

Supporting Information (om-2012-01046t)

Synthesis and Catalytic Activity of Molybdenum-Dinitrogen Complexes Bearing Unsymmetric PNP-Type Pincer Ligands

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Table S1. X-ray Crystallographic Data for $[\text{MoCl}_3(\mathbf{3b})]$ (**4b**), $[\text{MoCl}_3(\mathbf{3d})]$ (**4d**), $[\text{Mo}(\text{N}_2)_2(\mathbf{3b})]_2(\mu\text{-N}_2)$ (**5b**· $3\text{C}_6\text{H}_6$), and $[\text{Mo}(\text{N}_2)_2(\mathbf{3c})]_2(\mu\text{-N}_2)$ (**5c**).

	4b	4d	5b · $3\text{C}_6\text{H}_6$	5c
chemical formula	$\text{C}_{27}\text{H}_{35}\text{Cl}_3\text{MoNP}_2$	$\text{C}_{27}\text{H}_{47}\text{Cl}_3\text{MoNP}_2$	$\text{C}_{72}\text{H}_{88}\text{Mo}_2\text{N}_{12}\text{P}_4$	$\text{C}_{42}\text{H}_{78}\text{Mo}_2\text{N}_{12}\text{P}_4$
formula weight	637.83	649.92	1437.34	1066.93
dimensions of crystals	$0.15 \times 0.15 \times 0.15$	$0.40 \times 0.30 \times 0.20$	$0.30 \times 0.20 \times 0.10$	$0.30 \times 0.02 \times 0.02$
crystal system	orthorhombic	orthorhombic	monoclinic	monoclinic
space group	<i>Pbca</i>	<i>Pbca</i>	<i>C2/c</i>	<i>P2₁/n</i>
<i>a</i> , Å	16.484(2)	16.1351(4)	19.8028(4)	12.4475(4)
<i>b</i> , Å	18.120(2)	18.2992(4)	14.0359(3)	22.2321(7)
<i>c</i> , Å	19.005(2)	20.8235(5)	26.7018(5)	19.5339(7)
α , deg	90	90	90	90
β , deg	90	90	105.0150(7)	91.4060(9)
γ , deg	90	90	90	90
<i>V</i> , Å ³	5677(1)	6148.4(3)	7168.4(3)	5404.0(3)
<i>Z</i>	8	8	4	4
ρ_{calcd} , g cm ⁻³	1.493	1.404	1.332	1.311
<i>F</i> (000)	2616	2712	2992	2232
μ , cm ⁻¹	8.734	8.073	4.881	6.215
trans. factors range	0.792–0.877	0.698–0.851	0.766–0.952	0.767–0.988
no. reflections measured	43693	57644	34592	51032
no. unique reflections	6503 ($R_{\text{int}} = 0.030$)	7018 ($R_{\text{int}} = 0.041$)	8204 ($R_{\text{int}} = 0.032$)	12322 ($R_{\text{int}} = 0.090$)
no. parameters refined	342	354	374	619
<i>R</i> 1 ($I > 2 \sigma(I)$) ^a	0.0301	0.0266	0.0495	0.0502
<i>wR</i> 2 (all data) ^b	0.0510	0.0509	0.1167	0.0715
GOF (all data) ^c	1.038	1.007	1.020	1.011
max diff peak / hole, e Å ⁻³	0.63/−0.43	0.41/−0.51	2.28/−0.76	1.60/−1.50

^a $R1 = \sum ||F_o - |F_c|| / \sum |F_o|$. ^b $wR2 = [\sum w(F_o^2 - F_c^2)^2 / \sum w(F_o^2)^2]^{1/2}$, $w = 4F_o^2/q\sigma(F_o^2)$ [$q = 7.1$ (**4b**); $q = 3.9$ (**4d**); $q = 19.0$ (**5b**· $3\text{C}_6\text{H}_6$); $q = 1.8$ (**5c**)]. ^c GOF = $[\sum w(F_o^2 - F_c^2)^2 / (N_o - N_{\text{params}})]^{1/2}$.