Synthesis and Coordination Chemistry of a Tridentate
o-Phenylene-bridged Diphosphine-NHC System

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Experimental for X-Ray Crystallography

Data Collection

All crystals were mounted on a glass fiber and measurements were made on a Bruker X8 APEX diffractometer with graphite monochromated Mo-Kα radiation. The data were collected at a temperature of -100.0 + 0.1 °C.

Data Reduction

Data were collected and integrated using the Bruker SAINT software package. The linear absorption coefficient, μ, for Mo-Kα radiation is 7.96 cm⁻¹. Data were corrected for absorption effects using the multi-scan technique (SADABS). The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement
The structures were solved by direct methods\. Crystal system 7a exhibited merohedral twinning and a twin law of 00-1, 0-10, -100 was employed in the refinement of the structure. 7b crystallizes with disordered solvent in the lattice. This disordered solvent molecule could not be modeled reasonably; therefore the PLATON/SQUEEZE program was used to correct the data for any unresolved residual electron density in the lattice. The formula and any subsequent values calculated from it reflect the presence of one molecule of solvent in the asymmetric unit. Also one scan-set for 7b was omitted as the data was found to be inconsistent with other scan-sets. All non-hydrogen atoms were refined anisotropically, while all hydrogen atoms except H1 were placed in calculated positions but were not refined. H1 was located in a difference map and refined isotropically. All refinements were performed using the SHELXL\textsuperscript{5} crystallographic software package of Bruker-AXS.

References