Electronic Supplementary Material (ESI) for Dalton Transactions

Supplementary Information

A planar dianionic ditelluride and a cyclic tritelluride supported by P_2N_2 rings†

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Experimental Section

General Procedures and Starting Materials

All experiments were carried out in standard Schlenk glassware or in a glove box with strict exclusion of air and moisture, under a nitrogen or argon atmosphere. Solvents were dried on an MBraun solvent purification system and stored over molecular sieves prior to use. ^{n}BuNa,[1] *cis*-(^{t}BuNH)P(μ-N^{t}Bu)$_2$P(NH^{t}Bu)[2] and TeCl$_2$·tmtu[3] were prepared by the literature procedures. Tmtu refers to tetramethylthiourea (98 %), TMEDA to tetramethylethylenediamine.

All new compounds were fully characterised by ^{31}P{^{1}H} and, in most cases, by ^{1}H and ^{125}Te spectra. Measurements were performed at 25 °C; 85 % H_3PO_4 was used as external standard in ^{31}P, TMS as internal for ^{1}H, and Ph_2Te_2 for ^{125}Te. Raman spectra were collected on a PE System2000 spectrometer equipped with a near-IR excitation laser. In vacuo refers to a pressure of ca. 10 Pa.

Experimental Procedures

‡ [Na(tmeda)]$_2$**1c**: Solid ^{n}BuNa (0.580 g, 7.24 mmol) was added slowly to a solution of *cis*-(^{t}BuNH)P(μ-N^{t}Bu)$_2$P(NH^{t}Bu) (1.000 g, 2.87 mmol) in toluene (15 mL) at 0 °C. The reaction mixture was stirred for 1 h at 23 °C and then for 30 min at 40 °C. Toluene was removed under vacuum and the solid residue was dissolved in *n*-hexane (20 mL) and filtered to remove unreacted ^{n}BuNa. The solvent was removed in vacuo to give [Na$_4$(P$_2$(μ-N^{t}Bu)$_2$(N^{t}Bu)$_2$)$_2$] as a colorless solid (89 % yield).[17] ^{1}H NMR (d_8-toluene, 25 °C): δ = 1.36 (s, 18H, ^{t}Bu), 1.27 (s, 18 H, ^{t}Bu); ^{31}P NMR (d_8-toluene, 25 °C): δ = 136.5 (s). To a suspension of [Na$_4$(P$_2$(μ-N^{t}Bu)$_2$(N^{t}Bu)$_2$)$_2$] (0.500 g, 0.64 mmol) in toluene

(20 mL) was added tellurium powder (0.327 g, 2.56 mmol) and TMEDA (0.305 g, 2.62 mmol). The reaction mixture was stirred for 3 h at 80 °C and then allowed to cool to RT. The dark red mixture was filtered and the solvent was removed under reduced pressure. The residue was then washed with hexane to give [Na(tmeda)]$_2$**1c** as a pale yellow solid. (67 %)

NMR data for [Na(tmeda)]$_2$**1c:** ^{1}H NMR (d_8-toluene, 25 °C): δ = 2.04 (s, 24H, NMe$_2$), 2.01 (s, 18H, ^{t}Bu), 1.85 (s, 8H, CH$_2$), 1.69 (s, 18H, ^{t}Bu); ^{31}P NMR (d_8-toluene, 25 °C): δ = −74.9 (s, ^1J(P,Te) = 1590 Hz, ^2J(P,P) = 30 Hz); ^{125}Te NMR (d_8-toluene, 25 °C): δ = −148.7(d, ^1J(P,Te) = 1583 Hz).

[Li(tmeda)]$_2$**2:** [Li(tmeda)]$_2$[^{t}BuN(Te)P(μ-N^{t}Bu)$_2$P(Te)N^{t}Bu)] (0.500 g, 0.59 mmol) was suspended in toluene and cooled to −78 °C. A solution of I$_2$ (0.075 g, 0.29 mmol) in toluene (15 mL) was maintained at −78 °C and added dropwise over 15 min by cannula. The reaction mixture was stirred for 30 min before being allowed to warm to RT and stirred for an additional 1 h. The mixture was then filtered to remove the released elemental Te and the solvent was removed under reduced pressure. The solid residue was dissolved in hexane, filtered and the solvent was removed to afford a dark reddish black material. Recrystallisation from hexane afforded black crystals that were filtered dried and identified as [Li(tmeda)]$_2$**2** by single crystal XRD (estimated yield 33 % by NMR). NMR data for [Li(tmeda)]$_2$**2:** ^{31}P NMR (d_8-toluene, 25 °C): δ = −78.5 (s, $^1J_{PTe}$ = 1670 Hz), −117.0 (s, $^1J_{PTe}$ = 1219 Hz). The compound is very air-sensitive and thermally unstable; it decomposed to give (^{t}BuNH)P(μ-N^{t}Bu)$_2$P(N^{t}Bu)TeTe(^{t}BuN)P(μ-N^{t}Bu)$_2$P(HN^{t}Bu) (**4**) as upon numerous attempts to isolate it.

NMR data for **4**: ^{31}P NMR (d_8-toluene, 25 °C): δ = 75.8 (s), −65.5 (s, $^{1}J_{PTe}$ = 1549 Hz); ^{125}Te NMR (d_8-toluene, 25 °C): δ = −207.0 (d, $^{1}J_{PTe}$ = 1536 Hz) MS (CI$^+$, *m/z*), 951 [M+H]$^+$. HRMS (CI$^+$, *m/z*) 951.3136 [M+H]$^+$ (calculated: 951.3155 [M+H]$^+$).

3: **Method A**: A solution of iodine (0.145 g, 0.57 mmol) in toluene (15 mL) cooled to −78 °C was added dropwise (15 min) by cannula to a suspension of [Na(tmeda)]$_2$**1c** (0.500 g, 0.57 mmol) in toluene (20 mL) cooled to −78 °C. The reaction mixture was stirred for 30 min at −78 °C and then at RT for 1 h. After filtration the solvent was removed under reduced pressure, the residue was dissolved in hexane, then filtered and the solvent was removed to give a dark red-black solid. Recrystallisation from pentane afforded black crystals of **3** (0.058 g, 14 % yield).

Method B: A solution of $TeCl_2$·tmtu (0.189 g, 0.57 mmol) in toluene (15 mL) cooled to −78 °C was added dropwise (15 min) by cannula to a suspension of [Na(tmeda)]$_2$**1c** (0.500 g, 0.57 mmol) in toluene (20 mL) cooled to −78 °C. The reaction mixture was stirred for 3 h at −78 °C and then for 2 h at RT. The precipitate (NaCl) was removed by filtration and the solvent was removed from the filtrate under reduced pressure to give black crystals of **3** (0.195 g, 41 % yield). Mp, T_{dec}: 156 °C. NMR data for **3**: ^{1}H NMR (d_8-toluene, 25 °C): 1.65 (s, 18H, ^{t}Bu), 1.39 (s, 18H, ^{t}Bu, ^{3}J(P,H) = 1.0 Hz); ^{31}P NMR (d_8-toluene, 25 °C): δ = −134.5 (s, ^{1}J(P,Te) = 1029 Hz, ^{2}J(P,P) = 31 Hz); ^{125}Te NMR (d_8-toluene, 25 °C): δ = −442.8 (dd, ^{1}J(P,Te) = 1031 Hz, ^{3}J(P,Te) = 41 Hz), 361.9 (t, ^{2}J(P,Te) = 35 Hz). MS (EI$^+$, *m/z*), 730 [M+H]$^+$. CHN Anal.: calcd (%) for $C_{16}H_{36}N_4P_2Te_3$: C 26.35, H 4.98, N 7.68, found: C 26.34, H 4.93, N 7.63.

X-ray Crystallography

Data for $[Na(tmeda)]_2$**1c**, **3** and $[Na_4(P_2(\mu\text{-}N^tBu)_2(N^tBu)_2)_2]$ were collected with a Nonius Kappa CCD diffractometer with use of monochromated Mo-Kα radiation (λ = 0.71073 Å) at 173 K. The structures were solved by direct methods using the program SHELXS-97[4] and refined with SHELXL-97[4] and by full-matrix least-squares with anisotropic thermal parameters for the non-hydrogen atoms. Hydrogen atoms were included in calculated positions and were refined by riding model. In $[Na(tmeda)]_2$**1c**, one of the *tert*-butyl group having carbon atom (C11) was disordered over two positions which were fixed. Data for $[Li(tmeda)]_2$**2** and **4** were collected at 93 K by using a Rigaku MM007 High brilliance RA generator/confocal optics and Mercury CCD system with graphite monochromated Mo-Kα radiation (λ = 0.71073 Å). Intensity data were collected using ω steps accumulating area detector images spanning at least a hemisphere of reciprocal space. All data were corrected for Lorentz polarization effects. The data for the complexes analysed was collected and processed by using CrystalClear (Rigaku).[5] Absorption effects were corrected on the basis of multiple equivalent reflections or by semi-empirical methods. Structures were solved by direct methods or heavy-atom Patterson methods[6] and refined by full-matrix least-squares against F2 by using the program SHELXL using Fourier Techniques.[4] Hydrogen atoms were refined using the ridging method and constrained to idealised geometries. All calculations were performed using the CrystalStructure[7] crystallographic software package except for refinement, which was performed using SHELXL-97.[4]

Crystallographic data for $[Na_4(P_2(\mu\text{-}N^tBu)_2(N^tBu)_2)_2]$ and **4** are given in Table 1. Data were collected with a Nonius Kappa CCD diffractometer with use of monochromated Mo-Kα radiation (λ = 0.71073 Å) at –150 ºC. The structures were solved by direct methods using the program SHELXS-97[4] and refined with SHELXL-97[4] and by full-matrix least-squares with anisotropic thermal parameters for the non-hydrogen atoms. Hydrogen atoms were included in calculated positions and were refined by riding model. Structure of $[Na_4(P_2(\mu\text{-}N^tBu)_2(N^tBu)_2)_2]$ having carbon atoms (C16, C27, C28, C43, C55, C59, C62, C63, C64) were disordered over two positions which were fixed. Two disordered solvent hexane molecules in asymmetric unit were removed from the diffraction data with SQUEEZE. *SQUEEZE* estimated the electron count in the void is 445. This value is closely related to the eight hexane molecules (400) in cell.

Computational Methods

Dispersion-corrected (Grimme's D3)[8] relativistic (ZORA)[9] GGA (PBE)[10] DFT calculations were performed using the ADF[11] package. Molecular geometries were fully optimized, but vibrational calculations were not performed given the computational expense of the numerical method.

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Table S1. Crystallographic data for $[Na_4(P_2(\mu\text{-}N^tBu)_2(N^tBu)_2)_2]$ and $(^tBuNH)P(\mu\text{-}N^tBu)_2P(N^tBu)TeTe(^tBuN)P(\mu\text{-}N^tBu)_2P(HN^tBu)$ (**4**)

	$[Na_4(P_2(\mu\text{-}N^tBu)_2(N^tBu)_2)_2]$	**4**
Empirical formula	C76 H172 N16 P8 Na8	C32 H74 N8 P4 Te2
Formula weight	1741.98	950.09
Temperature (K)	173(2)	93
Wavelength (Å)	0.71073	0.71075
Crystal system	monoclinic	monoclinic
Space group	*P* $2_1/n$	*C* 2/*c*
a (Å), alpha (deg)	21.710(4), 90	13.232(4), 90
b (Å), beta (deg)	21.271(4), 106.51(3)	14.228(4), 104.587(12)
c (Å), gamma (deg)	22.414(5), 90	24.627(8), 90
Volume ($Å^3$)	9924(3)	4487(3)
Z	4	4
Calculated density (mg/m^3)	1.166	1.406
Absorption coefficient (mm^{-1})	0.222	1.474
F(000)	3792	1944
Crystal size (mm^3)	0.27 x 0.16 x 0.08	0.12 x 0.09 x 0.06
Theta range (deg.)	1.4 to 27.50	2.46 to 26.59
Limiting indices	-21<=h<=21, -21<=k<=21, -22<=l<=22	-12<=h<=15, -16<=k<=16, -29<=l<=22
Reflections collected/unique	19356 / 10561 [R(int) = 0.0771]	3863 / 2869 [R(int) = 0.1179]
Completeness to theta	99.0 %	97.7 %
Max. and min. transmission	0.9825 and 0.9425	0.957 and 0.187
Refinement method	Full-matrix least-squares on F2	Full-matrix least-squares on F2
Data / restraints / parameters	10561 / 42 / 895	3863 / 0 / 208
Goodness-of-fit on F^2	1.036	1.066
R1, wR2 [I>2sigma(I)]	0.0991, 0.2652	0.0554, 0.1179
R1, wR2 (all data)	0.1330, 0.2919	-
Largest diff. peak and hole ($e.Å^{-3}$)	0.578 and -0.472	0.980 and -0.570

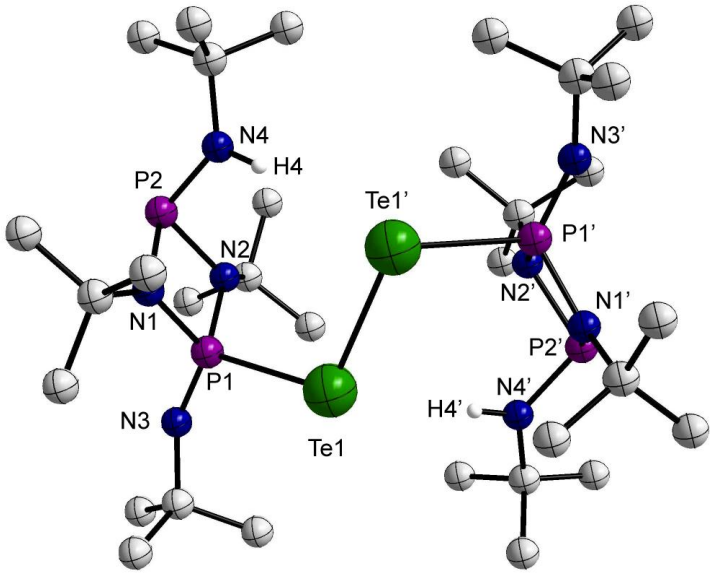

Figure S1

Molecular structure of (^{t}BuNH)P(μ-N^{t}Bu)$_2$P(N^{t}Bu)TeTe(^{t}BuN)P(μ-N^{t}Bu)$_2$P(HN^{t}Bu) (**4**). Bond lengths (Å): Te1–Te1' 2.7204(9), Te1–P1 2.552(2), P1–N1 1.666(6), P1–N2 1.670(6), P1–N3 1.493(5), P2–N1 1.740(6), P2–N2 1.746(6), P2–N4 1.658(5), N1–C1 1.492(9), N2–C5 1.480(9), N3–C9 1.466(9), N4–C13 1.479(9), C1–C2 1.519(9), C1–C3 1.531(9), C1–C4 1.520(11), C5–C6 1.525(9), C5–C7 1.529(11), C5–C8 1.519(9), C9–C10 1.517(11), C9–C11 1.514(12), C9–C12 1.516(12), C13–C14 1.529(11), C13–C15 1.514(12), C13–C16 1.515(11), N4–H4 0.8800, N3–C2 3.45(1), N3–C5 3.402(8), N3–C6 3.461(10), N3–C7 3.537(9), N4–C1 3.546(8), N4–C8 3.549(9); Bond angles (°): Te1'–Te1–P1 106.45(5), Te1–P1–P2 121.38(7), Te1–P1–N1 110.15(19), Te1–P1–N2 111.58(19), Te1–P1–N3 108.5(3), N1–P1–N2 84.4(3), N1–P1–N3 121.4(3), N2–P1–N3 119.0(3), N1–P2–N2 80.0(3), N1–P2–N4 106.0(3), N2–P2–N4 104.5(3), P1–N1–P2 97.9(3), P1–N1–C1 133.4(5), P2–N1–C1 128.7(5), P1–N2–P2 97.5(3), P1–N2–C5 129.2(4), P2–N2–C5 126.0(5), P1–N3–C9 151.1(6), P2–N4–C13 128.7(5), N1–C1–C2 109.9(5), N1–C1–C3 110.1(5), N1–C1–C4 107.1(6), C2–C1–C3 109.1(6), C2–C1–C4

111.3(6), C3–C1–C4 109.3(6), N2–C5–C6 111.4(6), N2–C5–C7 106.7(6), N2–C5–C8 108.6(5), C6–C5–C7 110.0(6), C6–C5–C8 109.3(6), C7–C5–C8 110.8(6), N3–C9–C10 106.6(6), N3–C9–C11 111.7(6), N3–C9–C12 110.7(6), C10–C9–C11 108.0(7), C10–C9–C12 110.9(6), C11–C9–C12 109.0(7), N4–C13–C14 107.8(6), N4–C13–C15 110.5(5), N4–C13–C16 108.1(6), C14–C13–C15 111.1(7), C14–C13–C16 109.3(6), C15–C13–C16 110.0(7), P2–N4–H4 115.641; Torsion angle (°): P1–Te1–Te1'–P1' 123.84(6).

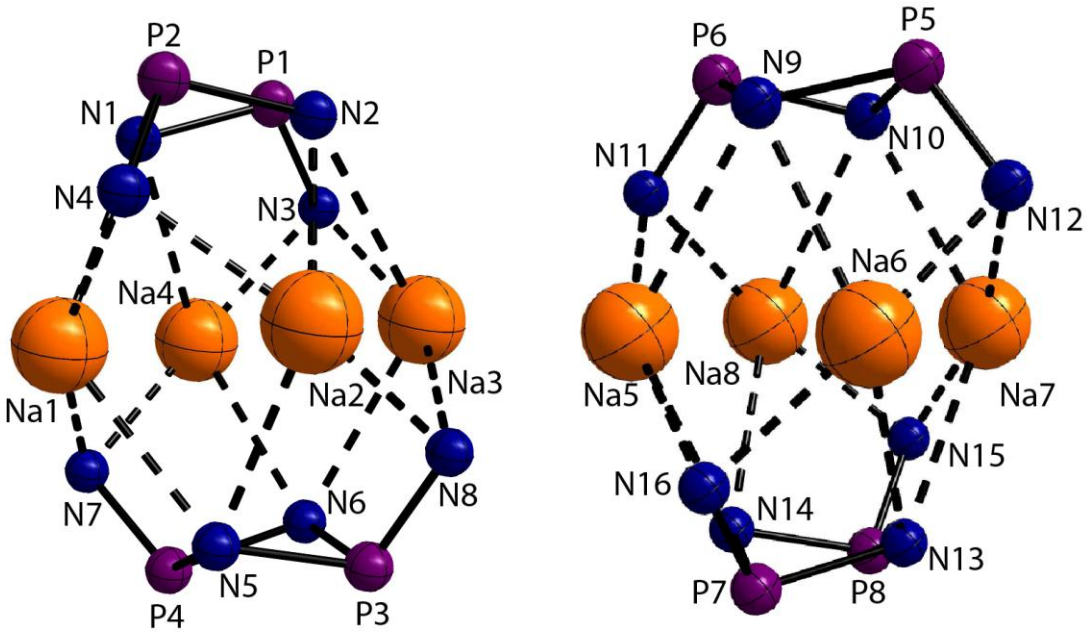

Figure S2

Molecular structure of $[Na_4(P_2(\mu\text{-}N^tBu)_2(N^tBu)_2)_2]$. Bond lengths (Å): Na1–N4 2.350(8), Na1–N8 2.350(8), Na1–N1 2.801(7), Na1–N6 2.849(7), Na1–Na4 3.104(5), Na1–Na2 3.109(5), Na1–P2 3.170(4), Na1–P4 3.208(4), Na2–N7 2.346(7), Na2–N4 2.348(7), Na2–N2 2.766(7), Na2–N6 2.860(8), Na2–P2 3.166(4), Na2–P3 3.203(4), Na3–N3 2.304(8), Na3–N7 2.310(7), Na3–N5 2.886(8), Na3–N2 2.961(7), Na3–P3 3.195(4), Na3–P1 3.239(4), Na4–N8 2.313(8), Na4–N3 2.337(8), Na4–N5 2.833(8), Na4–N1 2.960(8), Na4–P4 3.167(4), Na4–P1 3.224(5), Na5–N16 2.314(8), Na5–N11 2.329(8), Na5–N14 2.847(8), Na5–N9 2.864(7), Na5–P7 3.154(4), Na5–P6 3.174(4), Na6–N16 2.339(8), Na6–N12 2.344(7), Na6–N9 2.903(7), Na6–N13 3.018(7), Na7–N15 2.335(7), Na7–N12 2.343(7), Na7–N10 2.762(7), Na7–N13 2.808(7), Na7–P5 3.166(4), Na7–P8 3.167(4), Na8–N11 2.317(7), Na8–N15 2.327(8), Na8–N14 2.915(7), Na8–N10 3.045(7), Na8–P8 3.214(4), Na8–P6 3.283(4), P1–N3 1.637(8), P1–N2 1.768(7), P1–N1 1.792(7), P2–N4 1.640(7), P2–N2 1.790(7), P2–N1 1.793(7), P3–N7 1.650(7), P3–N5 1.794(7), P3–N6 1.798(6), P4–N8 1.629(7), P4–N5 1.795(7), P4–N6 1.801(7), P5–N12 1.648(6), P5–N10 1.782(6), P5–N9 1.795(7), P6–N11 1.644(7), P6–N10 1.790(6), P6–N9 1.799(6), P7–N16 1.627(7), P7–N13 1.787(7), P7–N14 1.799(7), P8–N15 1.645(7), P8–N14 1.788(7), P8–N13 1.794(7), N1–C1 1.512(11), N2–C5 1.500(11), N3–C13 1.497(12), N4–C9 1.498(10), N5–C17 1.518(10), N6–C21 1.506(10), N7–C29 1.496(10), N8–C25

1.516(11), N9–C33 1.497(10), N10–C37 1.506(10), N11–C41 1.488(10), N12–C45 1.499(10), N13–C49 1.511(11), N14–C53 1.502(11), N15–C57 1.491(11), N16–C61 1.540(12). Bond angles (°): N4–Na1–N8 155.9(3), N4–Na1–N1 64.4(2), N8–Na1–N1 105.5(3), N4–Na1–N6 102.9(2), N8–Na1–N6 63.1(2), N1–Na1–N6 123.4(2), N4–Na1–P2 30.17(18), N8–Na1–P2 136.1(2), N1–Na1–P2 34.24(15), N6–Na1–P2 116.75(18), Na4–Na1–P2 88.33(12), N4–Na1–P4 134.2(2), N8–Na1–P4 29.22(18), N1–Na1–P4 118.67(18), N6–Na1–P4 33.93(15), P2–Na1–P4 134.89(13), N7–Na2–N4 156.5(3), N7–Na2–N2 105.6(2), N4–Na2–N2 64.5(2), N7–Na2–N6 63.7(2), N4–Na2–N6 102.6(2), N2–Na2–N6 123.1(2), N7–Na2–P2 136.5(2), N4–Na2–P2 30.23(17), N2–Na2–P2 34.30(14), N6–Na2–P2 116.55(18), N7–Na2–P3 29.80(16), N4–Na2–P3 134.1(2), N2–Na2–P3 118.69(18), N6–Na2–P3 33.91(13), P2–Na2–P3 134.99(14), N3–Na3–N7 150.7(3), N3–Na3–N5 103.4(3), N7–Na3–N5 63.5(2), N3–Na3–N2 61.2(2), N7–Na3–N2 100.7(2), N5–Na3–N2 119.2(2), N3–Na3–P3 132.4(2), N7–Na3–P3 29.71(17), N5–Na3–P3 33.84(15), N2–Na3–P3 113.18(18), N3–Na3–P1 28.48(19), N7–Na3–P1 130.2(2), N5–Na3–P1 114.92(18), N2–Na3–P1 32.74(14), P3–Na3–P1 129.87(14), N8–Na4–N3 152.5(3), N8–Na4–N5 64.0(2), N3–Na4–N5 104.1(3), N8–Na4–N1 101.7(3), N3–Na4–N1 62.4(2), N5–Na4–N1 122.3(2), N8–Na4–P4 29.72(18), N3–Na4–P4 133.8(2), N5–Na4–P4 34.26(14), N1–Na4–P4 115.17(18), N8–Na4–P1 131.8(2), N3–Na4–P1 29.04(19), N5–Na4–P1 116.96(19), N1–Na4–P1 33.35(14), P4–Na4–P1 132.11(15), N16–Na5–N11 156.1(3), N16–Na5–N14 64.3(2), N11–Na5–N14 104.3(2), N16–Na5–N9 104.6(3), N11–Na5–N9 64.2(2), N14–Na5–N9 127.0(2), N16–Na5–P7 29.88(18), N11–Na5–P7 135.3(2), N14–Na5–P7 34.40(15), N9–Na5–P7 119.55(18), N16–Na5–P6 135.2(2), N11–Na5–P6 30.07(17), N14–Na5–P6 119.07(19), N9–Na5–P6 34.17(13), P7–Na5–P6 135.70(15), N16–Na6–N12 148.4(3), N16–Na6–N9 102.8(3), N12–Na6–N9 63.0(2), N16–Na6–N13 59.9(2), N12–Na6–N13 100.2(2), N9–Na6–N13 117.9(2), N16–Na6–P5 131.5(2), N12–Na6–P5 29.48(16), N9–Na6–P5 33.62(14), N13–Na6–P5 112.97(17), N16–Na6–P7 27.42(19), N12–Na6–P7 129.2(2), N9–Na6–P7 113.97(18), N13–Na6–P7 32.52(14), P5–Na6–P7 129.49(13), N15–Na7–N12 161.1(3), N15–Na7–N10 107.0(2), N12–Na7–N10 64.5(2), N15–Na7–N13 64.5(2), N12–Na7–N13 106.4(2), N10–Na7–N13 128.4(2), N15–Na7–P5 138.4(2), N12–Na7–P5 30.39(16), N10–Na7–P5 34.13(13), N13–Na7–P5 120.89(18), N15–Na7–P8 30.24(17), N12–Na7–P8 138.3(2),

N10–Na7–P8 121.60(18), N13–Na7–P8 34.27(15), P5–Na7–P8 138.49(13), N11–Na8–N15 147.5(3), N11–Na8–N14 102.5(3), N15–Na8–N14 62.7(2), N11–Na8–N10 60.4(2), N15–Na8–N10 98.8(2), N14–Na8–N10 117.1(2), N11–Na8–P8 131.0(2), N15–Na8–P8 29.33(18), N14–Na8–P8 33.47(15), N10–Na8–P8 111.65(16), N11–Na8–P6 27.92(17), N15–Na8–P6 127.9(2), N14–Na8–P6 113.69(18), N10–Na8–P6 32.59(13), C55–Na8–P6 130.6(5), P8–Na8–P6 128.55(13), N3–P1–N2 107.0(4), N3–P1–N1 109.0(3), N2–P1–N1 80.0(3), N3–P1–Na4 43.9(3), N2–P1–Na4 100.3(2), N1–P1–Na4 65.2(2), N3–P1–Na3 42.1(3), N2–P1–Na3 64.9(2), N1–P1–Na3 102.6(2), Na4–P1–Na3 57.66(11), N4–P2–N2 106.6(3), N4–P2–N1 107.6(3), N2–P2–N1 79.3(3), N4–P2–Na2 46.1(2), N2–P2–Na2 60.5(2), N1–P2–Na2 98.6(2), N4–P2–Na1 46.0(3), N2–P2–Na1 97.7(2), N1–P2–Na1 61.5(2), Na2–P2–Na1 58.76(10), N7–P3–N5 107.5(3), N7–P3–N6 107.5(3), N5–P3–N6 80.1(3), N7–P3–Na3 43.9(2), N5–P3–Na3 63.6(2), N6–P3–Na3 100.0(2), N7–P3–Na2 45.0(2), N5–P3–Na2 100.2(2), N6–P3–Na2 62.5(2), Na3–P3–Na2 58.46(10), N8–P4–N5 107.4(3), N8–P4–N6 106.7(3), N5–P4–N6 80.0(3), N8–P4–Na4 44.7(3), N5–P4–Na4 62.6(2), N6–P4–Na4 98.6(2), N8–P4–Na1 44.8(3), N5–P4–Na1 100.1(2), N6–P4–Na1 62.0(2), Na4–P4–Na1 58.28(11), N12–P5–N10 106.4(3), N12–P5–N9 107.9(3), N10–P5–N9 80.5(3), N12–P5–Na7 46.0(2), N10–P5–Na7 60.4(2), N9–P5–Na7 97.4(2), N12–P5–Na6 44.4(2), N10–P5–Na6 101.4(2), N9–P5–Na6 63.6(2), Na7–P5–Na6 58.73(10), N11–P6–N10 107.5(3), N11–P6–N9 108.6(3), N10–P6–N9 80.2(3), N11–P6–Na5 45.2(2), N10–P6–Na5 98.7(2), N9–P6–Na5 63.4(2), N11–P6–Na8 41.3(2), N10–P6–Na8 66.4(2), N9–P6–Na8 104.0(2), Na5–P6–Na8 57.90(11), N16–P7–N13 106.5(3), N16–P7–N14 108.5(3), N13–P7–N14 79.9(3), N16–P7–Na5 45.1(3), N13–P7–Na5 98.2(2), N14–P7–Na5 63.4(2), N16–P7–Na6 41.5(3), N13–P7–Na6 65.2(2), N14–P7–Na6 103.1(2), Na5–P7–Na6 57.77(11), N15–P8–N14 107.8(3), N15–P8–N13 107.5(3), N14–P8–N13 80.0(3), N15–P8–Na7 45.6(2), N14–P8–Na7 98.0(2), N13–P8–Na7 61.8(2), N15–P8–Na8 43.9(3), N14–P8–Na8 64.0(2), N13–P8–Na8 102.2(2), Na7–P8–Na8 58.90(10), C1–N1–P1 116.0(5), C1–N1–P2 116.3(5), P1–N1–P2 93.9(3), C1–N1–Na1 108.6(5), P1–N1–Na1 130.6(3), P2–N1–Na1 84.2(3), C1–N1–Na4 108.0(5), P1–N1–Na4 81.4(3), P2–N1–Na4 132.3(3), Na1–N1–Na4 65.15(17), C5–N2–P1 116.8(5), C5–N2–P2 115.7(5), P1–N2–P2 94.8(3), C5–N2–Na2 105.4(5), P1–N2–Na2 132.5(3), P2–N2–Na2 85.2(3), C5–N2–Na3 105.0(5), P1–N2–Na3 82.3(3), P2–N2–Na3 135.2(3), Na2–N2–Na3

66.02(17), C13–N3–P1 117.6(6), C13–N3–Na3 116.3(6), P1–N3–Na3 109.4(4), C13–N3–Na4 117.2(6), P1–N3–Na4 107.1(4), Na3–N3–Na4 84.4(3), C9–N4–P2 117.7(5), C9–N4–Na2 120.4(5), P2–N4–Na2 103.7(3), C9–N4–Na1 122.2(5), P2–N4–Na1 103.8(3), Na2–N4–Na1 82.9(2), C17–N5–P3 115.0(5), C17–N5–P4 117.8(5), P3–N5–P4 93.9(3), C17–N5–Na4 108.8(5), P3–N5–Na4 131.5(3), P4–N5–Na4 83.1(3), C17–N5–Na3 105.9(5), P3–N5–Na3 82.5(3), P4–N5–Na3 132.9(3), Na4–N5–Na3 66.03(18), C21–N6–P3 116.0(5), C21–N6–P4 116.0(5), P3–N6–P4 93.6(3), C21–N6–Na1 106.5(4), P3–N6–Na1 133.3(3), P4–N6–Na1 84.0(3), C21–N6–Na2 106.8(5), P3–N6–Na2 83.6(2), P4–N6–Na2 133.3(3), Na1–N6–Na2 65.98(17), C29–N7–P3 116.6(5), C29–N7–Na3 118.2(5), P3–N7–Na3 106.4(3), C29–N7–Na2 121.1(5), P3–N7–Na2 105.2(3), Na3–N7–Na2 84.3(2), C25–N8–P4 117.9(6), C25–N8–Na4 119.6(6), P4–N8–Na4 105.6(3), C25–N8–Na1 118.7(5), P4–N8–Na1 106.0(4), Na4–N8–Na1 83.5(3), C33–N9–P5 117.3(5), C33–N9–P6 117.4(5), P5–N9–P6 93.7(3), C33–N9–Na5 108.5(5), P5–N9–Na5 129.8(3), P6–N9–Na5 82.4(2), C33–N9–Na6 105.6(5), P5–N9–Na6 82.8(2), P6–N9–Na6 132.7(3), Na5–N9–Na6 65.44(17), C37–N10–P5 116.7(5), C37–N10–P6 116.8(5), P5–N10–P6 94.5(3), C37–N10–Na7 108.0(4), P5–N10–Na7 85.4(2), P6–N10–Na7 129.3(3), C37–N10–Na8 104.6(4), P5–N10–Na8 135.2(3), P6–N10–Na8 81.0(2), Na7–N10–Na8 65.20(16), C41–N11–P6 118.7(5), C41–N11–Na8 114.7(5), P6–N11–Na8 110.8(3), C41–N11–Na5 118.1(5), P6–N11–Na5 104.7(3), Na8–N11–Na5 84.6(2), C45–N12–P5 118.4(5), C45–N12–Na7 121.5(5), P5–N12–Na7 103.6(3), C45–N12–Na6 117.7(5), P5–N12–Na6 106.1(3), Na7–N12–Na6 83.8(2), C49–N13–P7 116.3(6), C49–N13–P8 115.5(5), P7–N13–P8 94.8(3), C49–N13–Na7 109.3(5), P7–N13–Na7 129.6(3), P8–N13–Na7 83.9(3), C49–N13–Na6 106.2(5), P7–N13–Na6 82.3(3), P8–N13–Na6 134.3(3), Na7–N13–Na6 64.88(16), C53–N14–P8 115.3(6), C53–N14–P7 116.8(5), P8–N14–P7 94.6(3), C53–N14–Na5 110.8(5), P8–N14–Na5 129.4(3), P7–N14–Na5 82.2(3), C53–N14–Na8 105.8(5), P8–N14–Na8 82.5(2), P7–N14–Na8 133.7(3), Na5–N14–Na8 65.73(17), C57–N15–P8 118.7(5), C57–N15–Na8 116.2(6), P8–N15–Na8 106.8(4), C57–N15–Na7 120.9(6), P8–N15–Na7 104.1(3), Na8–N15–Na7 84.6(2), C61–N16–P7 117.9(5), C61–N16–Na5 120.1(6), P7–N16–Na5 105.0(3), C61–N16–Na6 113.7(5), P7–N16–Na6 111.1(4), Na5–N16–Na6 84.2(3), C4–C1–N1 114.5(7), C4–C1–C3 110.0(9),

N1–C1–C3 108.8(7), C4–C1–C2 107.4(8), N1–C1–C2 107.7(7), C3–C1–C2 108.4(9), N2–C5–C8 110.4(7), N2–C5–C7 107.7(7), C8–C5–C7 107.4(8), N2–C5–C6 113.5(8).