The reaction of lithiated Ph(H)PCH₂P(H)Ph with (ClCH₂)₂PPh yields 1,3,5-Triphenyl-1,3,5-triphosphorinane (1) as a mixture of two isomers. The isomers have been separated by column chromatography and characterized by NMR spectroscopy and single crystal X-ray diffraction as the eq-eq-eq (1a) and eq-eq-ax-isomers (1b). (1a) crystallizes with $a = 1326.9(3)$ pm and $c = 933.4(3)$ pm in the rhombohedral space group $R3$ and displays 3-fold symmetry, showing an eq-eq-eq conformation. (1b) crystallizes in the monoclinic space group $P2_1/c$ with $a = 984.09(12)$ pm, $b = 1757.5(2)$ pm, $c = 1083.13(12)$ pm, $\beta = 93.329(2)^\circ$ and displays an ax-eq-eq conformation.

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