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Metal phosphine complexes incorporating alkyl substituents with ethane and ethylene backbones

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Abstract

The new chelating bis-phosphine ligands 1,2-bis(butylphenylphosphino)ethane (**bppe**) and *cis*-1,2-bis(butylphenylphosphino)ethylene (**bppey**) have been synthesised by (i) a one-pot synthesis from Ph_2PBu and Li/dihalo alkyl, (ii) the reaction of 1,2-bis(diphenylphosphino)ethane (**dppe**) or *cis*-1,2-bis(diphenylphosphino)ethylene (**dppey**) and Li/*n*-BuCl and, (iii) in the case of **bppe** by sequential synthesis (all intermediates were isolated) from Ph_2PBu *via* PhBuPLi and Ph_2PH. **bppe** and **bppey** as well as the new lithium phosphide $[(TMEDA)\bullet LiPPh(Bu)]_2$ (**17**) were fully characterised by multinuclear NMR spectroscopy, mass spectrometry and, in the case of $[(TMEDA)\bullet LiPPh(Bu)]_2$ (**17**), by X-ray crystallography.

Reaction of the bis-phosphines **bppe** and **bppey** with suitable metal precursors yielded the corresponding metal complexes: $[PdCl_2(bppe)]$ (**18**), $[Pd(bppe)_2](ClO_4)_2$ (**20**), $[(AuCl)_2(bppe)]$ (**21a**), $[(AuCl)_2(bppey)]$ (**21b**), $[Au(bppe)_2]Cl$ (**22a**), $[Au(bppey)_2]Cl$ (**22b**), $[(AgNO_3)_2(bppe)]$ (**23**) and $[Au(bppe)_2]ClO_4$ (**24**) in moderate to good yields. All were characterised by multinuclear NMR spectroscopy and mass spectrometry, while **18** and **20** were further characterised by X-ray crystallography.

Preliminary stability tests showed, that of all new metal complexes only **18**, **20** and **22a** were adequately stable to justify further tests for anti-tumour activity. The cationic complexes **20** and **22a** showed activity against HeLa cells while the neutral complex **18** was not active. A comparison with the previously investigated analogous **dppe** and **dppey** complexes revealed that **20** and **22a** were found to be less active as a result of the replacement of a Ph groups with butyl groups in the phosphine ligand.