Cp*Rh(S₂N₂) was prepared as a microcrystalline solid using [S₄N₃]Cl in liquid ammonia or [nBu₂Sn(S₂N₂)]₂. It was characterised by NMR, IR and Raman spectroscopy and mass spectrometry. Low-temperature crystal structures of Cp*Co(S₂N₂) and Cp*Ir(S₂N₂) were determined. The experimental characterisation of the Cp*M(S₂N₂) complexes is complemented by calculated geometries and bond orders at the DFT/B1B95 level of theory.

The reaction of hydrazide with carbonyl chloride in the presence of sodium carbonates lead to the corresponding 1,2-diacylhydrazines (1a-t, R¹C(O)NHNHC(O)R², R¹ = aryl, R² = aryl or alkyl) in moderate to excellent yield (57 - 90%). The latter react with 2,4-bis(phenyl)-1,3-diselenadiphenethane-2,4-diselenide (Woollins’ reagent, WR) in refluxing toluene to give a series of new 2,5-disubstituted-1,3,4-selenadiazoles (2a-t, 51-99% yield). All compounds were characterized spectroscopically and six compounds were characterized crystallographically.

In the isomorphous binuclear Cu₂X₂L₂ systems (L = (8-phenylthionaphth-1-yl)diphenylphosphine the Cu…Cu separation is reduced as the halide size increases.


References
Facile Synthesis and Structure of Novel 2,5-Disubstituted 1,3,4-Selenadiazoles G Hua, Y Li, A L. Fuller, A M. Z. Slawin and J. D Woollins, Eur J Org Chem 2009, 1612–1618 DOI: 10.1002/ejoc.200900013