERT_2_C Ratio of Maximum / Minimum Residual Density .... 2.11 Report
PLAT220_ALERT_2_C NonSolvent Resd 1 C Ueq(max) / Ueq(min) Range 4.0 Ratio
PLAT222_ALERT_3_C NonSolvent Resd 1 H Uiso(max)/Uiso(min) Range 4.1 Ratio
PLAT234_ALERT_4_C Large Hirshfeld Difference C37 --C38 . 0.22 Ang.
PLAT242_ALERT_2_C Low MainMol Ueq as Compared to Neighbors of C19 Check
PLAT342_ALERT_3_C Low Bond Precision on C-C Bonds ..... 0.01005 Ang.
PLAT360_ALERT_2_C Short C(sp3)-C(sp3) Bond C37 - C39 . 1.39 Ang.
PLAT410_ALERT_2_C Short Intra H...H Contact H1B ..H4 . 1.97 Ang.
x,y,z = 1_555 Check
PLAT410_ALERT_2_C Short Intra H...H Contact H22A ..H25 . 1.98 Ang.
x,y,z = 1_555 Check
PLAT410_ALERT_2_C Short Intra H...H Contact H43B ..H46 . 1.97 Ang.
x,y,z = 1_555 Check
PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L= 0.600 310 Report
PLAT918_ALERT_3_C Reflection(s) with I(obs) much Smaller I(calc) . 1 Check

PLAT971_ALERT_2_C Check Calcd Resid. Dens. 1.06A   From Er1 1.67 eA-3
PLAT971_ALERT_2_C Check Calcd Resid. Dens. 1.15A   From Br2 1.66 eA-3
PLAT971_ALERT_2_C Check Calcd Resid. Dens. 1.14A   From Er1 1.59 eA-3
PLAT971_ALERT_2_C Check Calcd Resid. Dens. 1.13A   From Br2 1.57 eA-3
PLAT971_ALERT_2_C Check Calcd Resid. Dens. 0.81A   From Br3 1.54 eA-3
PLAT973_ALERT_2_C Check Calcd Positive Resid. Density on Er1 1.24 eA-3
PLAT977_ALERT_2_C Check Negative Difference Density on H38A -0.32 eA-3
PLAT978_ALERT_2_C Number C-C Bonds with Positive Residual Density. 0 Info

---

Alert level G

PLAT072_ALERT_2_G SHELXL First Parameter in WGHT Unusually Large 0.13 Report
PLAT083_ALERT_2_G SHELXL Second Parameter in WGHT Unusually Large 11.95 Why ?
PLAT398_ALERT_2_G Deviating C-O-C Angle From 120 for O10 109.7 Degree
PLAT432_ALERT_2_G Short Inter X...Y Contact Br3 ..C12 3.31 Ang.
1-x,1-y,1-z = 3_666 Check
PLAT790_ALERT_4_G Centre of Gravity not Within Unit Cell: Resd. # 2 Note
C4 H8 O
PLAT794_ALERT_5_G Tentative Bond Valency for Er1 (III) . 3.19 Info
PLAT883_ALERT_1_G No Info/Value for _atom_sites_solution_primary . Please Do !
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L= 0.600 148 Note
PLAT933_ALERT_2_G Number of OMIT Records in Embedded .res File ... 6 Note
PLAT960_ALERT_3_G Number of Intensities with I < - 2*sig(I) ... 33 Check

2.13 Complex 31

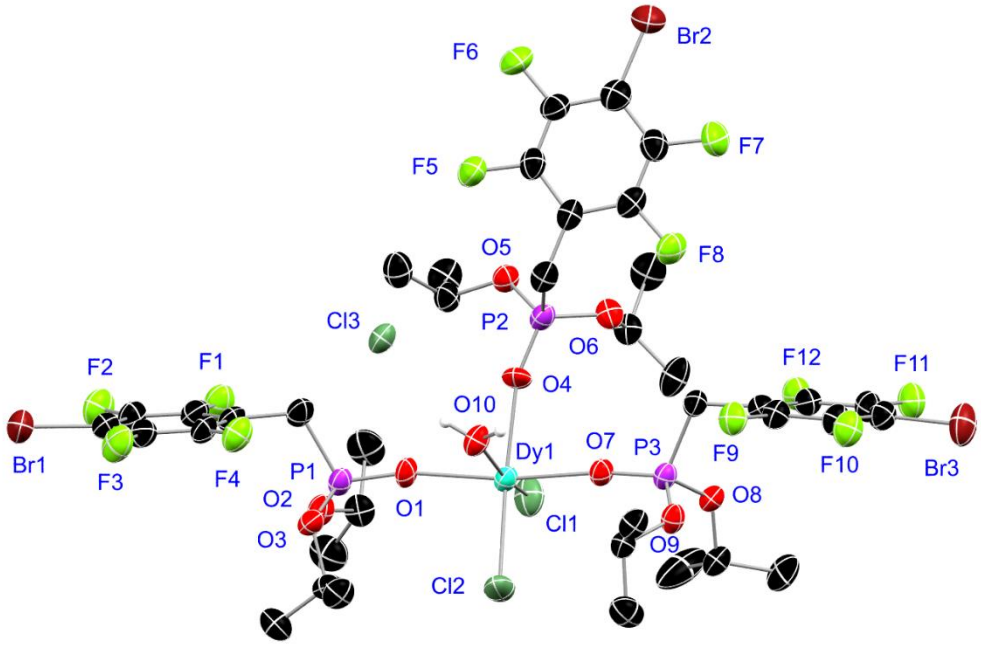

Figure 52: Asymmetric unit of **31**.

31 crystalizes as $C_{39}H_{50}Br_3Cl_2DyF_{12}O_{10}P_3 \cdot Cl^-$ in the space group $P\bar{1}$ and contains one molecule in the asymmetric unit. A H_2O ligand replaces one of the chloride anions causing the formation of a partially solvent separated ion pair.

Bond lengths [Å] and angles [°] of **31**

C(1)–C(2)	1.512(12)	Br(2)–C(18)	1.861(11)
C(1)–P(1)	1.775(9)	O(2)–C(8)	1.490(11)
F(1)–C(3)	1.354(11)	P(2)–O(4)	1.465(6)
Br(1)–C(5)	1.888(10)	P(2)–O(6)	1.548(6)
O(1)–P(1)	1.479(6)	P(2)–O(5)	1.564(6)
O(1)–Dy(1)	2.251(5)	P(2)–C(14)	1.783(9)
P(1)–O(3)	1.544(6)	C(3)–C(4)	1.382(13)
P(1)–O(2)	1.555(7)	F(3)–C(6)	1.336(11)
Cl(1)–Dy(1)	2.587(3)	Br(3)–C(31)	1.864(9)
Dy(1)–O(7)	2.253(6)	O(3)–C(11)	1.472(10)
Dy(1)–O(4)	2.265(6)	P(3)–O(7)	1.487(6)
Dy(1)–O(10)	2.331(7)	P(3)–O(9)	1.548(6)
Dy(1)–Cl(2)	2.561(2)	P(3)–O(8)	1.550(6)
C(2)–C(3)	1.363(13)	P(3)–C(27)	1.781(9)
C(2)–C(7)	1.372(13)	C(4)–C(5)	1.349(14)
F(2)–C(4)	1.331(10)	F(4)–C(7)	1.344(10)

C(5)–C(6)	1.382(14)	C(34)–C(36)	1.491(14)
F(5)–C(16)	1.345(10)	C(37)–C(38)	1.487(13)
O(5)–C(21)	1.458(10)	C(37)–C(39)	1.487(12)
C(6)–C(7)	1.371(13)		
F(6)–C(17)	1.344(10)	C(2)–C(1)–P(1)	115.1(7)
O(6)–C(24)	1.480(10)	P(1)–O(1)–Dy(1)	165.8(4)
F(7)–C(19)	1.356(11)	O(1)–P(1)–O(3)	115.2(4)
C(8)–C(10)	1.489(14)	O(1)–P(1)–O(2)	113.4(4)
C(8)–C(9)	1.501(15)	O(3)–P(1)–O(2)	105.4(4)
F(8)–C(20)	1.359(10)	O(1)–P(1)–C(1)	111.2(4)
O(8)–C(34)	1.466(11)	O(3)–P(1)–C(1)	101.9(4)
F(9)–C(29)	1.341(10)	O(2)–P(1)–C(1)	108.9(4)
O(9)–C(37)	1.489(11)	O(1)–Dy(1)–O(7)	169.6(2)
F(10)–C(30)	1.336(10)	O(1)–Dy(1)–O(4)	90.3(2)
O(10)–H(1O)	0.83(11)	O(7)–Dy(1)–O(4)	83.9(2)
O(10)–H(2O)	0.81(9)	O(1)–Dy(1)–O(10)	84.9(2)
F(11)–C(32)	1.341(10)	O(7)–Dy(1)–O(10)	85.6(2)
C(11)–C(12)	1.491(13)	O(4)–Dy(1)–O(10)	79.8(2)
C(11)–C(13)	1.507(13)	O(1)–Dy(1)–Cl(2)	94.63(17)
F(12)–C(33)	1.342(10)	O(7)–Dy(1)–Cl(2)	89.85(16)
C(14)–C(15)	1.474(13)	O(4)–Dy(1)–Cl(2)	169.89(18)
C(15)–C(20)	1.381(13)	O(10)–Dy(1)–Cl(2)	91.78(19)
C(15)–C(16)	1.398(13)	O(1)–Dy(1)–Cl(1)	89.04(17)
C(16)–C(17)	1.359(14)	O(7)–Dy(1)–Cl(1)	99.31(16)
C(17)–C(18)	1.370(14)	O(4)–Dy(1)–Cl(1)	87.85(18)
C(18)–C(19)	1.394(14)	O(10)–Dy(1)–Cl(1)	166.23(18)
C(19)–C(20)	1.371(14)	Cl(2)–Dy(1)–Cl(1)	101.05(10)
C(21)–C(22)	1.505(13)	C(3)–C(2)–C(7)	116.6(8)
C(21)–C(23)	1.512(13)	C(3)–C(2)–C(1)	122.6(9)
C(27)–C(28)	1.521(12)	C(7)–C(2)–C(1)	120.8(8)
C(26)–C(24)	1.492(14)	C(8)–O(2)–P(1)	123.9(6)
C(25)–C(24)	1.466(13)	O(4)–P(2)–O(6)	115.6(4)
C(28)–C(29)	1.356(13)	O(4)–P(2)–O(5)	111.2(4)
C(28)–C(33)	1.376(13)	O(6)–P(2)–O(5)	105.5(3)
C(29)–C(30)	1.380(12)	O(4)–P(2)–C(14)	112.9(4)
C(30)–C(31)	1.388(13)	O(6)–P(2)–C(14)	102.6(4)
C(31)–C(32)	1.369(13)	O(5)–P(2)–C(14)	108.4(4)
C(32)–C(33)	1.390(13)	F(1)–C(3)–C(2)	120.1(8)
C(34)–C(35)	1.449(15)	F(1)–C(3)–C(4)	117.4(8)

C(2)–C(3)–C(4)	122.5(9)	F(5)–C(16)–C(17)	118.7(8)
C(11)–O(3)–P(1)	123.9(5)	F(5)–C(16)–C(15)	118.4(9)
O(7)–P(3)–O(9)	113.4(3)	C(17)–C(16)–C(15)	122.9(9)
O(7)–P(3)–O(8)	114.7(3)	F(6)–C(17)–C(16)	119.3(9)
O(9)–P(3)–O(8)	105.7(3)	F(6)–C(17)–C(18)	118.2(9)
O(7)–P(3)–C(27)	112.1(4)	C(16)–C(17)–C(18)	122.5(9)
O(9)–P(3)–C(27)	108.6(4)	C(17)–C(18)–C(19)	115.7(10)
O(8)–P(3)–C(27)	101.6(4)	C(17)–C(18)–Br(2)	123.8(8)
F(2)–C(4)–C(5)	120.5(9)	C(19)–C(18)–Br(2)	120.5(8)
F(2)–C(4)–C(3)	119.5(9)	F(7)–C(19)–C(20)	118.3(9)
C(5)–C(4)–C(3)	119.9(9)	F(7)–C(19)–C(18)	120.3(10)
P(2)–O(4)–Dy(1)	163.2(4)	C(20)–C(19)–C(18)	121.4(9)
C(4)–C(5)–C(6)	118.9(9)	F(8)–C(20)–C(19)	118.1(9)
C(4)–C(5)–Br(1)	121.4(7)	F(8)–C(20)–C(15)	118.7(9)
C(6)–C(5)–Br(1)	119.7(7)	C(19)–C(20)–C(15)	123.2(9)
C(21)–O(5)–P(2)	123.0(6)	O(5)–C(21)–C(22)	106.9(8)
F(3)–C(6)–C(7)	118.8(9)	O(5)–C(21)–C(23)	108.5(8)
F(3)–C(6)–C(5)	121.1(9)	C(22)–C(21)–C(23)	113.8(8)
C(7)–C(6)–C(5)	120.1(9)	C(28)–C(27)–P(3)	112.7(6)
C(24)–O(6)–P(2)	122.7(6)	C(25)–C(24)–O(6)	107.8(8)
F(4)–C(7)–C(6)	118.1(9)	C(25)–C(24)–C(26)	115.2(9)
F(4)–C(7)–C(2)	119.9(8)	O(6)–C(24)–C(26)	106.5(8)
C(6)–C(7)–C(2)	121.9(9)	C(29)–C(28)–C(33)	117.2(8)
P(3)–O(7)–Dy(1)	172.9(4)	C(29)–C(28)–C(27)	122.4(8)
C(10)–C(8)–O(2)	110.8(8)	C(33)–C(28)–C(27)	120.3(8)
C(10)–C(8)–C(9)	112.8(9)	F(9)–C(29)–C(28)	120.2(8)
O(2)–C(8)–C(9)	105.2(8)	F(9)–C(29)–C(30)	117.1(8)
C(34)–O(8)–P(3)	125.2(6)	C(28)–C(29)–C(30)	122.7(9)
C(37)–O(9)–P(3)	126.6(5)	F(10)–C(30)–C(29)	120.0(8)
Dy(1)–O(10)–H(1O)	125(7)	F(10)–C(30)–C(31)	120.3(8)
Dy(1)–O(10)–H(2O)	114(6)	C(29)–C(30)–C(31)	119.8(9)
H(1O)–O(10)–H(2O)	116(9)	C(32)–C(31)–C(30)	118.4(9)
O(3)–C(11)–C(12)	107.9(8)	C(32)–C(31)–Br(3)	121.3(7)
O(3)–C(11)–C(13)	105.2(7)	C(30)–C(31)–Br(3)	120.3(7)
C(12)–C(11)–C(13)	114.1(8)	F(11)–C(32)–C(31)	120.8(9)
C(15)–C(14)–P(2)	112.2(6)	F(11)–C(32)–C(33)	118.7(9)
C(20)–C(15)–C(16)	114.1(9)	C(31)–C(32)–C(33)	120.4(9)
C(20)–C(15)–C(14)	123.8(8)	F(12)–C(33)–C(28)	121.1(8)
C(16)–C(15)–C(14)	122.0(8)	F(12)–C(33)–C(32)	117.4(8)

C(28)–C(33)–C(32)	121.5(9)	C(38)–C(37)–C(39)	113.7(8)
C(35)–C(34)–O(8)	109.8(8)	C(38)–C(37)–O(9)	106.9(7)
C(35)–C(34)–C(36)	113.6(11)	C(39)–C(37)–O(9)	106.9(7)
O(8)–C(34)–C(36)	106.3(8)		

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) i3070

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: i3070

Bond precision: C-C = 0.0149 A Wavelength=0.71073

Cell: a=13.4866(11) b=14.6216(17) c=16.3342(12)
alpha=110.663(7) beta=110.117(6) gamma=91.210(8)
Temperature: 100 K

	Calculated	Reported
Volume	2792.5(5)	2792.5(5)
Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	C39 H50 Br3 Cl2 Dy F12 O10 ? P3, Cl	
Sum formula	C39 H50 Br3 Cl3 Dy F12 O10 C39 H50 Br3 Cl3 Dy F12O10	
	P3	P3
Mr	1508.25	1508.28
Dx,g cm-3	1.794	1.794
Z	2	2
Mu (mm-1)	3.802	3.802
F000	1478.0	1478.0
F000'	1478.28	
h,k,lmax	16,17,19	16,17,19
Nref	10735	10468
Tmin,Tmax	0.701,0.927	0.596,0.912
Tmin'	0.481	

Correction method= # Reported T Limits: Tmin=0.596 Tmax=0.912 AbsCorr = MULTI-

SCAN

Data completeness= 0.975	Theta(max)= 25.798
R(reflections)= 0.0597(7266)	wR2(reflections)= 0.1435(10468)
S = 1.097	Npar= 660

The following ALERTS were generated. Each ALERT has the format

***test-name_ALERT_alert-type_alert-level**.*

Click on the hyperlinks for more details of the test.

Alert level C

PLAT213_ALERT_2_C Atom C36 has ADP max/min Ratio ..... 3.2 prolat
PLAT222_ALERT_3_C NonSolvent Resd 1 H Uiso(max)/Uiso(min) Range 7.9 Ratio
PLAT242_ALERT_2_C Low 'MainMol' Ueq as Compared to Neighbors of C34 Check
*PLAT245_ALERT_2_C U(iso) H2O Smaller than U(eq) O10 by 0.020 Ang**2*

PLAT334_ALERT_2_C Small Aver. Benzene C-C Dist C2 -C7 1.37 Ang.

PLAT342_ALERT_3_C Low Bond Precision on C-C Bonds ..... 0.01494 Ang.
PLAT434_ALERT_2_C Short Inter HL..HL Contact F3 ..F3 2.58 Ang.
2-x,2-y,2-z = 2_777 Check
PLAT906_ALERT_3_C Large K Value in the Analysis of Variance ..... 2.541 Check
PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L= 0.600 46 Report
PLAT918_ALERT_3_C Reflection(s) with I(obs) much Smaller I(calc) . 2 Check
PLAT934_ALERT_3_C Number of (Iobs-Icalc)/Sigma(W) > 10 Outliers .. 1 Check
PLAT972_ALERT_2_C Check Calcd Resid. Dens. 0.72A From Dy1 -1.68 eA-3
PLAT972_ALERT_2_C Check Calcd Resid. Dens. 1.10A From Dy1 -1.60 eA-3

Alert level G

PLAT083_ALERT_2_G SHELXL Second Parameter in WGHT Unusually Large 30.14 Why ?
PLAT175_ALERT_4_G The CIF-Embedded .res File Contains SAME Records 1 Report
PLAT432_ALERT_2_G Short Inter X...Y Contact F2 ..C17 2.93 Ang.
2-x,1-y,1-z = 2_766 Check
PLAT434_ALERT_2_G Short Inter HL..HL Contact Br3 ..F7 3.12 Ang.
-x,-y,-z = 2_555 Check
PLAT794_ALERT_5_G Tentative Bond Valency for Dy1 (III) . 3.20 Info
PLAT883_ALERT_1_G No Info/Value for _atom_sites_solution_primary . Please Do !
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L= 0.600 222 Note
PLAT941_ALERT_3_G Average HKL Measurement Multiplicity ..... 1.8 Low
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density. 0 Info

2.14 Complex 32

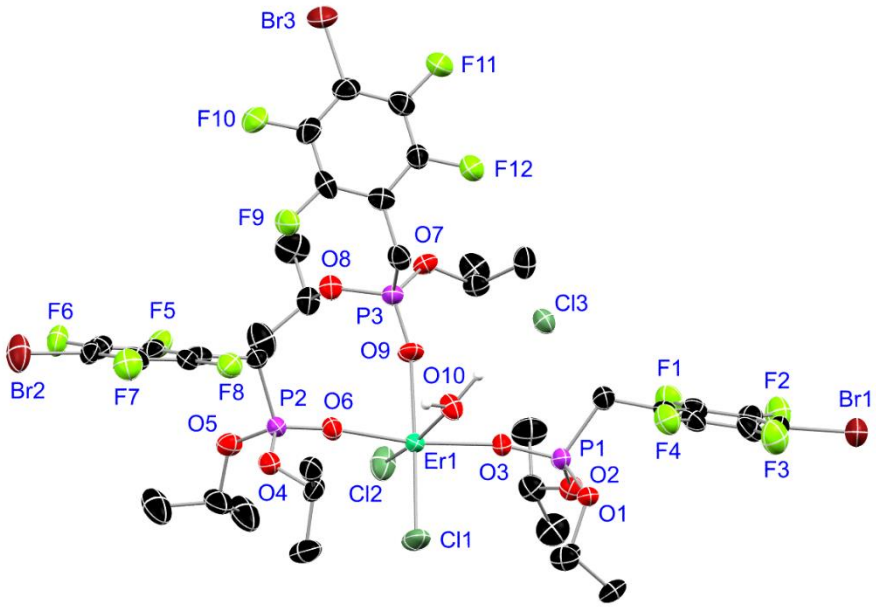

Figure 53: Asymmetric unit of **32**.

32 crystalizes as $C_{39}H_{50}Br_3Cl_2ErF_{12}O_{10}P_3 \cdot Cl^-$ in the space group $P\bar{1}$ and contains one molecule in the asymmetric unit. A H_2O ligand replaces one of the chloride anions causing the formation of a partially solvent separated ion pair. O5 and C24, C25 and C26 are refined with restraints for the anisotropic displacement parameters to stabilize the refinement. A high residual electron density located close to the heavy Erbium ion can be expected due to minor crystal quality, therefore causing a B-Alert upon cif-file check.

Bond lengths [Å] and angles [°] of **32**

C(1)–C(2)	1.350(13)	C(8)–C(10)	1.496(12)
C(1)–C(6)	1.371(13)	C(8)–O(1)	1.455(11)
C(1)–F(1)	1.326(10)	C(11)–C(12)	1.480(14)
C(2)–C(3)	1.362(14)	C(11)–C(13)	1.463(15)
C(2)–F(2)	1.316(11)	C(11)–O(2)	1.459(11)
C(3)–C(4)	1.364(15)	C(14)–C(15)	1.364(14)
C(3)–Br(1)	1.856(10)	C(14)–C(19)	1.373(13)
C(4)–C(5)	1.351(14)	C(14)–F(5)	1.331(10)
C(4)–F(3)	1.330(11)	C(15)–C(16)	1.348(13)
C(5)–C(6)	1.367(13)	C(15)–F(6)	1.331(11)
C(5)–F(4)	1.322(10)	C(16)–C(17)	1.365(13)
C(6)–C(7)	1.485(13)	C(16)–Br(2)	1.844(9)
C(7)–P(1)	1.782(10)	C(17)–C(18)	1.361(14)
C(8)–C(9)	1.491(12)	C(17)–F(7)	1.331(11)

C(18)–C(19)	1.361(13)	O(5)–P(2)	1.534(6)
C(18)–F(8)	1.345(10)	O(6)–P(2)	1.452(6)
C(19)–C(20)	1.483(13)	O(7)–P(3)	1.542(6)
C(20)–P(2)	1.757(8)	O(8)–P(3)	1.540(6)
C(21)–C(22)	1.490(14)	O(9)–P(3)	1.445(6)
C(21)–C(23)	1.462(13)		
C(21)–O(4)	1.458(10)	C(2)–C(1)–C(6)	121.4(8)
C(24)–C(25)	1.380(17)	F(1)–C(1)–C(2)	118.2(9)
C(24)–C(26)	1.484(17)	F(1)–C(1)–C(6)	120.4(9)
C(24)–O(5)	1.455(12)	C(1)–C(2)–C(3)	121.1(9)
C(27)–C(28)	1.376(14)	F(2)–C(2)–C(1)	119.6(9)
C(27)–C(32)	1.372(13)	F(2)–C(2)–C(3)	119.3(9)
C(27)–F(9)	1.318(11)	C(2)–C(3)–C(4)	118.1(9)
C(28)–C(29)	1.362(15)	C(2)–C(3)–Br(1)	121.9(8)
C(28)–F(10)	1.321(11)	C(4)–C(3)–Br(1)	119.8(8)
C(29)–C(30)	1.367(14)	C(5)–C(4)–C(3)	120.7(9)
C(29)–Br(3)	1.843(10)	F(3)–C(4)–C(3)	121.5(9)
C(30)–C(31)	1.342(14)	F(3)–C(4)–C(5)	117.8(9)
C(30)–F(11)	1.333(12)	C(4)–C(5)–C(6)	121.8(9)
C(31)–C(32)	1.353(13)	F(4)–C(5)–C(4)	118.7(9)
C(31)–F(12)	1.336(11)	F(4)–C(5)–C(6)	119.4(9)
C(32)–C(33)	1.471(13)	C(1)–C(6)–C(7)	121.7(8)
C(33)–P(3)	1.775(9)	C(5)–C(6)–C(1)	116.9(9)
C(34)–C(35)	1.474(14)	C(5)–C(6)–C(7)	121.4(9)
C(34)–C(36)	1.493(13)	C(6)–C(7)–P(1)	114.4(7)
C(34)–O(7)	1.443(11)	C(9)–C(8)–C(10)	113.1(8)
C(37)–C(38)	1.474(14)	O(1)–C(8)–C(9)	106.5(8)
C(37)–C(39)	1.475(15)	O(1)–C(8)–C(10)	106.0(7)
C(37)–O(8)	1.463(10)	C(13)–C(11)–C(12)	111.5(10)
Cl(1)–Er(1)	2.512(2)	O(2)–C(11)–C(12)	110.5(9)
Cl(2)–Er(1)	2.530(2)	O(2)–C(11)–C(13)	106.2(9)
Er(1)–O(3)	2.192(6)	C(15)–C(14)–C(19)	122.6(9)
Er(1)–O(6)	2.212(6)	F(5)–C(14)–C(15)	117.8(8)
Er(1)–O(9)	2.224(6)	F(5)–C(14)–C(19)	119.6(8)
Er(1)–O(10)	2.286(6)	C(16)–C(15)–C(14)	120.7(9)
O(1)–P(1)	1.533(6)	F(6)–C(15)–C(14)	118.7(9)
O(2)–P(1)	1.537(7)	F(6)–C(15)–C(16)	120.6(9)
O(3)–P(1)	1.473(6)	C(15)–C(16)–C(17)	118.3(9)
O(4)–P(2)	1.535(6)	C(15)–C(16)–Br(2)	121.7(7)

C(17)–C(16)–Br(2)	119.9(7)	C(38)–C(37)–C(39)	114.6(10)
C(18)–C(17)–C(16)	119.9(9)	O(8)–C(37)–C(38)	107.3(8)
F(7)–C(17)–C(16)	121.3(9)	O(8)–C(37)–C(39)	106.9(8)
F(7)–C(17)–C(18)	118.7(9)	Cl(1)–Er(1)–Cl(2)	100.36(10)
C(17)–C(18)–C(19)	123.5(9)	O(3)–Er(1)–Cl(1)	95.13(17)
F(8)–C(18)–C(17)	116.9(8)	O(3)–Er(1)–Cl(2)	89.57(17)
F(8)–C(18)–C(19)	119.7(8)	O(3)–Er(1)–O(6)	169.6(2)
C(14)–C(19)–C(20)	122.8(8)	O(3)–Er(1)–O(9)	89.4(2)
C(18)–C(19)–C(14)	114.9(9)	O(3)–Er(1)–O(10)	84.6(2)
C(18)–C(19)–C(20)	122.3(8)	O(6)–Er(1)–Cl(1)	89.57(17)
C(19)–C(20)–P(2)	112.0(6)	O(6)–Er(1)–Cl(2)	98.68(16)
C(23)–C(21)–C(22)	112.4(8)	O(6)–Er(1)–O(9)	84.7(2)
O(4)–C(21)–C(22)	107.5(8)	O(6)–Er(1)–O(10)	86.1(2)
O(4)–C(21)–C(23)	108.0(7)	O(9)–Er(1)–Cl(1)	170.56(18)
C(25)–C(24)–C(26)	114.9(12)	O(9)–Er(1)–Cl(2)	87.95(18)
C(25)–C(24)–O(5)	111.6(10)	O(9)–Er(1)–O(10)	81.0(2)
O(5)–C(24)–C(26)	105.2(9)	O(10)–Er(1)–Cl(1)	91.19(18)
C(32)–C(27)–C(28)	120.8(9)	O(10)–Er(1)–Cl(2)	167.48(17)
F(9)–C(27)–C(28)	117.9(9)	C(8)–O(1)–P(1)	122.9(6)
F(9)–C(27)–C(32)	121.3(9)	C(11)–O(2)–P(1)	124.5(6)
C(29)–C(28)–C(27)	121.7(9)	P(1)–O(3)–Er(1)	164.8(4)
F(10)–C(28)–C(27)	118.1(9)	C(21)–O(4)–P(2)	126.5(6)
F(10)–C(28)–C(29)	120.2(9)	C(24)–O(5)–P(2)	125.3(7)
C(28)–C(29)–C(30)	116.8(9)	P(2)–O(6)–Er(1)	172.9(4)
C(28)–C(29)–Br(3)	120.7(8)	C(34)–O(7)–P(3)	123.6(5)
C(30)–C(29)–Br(3)	122.5(8)	C(37)–O(8)–P(3)	122.4(6)
C(31)–C(30)–C(29)	121.0(10)	P(3)–O(9)–Er(1)	162.9(4)
F(11)–C(30)–C(29)	119.3(9)	O(1)–P(1)–C(7)	101.5(4)
F(11)–C(30)–C(31)	119.7(9)	O(1)–P(1)–O(2)	105.6(3)
C(30)–C(31)–C(32)	123.6(9)	O(2)–P(1)–C(7)	109.0(4)
F(12)–C(31)–C(30)	117.6(9)	O(3)–P(1)–C(7)	110.8(4)
F(12)–C(31)–C(32)	118.8(9)	O(3)–P(1)–O(1)	115.8(4)
C(27)–C(32)–C(33)	120.8(9)	O(3)–P(1)–O(2)	113.4(4)
C(31)–C(32)–C(27)	116.2(9)	O(4)–P(2)–C(20)	108.1(4)
C(31)–C(32)–C(33)	123.0(9)	O(5)–P(2)–C(20)	102.4(4)
C(32)–C(33)–P(3)	111.7(6)	O(5)–P(2)–O(4)	105.5(4)
C(35)–C(34)–C(36)	114.4(9)	O(6)–P(2)–C(20)	111.8(4)
O(7)–C(34)–C(35)	109.0(8)	O(6)–P(2)–O(4)	113.4(3)
O(7)–C(34)–C(36)	106.0(8)	O(6)–P(2)–O(5)	114.8(4)

O(7)–P(3)–C(33)	108.9(4)	O(9)–P(3)–C(33)	112.9(4)
O(8)–P(3)–C(33)	102.9(4)	O(9)–P(3)–O(7)	111.1(4)
O(8)–P(3)–O(7)	105.7(3)	O(9)–P(3)–O(8)	114.7(4)

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) i3071

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No syntax errors found. CIF dictionary Interpreting this report

Datablock: i3071

Bond precision: *C-C = 0.0158 A* *Wavelength=0.71073*

Cell: *a=13.3443(19)* *b=14.464(2)* *c=16.184(2)*
alpha=111.069(10) *beta=110.094(11)* *gamma=91.110(12)*

Temperature: 100K

	Calculated	*Reported*
Volume	*2700.8(7)*	*2700.8(7)*
Space group	*P -1*	*P -1*
Hall group	*-P 1*	*-P 1*
Moiety formula	*C39 H50 Br3 Cl2 Er F12 O10*	*C39 H50 Br3 Cl2 Er F12O10*
	P3, Cl	*P3, Cl*
Sum formula	*C39 H50 Br3 Cl3 Er F12 O10*	*C39 H50 Br3 Cl3 Er F12O10*
	P3	*P3*
Mr	1513.01	1513.04
Dx,g cm-3	1.860	1.861
Z	2	2
Mu (mm-1)	4.102	4.102
F000	1482.0	1482.0
F000’	1482.16	
h,k,lmax	16,17,19	16,17,19

Nref	10420	10143
Tmin,Tmax	0.681,0.782	0.336,0.801
Tmin'	0.631	

Correction method= # Reported T Limits: Tmin=0.336 Tmax=0.801 AbsCorr = MULTI-SCAN

Data completeness= 0.973 *Theta(max)= 25.829*

R(reflections)= 0.0594(7205) *wR2(reflections)= 0.1681(10143)*

S = 1.121 *Npar= 653*

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.

Click on the hyperlinks for more details of the test.

Alert level B

PLAT973_ALERT_2_B Check Calcd Positive Resid. Density on Er1 1.80 eA-3

Alert level C

PLAT334_ALERT_2_C Small Aver. Benzene C-C Dist C1 -C6 1.36 Ang.
PLAT334_ALERT_2_C Small Aver. Benzene C-C Dist C14 -C19 1.36 Ang.
PLAT334_ALERT_2_C Small Aver. Benzene C-C Dist C27 -C32 1.36 Ang.
PLAT342_ALERT_3_C Low Bond Precision on C-C Bonds ..... 0.01582 Ang.
PLAT360_ALERT_2_C Short C(sp3)-C(sp3) Bond C24 - C25 . 1.38 Ang.
PLAT434_ALERT_2_C Short Inter HL..HL Contact F3 ..F3 2.55 Ang.
-x,-y,-z = 2_555 Check
PLAT906_ALERT_3_C Large K Value in the Analysis of Variance ..... 2.036 Check
PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L= 0.600 53 Report

PLAT971_ALERT_2_C	Check	Calcd	Resid.	Dens.	1.04A	From	Er1	2.24 eA-3
PLAT971_ALERT_2_C	Check	Calcd	Resid.	Dens.	1.00A	From	Er1	2.17 eA-3
PLAT971_ALERT_2_C	Check	Calcd	Resid.	Dens.	1.02A	From	Er1	2.13 eA-3
PLAT971_ALERT_2_C	Check	Calcd	Resid.	Dens.	1.12A	From	Er1	2.10 eA-3
PLAT971_ALERT_2_C	Check	Calcd	Resid.	Dens.	0.93A	From	Er1	1.92 eA-3

PLAT977_ALERT_2_C Check Negative Difference Density on H12A -0.31 eA-3
PLAT977_ALERT_2_C Check Negative Difference Density on H25C -0.42 eA-3
PLAT977_ALERT_2_C Check Negative Difference Density on H26A -0.32 eA-3
PLAT977_ALERT_2_C Check Negative Difference Density on H36A -0.33 eA-3

Alert level G

PLAT003_ALERT_2_G Number of Uiso or Uij Restrained non-H Atoms ... 4 Report
PLAT007_ALERT_5_G Number of Unrefined Donor-H Atoms ..... 2 Report
PLAT083_ALERT_2_G SHELXL Second Parameter in WGHT Unusually Large 13.76 Why ?
PLAT187_ALERT_4_G The CIF-Embedded .res File Contains RIGU Records 1 Report
PLAT432_ALERT_2_G Short Inter X...Y Contact F2 ..C30 2.91 Ang.
-x,1-y,1-z = 2_566 Check
PLAT434_ALERT_2_G Short Inter HL..HL Contact Br2 ..F10 3.07 Ang.

	2-x,2-y,2-z	=	2_777 Check
PLAT434_ALERT_2_G Short Inter HL..HL Contact Br3	..Cl2		3.48 Ang.
	x,1+y,z	=	1_565 Check
PLAT794_ALERT_5_G Tentative Bond Valency for Er1 (III)		.	3.50 Info
PLAT860_ALERT_3_G Number of Least-Squares Restraints			18 Note
PLAT883_ALERT_1_G No Info/Value for _atom_sites_solution_primary .			Please Do !
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L= 0.600			222 Note
PLAT933_ALERT_2_G Number of OMIT Records in Embedded .res File ...			11 Note
PLAT941_ALERT_3_G Average HKL Measurement Multiplicity			1.8 Low
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density.			0 Info

3 PHOTOLUMINESCENCE

3.1 Absorption Spectra

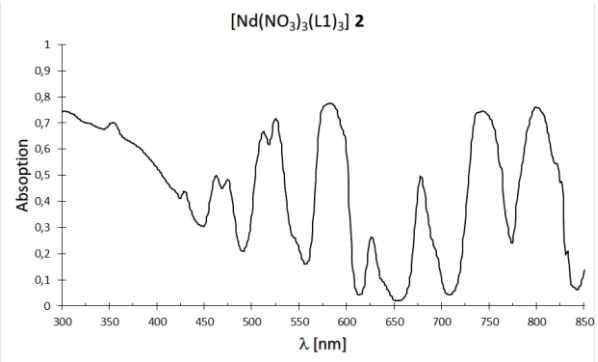

Figure 54: Absorption spectrum of complex **2**.

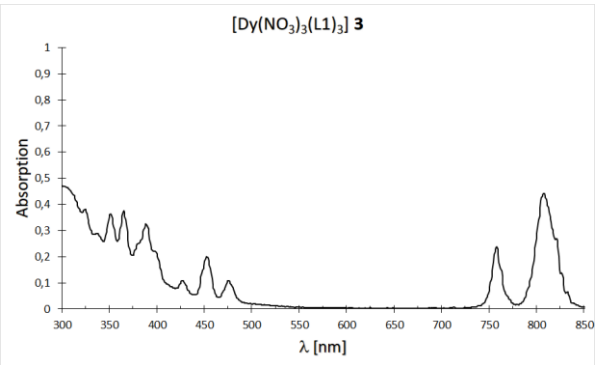

Figure 55: Absorption spectrum of complex **3**.

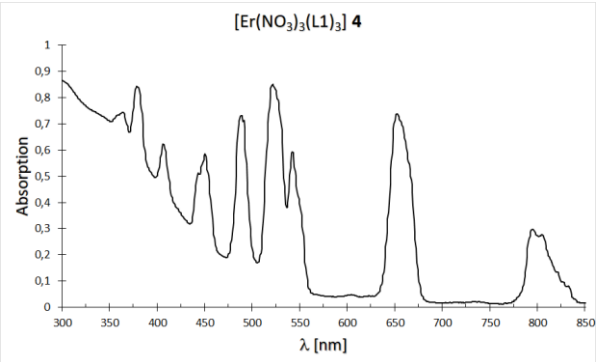

Figure 56: Absorption spectrum of complex **4**.

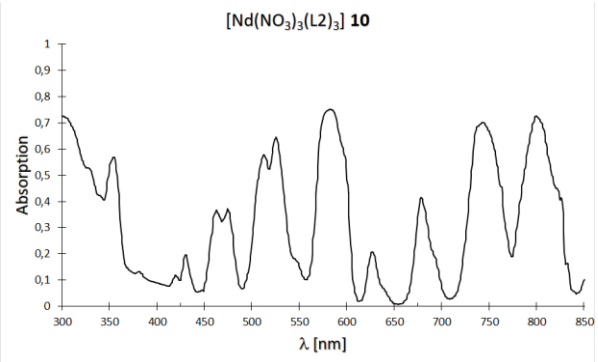

Figure 57: Absorption spectrum of complex **10**.

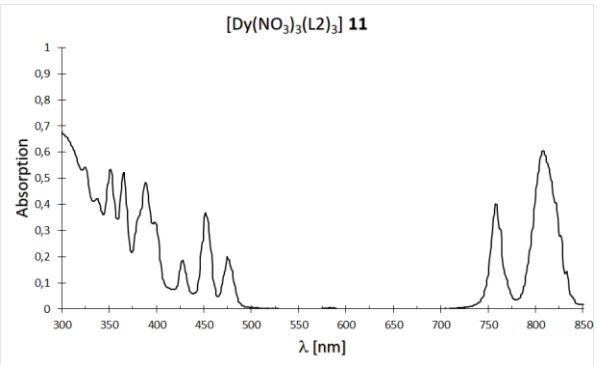

Figure 58: Absorption spectrum of complex **11**.

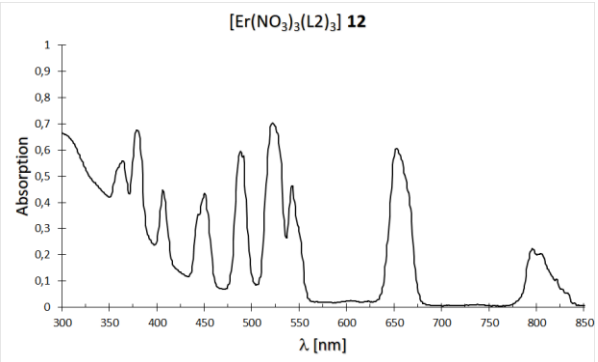

Figure 59: Absorption spectrum of complex **12**.

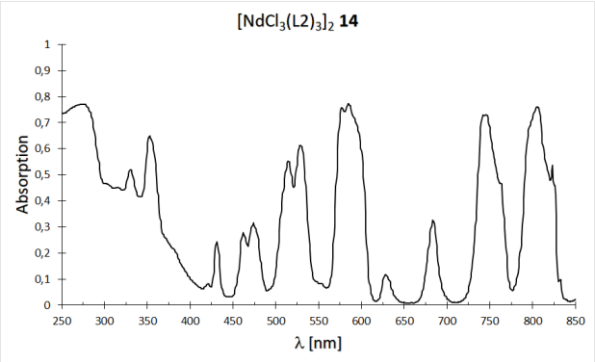

Figure 60: Absorption spectrum of complex **14**.

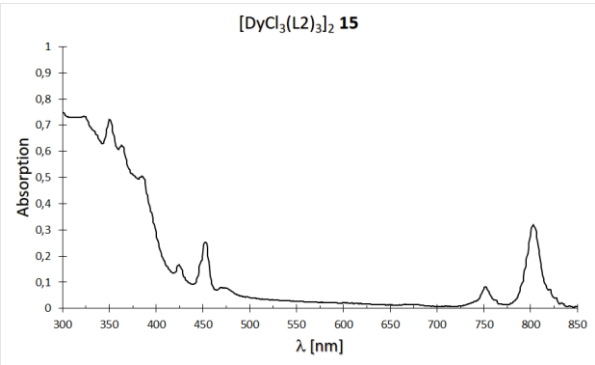

Figure 61: Absorption spectrum of complex **15**.

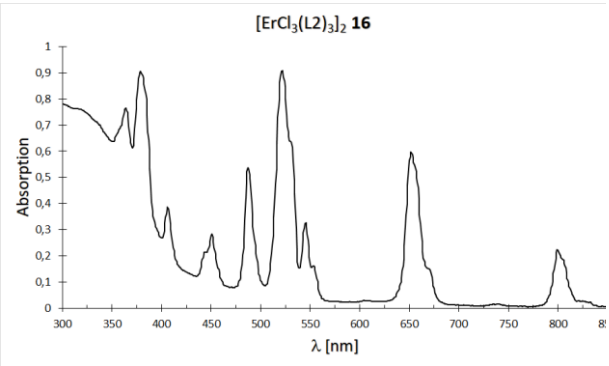

Figure 62: Absorption spectrum of complex **16**.

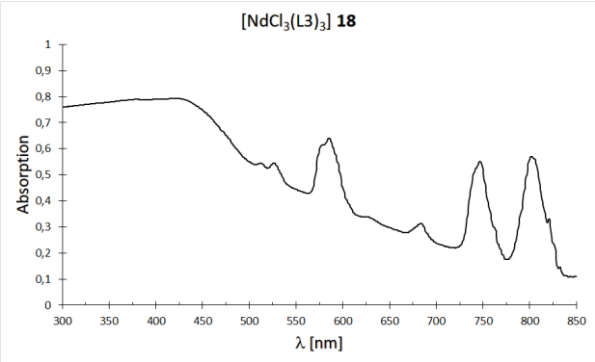

Figure 63: Absorption spectrum of complex **18**.

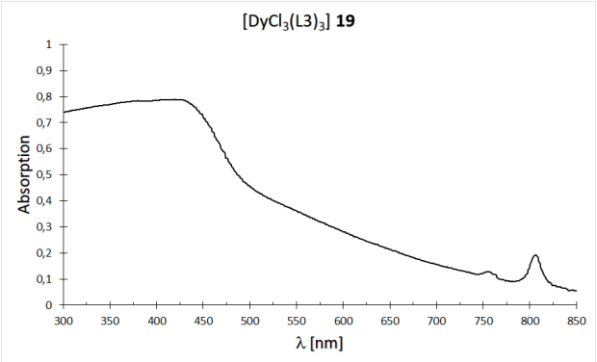

Figure 64: Absorption spectrum of complex **19**.

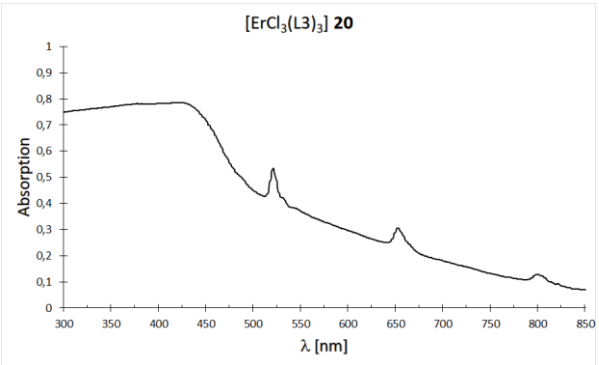

Figure 65: Absorption spectrum of complex **20**.

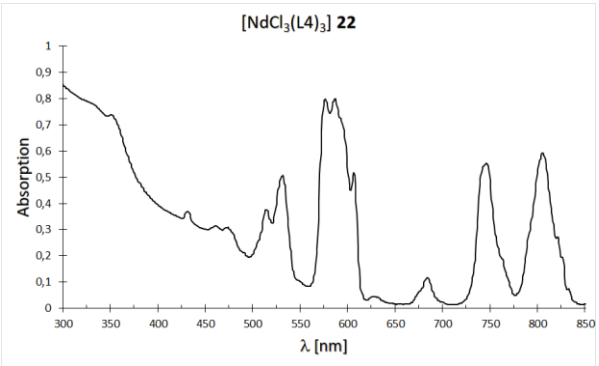

Figure 66: Absorption spectrum of complex **22**.

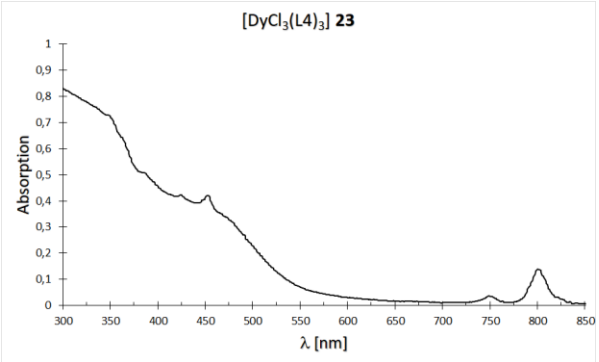

Figure 67: Absorption spectrum of complex **23**.

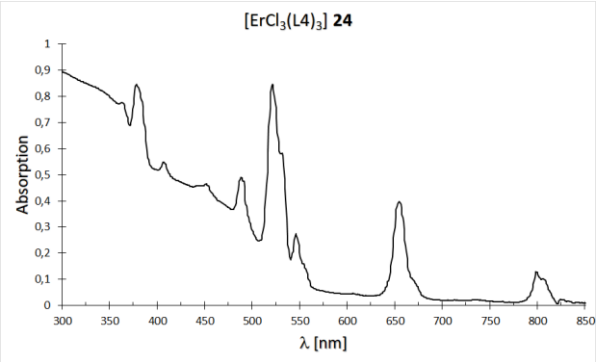

Figure 68: Absorption spectrum of complex **24**.

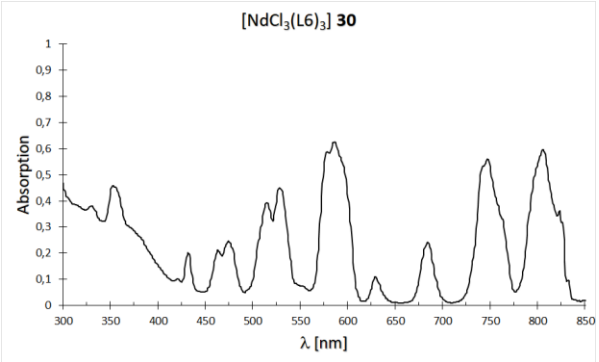

Figure 72: Absorption spectrum of complex **30**.

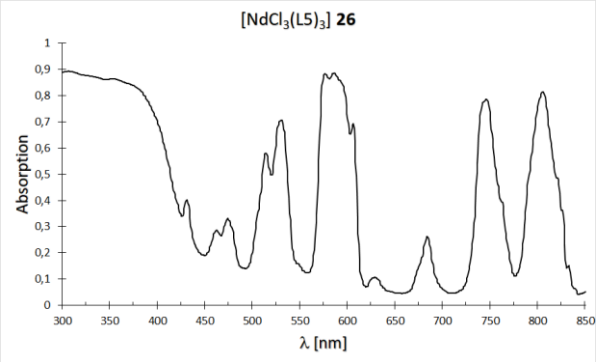

Figure 69: Absorption spectrum of complex **26**.

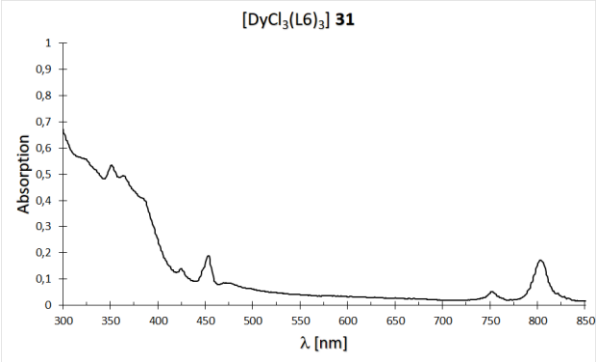

Figure 73: Absorption spectrum of complex **31**.

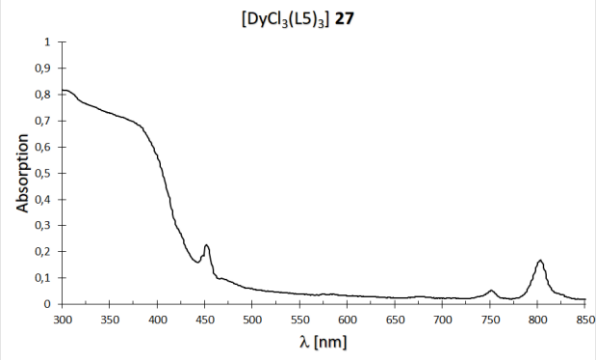

Figure 70: Absorption spectrum of complex **27**.

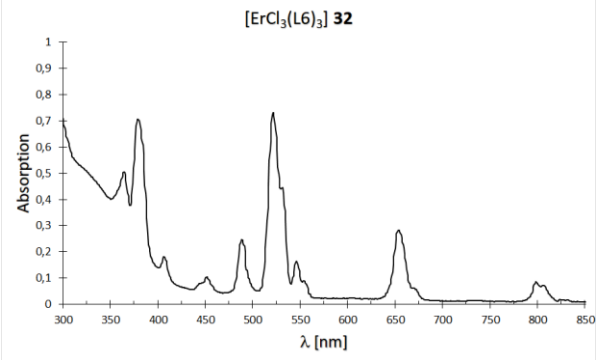

Figure 74: Absorption spectrum of complex **32**.

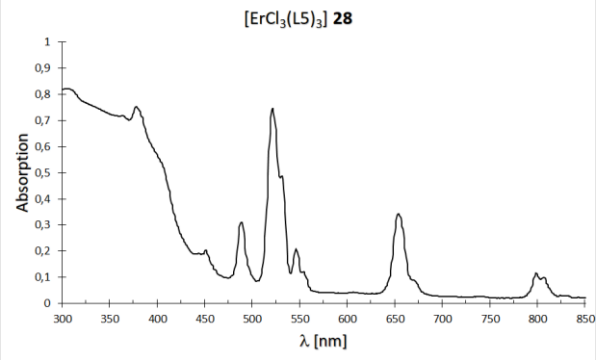

Figure 71: Absorption spectrum of complex **28**.

3.2 Emission Spectra

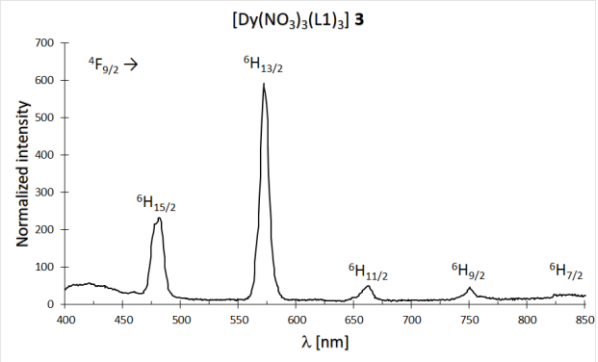

Figure 75: Emission spec. of complex **3** ($\lambda_{exc.}$ = 366 nm).

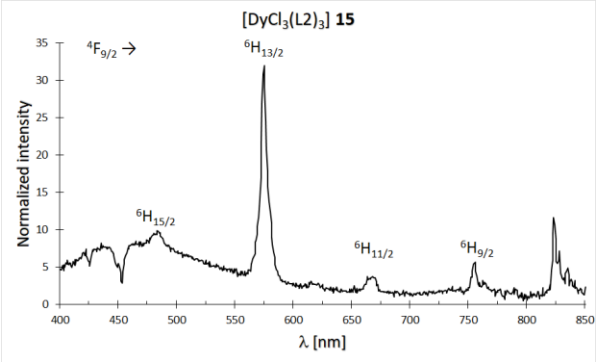

Figure 78: Emission spec. of complex **15** ($\lambda_{exc.}$ = 366 nm).

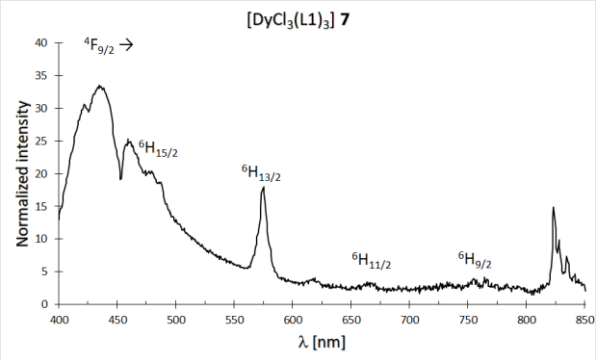

Figure 76: Emission spec. of complex **7** ($\lambda_{exc.}$ = 366 nm).

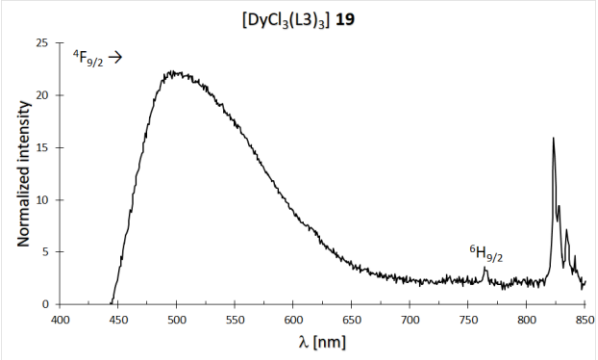

Figure 79: Emission spec. of complex **19** ($\lambda_{exc.}$ = 366 nm).

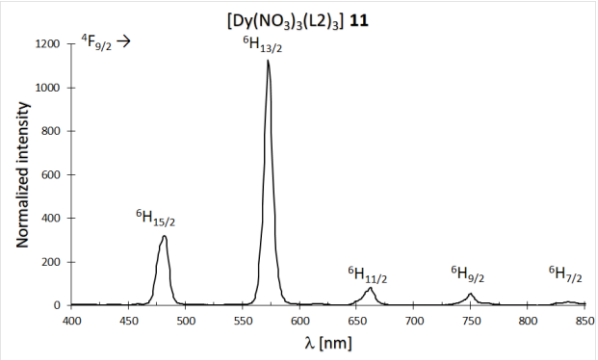

Figure 77: Emission spec. of complex **11** ($\lambda_{exc.}$ = 366 nm).

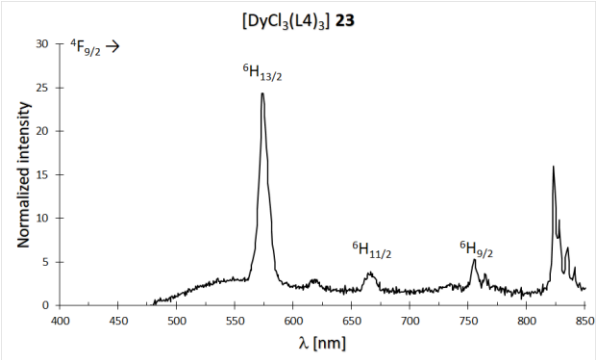

Figure 80: Emission spec. of complex **23** ($\lambda_{exc.}$ = 366 nm).

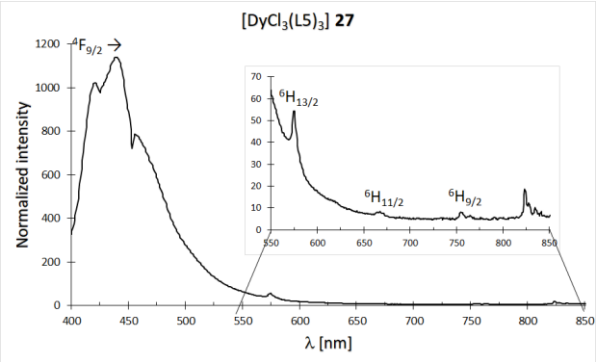

Figure 81: Emission spec. of complex **27** ($\lambda_{exc.}$ = 366 nm).

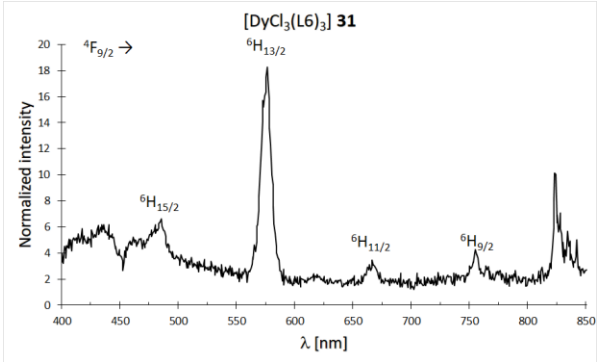

Figure 82: Emission spec. of complex **31** ($\lambda_{exc.}$ = 366 nm).

4 REFERENCES

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