

Formation of (1,2-dihydroxynaphth-4-yl)[tris(diethylamino)]phosphonium bromides in the reaction of 1,2-naphthoquinones with tris(diethylamino)phosphine

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Spectral characteristics for compounds 7 and 9

Compound 7

³¹P NMR (36.48 MHz, CDCl₃) δ_P: 50.5 [m (s)]. ¹H NMR (400 MHz, CDCl₃) δ_H: 7.63–7.11 (br. m, H⁵, H⁶, H⁷, H⁸), 6.58 (d, H³, ³J_{PCCH} 13.6 Hz), 4.42–3.37 (br. m, CH₂), 1.14–1.11 (br. m, Me). ¹³C NMR (150.9 MHz, CDCl₃) δ_C: 146.28 [m (d), C¹, ⁴J_{PCCCC} 3.3 Hz, ³J_{HCCC} 4.0–5.0 Hz, ³J_{HCCC} 7.0–8.0 Hz], 140.61 [br. dd (d), C², ³J_{PCCC} 21.2 Hz, ²J_{HCC} 3.8 Hz], 129.36 [dd (d), C³, ²J_{PCC} 14.3 Hz, ¹J_{HC} 159.4 Hz], 105.31 [ddd (d), C⁴, ¹J_{PC} 155.0 Hz, ³J_{HCCC} 3.6 Hz, ²J_{HCC} 1.5–1.7 Hz], 128.74 [m (d), C^{4a}, ²J_{PCC} 9.7–9.8 Hz], 125.51 [ddd (d), C⁵, ³J_{PCCC} 6.0 Hz, ¹J_{HC} 160.5 Hz, ³J_{HCCC} 8.4 Hz], 124.01 [dddd (br. s), C⁶, ¹J_{HC} 165.0 Hz, ³J_{HCCC} 4.8 Hz, ²J_{HCC} 3.2 Hz, ²J_{HCC} 1.5 Hz], 125.55 [dd (s), C⁷, coincides with C⁵], 124.01 [dddd (d), C⁸, ¹J_{HC} 157.0–158.0 Hz, ⁴J_{PCCCC} 4.3 Hz, ²J_{HCC} 3.1–3.5 Hz, ³J_{HCCC} 7.1–7.4 Hz, ⁴J_{HCCCC} 1.2 Hz], 126.73 [m (d), C^{8a}, ³J_{PCCC} 14.4 Hz, ³J_{HCCC} 6.8 Hz, ³J_{HCCC} 7.9 Hz], 40.92 [qm (d), NCH₂, ¹J_{HC} 127.6 Hz, ²J_{PNC} 4.7 Hz], 13.30 [qm (d), Me, ³J_{PNCC} 2.4 Hz, ¹J_{HC} 126.7 Hz]. IR (Nujol, n/cm⁻¹): 3040–3080 (br., OH), 1617, 1596 (C=C), 1574 (C=C), 1509, 1346, 1288, 1234, 1204, 1156 (P–N), 1108, 1078, 1056, 1016, 977, 924, 888, 771, 737, 659, 535, 445. Found (%): C, 59.68; H, 8.30; N, 9.37; P, 6.90; Cl, 7.94. Calc. for C₂₂H₃₇ClN₃O₂P (%): C, 59.79; H, 8.38; N, 9.51; P, 7.02; Cl, 8.04.

Compound 9

³¹P NMR (36.48 MHz, CDCl₃) δ_P: 45.20 [m (s)]. ¹H NMR (400 MHz, CDCl₃) δ_H: 1.24 (t, NCMe, ³J_{HCCH} 7.0 Hz), 3.26 (dq, NCH₂, ³J_{PNCH} 10.8 Hz, ³J_{HCCH} 7.0 Hz), 7.57 (dd, H⁸, ³J_{HCCH} 8.8 Hz, ⁵J_{PCCCCH} 1.3 Hz), 8.22 (dd, H⁷, ³J_{HCCH} 8.8 Hz, ⁴J_{HCCCH} 1.7 Hz), 8.27 (br. m, H⁵, ³J_{HCCH} 8.8 Hz, ⁴J_{HCCCH} 1.7 Hz). ¹³C NMR (150.9 MHz, CDCl₃) δ_C: 146.64 [br. m (d), C¹, ⁴J_{PCCCC} 2.9 Hz], 136.97 [br. d (d), C², ³J_{PCCC} 14.2 Hz], 133.80 [br. m (d), C³, ²J_{PCC} 4.8 Hz], 105.65 [dd (d), C⁴, ¹J_{PC} 156.5 Hz, ³J_{HCCC} 3.5 Hz], 130.92 [ddd (d), C^{4a}, ²J_{PCC} 6.6 Hz, ³J_{HCCC} 5.0–6.0 Hz], 126.88 [ddd (d), C⁵, ³J_{PCCC} 5.4 Hz, ¹J_{HC} 164.1 Hz, ⁴J_{HCCCC} 1.5 Hz], 122.00 [ddd (s), C⁶, ³J_{HCCC} 12.5 Hz, ²J_{HCC} 5.7 Hz, ²J_{HCC} 2.4 Hz], 129.34 [dd (s), C⁷, ¹J_{HC} 169.9 Hz, ³J_{HCCC} 6.1 Hz], 125.55 [dd (s), C⁸, ¹J_{HC} 167.2 Hz, ²J_{HCC} 1.2 Hz], 123.29 [br. ddd (d), C^{8a}, ³J_{PCCC} 12.5 Hz, ³J_{HCCC} 7.4–7.5 Hz, ³J_{HCCC} 7.4–7.5 Hz], 42.56 [tdq (d), NCH₂, ¹J_{HC} 127.3 Hz, ²J_{PNC} 4.5 Hz, ²J_{HCC} 3.7–4.1 Hz], 13.53 [qdt (d), Me, ¹J_{HC} 127.3 Hz, ³J_{PNCC} 3.0 Hz, ²J_{HCC} 3.0 Hz]. IR (Nujol, n/cm⁻¹): 3380 (br., OH), 3177 (br., OH), 1577 (C=C), 1345, 1306, 1275, 1202, 1153 (P–N), 1080, 1030, 1012, 967, 939, 797, 782, 723, 646, 498. Found (%): C, 43.29; H, 5.50; N, 6.98; P, 6.13; Br, 26.40; Cl, 5.90. Calc. for C₂₂H₃₅Br₂ClN₃O₂P (%): C, 44.03; H, 5.84; N, 7.00; P, 6.40; Br, 26.67; Cl, 5.92.