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Bis(p-allylhimachalene)dichlorodipalladium

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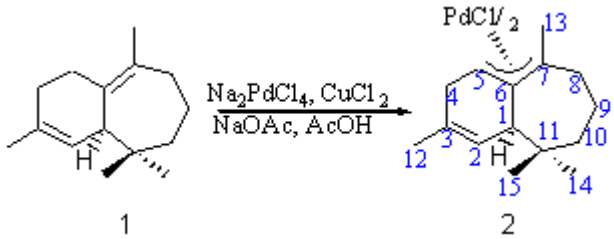
The title organometallic compound, potentially important synthetic intermediate, was prepared regio- and stereospecifically from b-himachalene ((4aR)-3,5,5,9-Tetramethyl-2,4a,5,6,7,8-hexahydro-1*H*-benzocycloheptene), essentiel component of *cedrus atlantica oil* [1].



A red solution of Na_2PdCl_4, prepared *in situ* by mixing $PdCl_2$ (100 mg, 0.56 mmol), LiCl (71.2 mg, 1.45 mmol) and sodium acetate (238 mg, 2.9 mmol) in glacial acetic acid (10 mL), was stirred at 85°C for 30 min. The mixture was then cooled to 60°C, after which $CuCl_2$ (227 mg, 1.69 mmol) and b-himachalene **1** (346 mg, 1.69 mmol) were added. The resulting mixture was further stirred for 12 h. The resulting mixture was filtered and extracted with ether (3x20 mL). The combined organic layers was neutralized with a saturated solution of $NaHCO_3$, dried over anhydrous $MgSO_4$ and concentrated to afford an orange oil. Flash chromatography of the crude product over silica gel using hexane-ethyl acetate (95:5) furnished analytically pur complex **2** (54% yield) as a yellow solid [2].

M.p. 124-125 °C (decomposition).

^{1}H NMR (400 MHz, CD_2Cl_2): 5.46 (1H, m, =CH), 4.32 (1H, t, J=3Hz, HC5), 3.37 (1H, d, J=3.3Hz, HC1), 1.7 (3H, s, CH_3), 1.15 (3H, s, CH_3), 1.01 (3H, s, CH_3), 0.72 (3H, s, CH_3).

^{13}C NMR (100 MHz, CD_2Cl_2) : 132.0 (C3), 120.1 (C2), 119.2 (C6), 86.5 (C7), 73.3 (C5), 45.9 (C1), 43.5 (CH_2), 37.43 (C11), 35.5 (CH_2), 31.8 (CH_2), 28.7 (CH_3), 23.6 (CH_3), 23.1 (CH_3), 22.5 (CH_3), 18.8 (CH_2).

Anal.Calc. for $(C_{15}H_{23}PdCl)_2$: C 52.21, H 6.66, Cl 10.14, Pd 30.84. Found: C 52.22, H 6.70, Cl 10.10, Pd 70.72.

References

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2. Chiaroni, A.; Riche, C.; El Firdoussi, L.; Benharref, A.; Karim, A. *Acta Cryst.* **1993**, *C49*, 365-368.

Sample availability: available from the authors and MDPI.