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Solvent-free procedure for the synthesis of tetrahydrosalen (H4salen) ligands by a solid state reaction using sodium borohydride as a reducing agent

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N,N´-bis(2-hydroxybenzyl) and N,N´-dibenzyl derivatives of 1,2, and 1,3-diamines, also known as tetrahydrosalen or salan ligands, are intimately involved with a number of metal coordination complexes, which include those elements located in groups 12, 13 and 14. These tetradentate ligands associated with metal centers display cis-octahedral coordination geometry, which can form two possible diastereomers (cis *fac-mer* and cis *fac-fac*), forming octahedral chiral complexes of the type [{ONNO}MX_2]. Different methods have been developed for the preparation of this class of compounds but the reduction of the corresponding diimines offers the possibility to produce a wide range of such diamines. A series of N,N´-dibenzyl or N,N´-(2-hydroxybenzyl)- ethane-1,2-diamine, -propane-1,3-diamine and -1,3-diamino-2-propanol (TetrahydroSalen ligands) were prepared in good yield by an efficient and highly eco-friendly protocol. The respective di-Schiff bases (Salen) precursors were prepared in good yield by using water as a solvent without the need for catalysis or the azeotropic removal of water. The reduction with sodium borohydride of the respective di-Schiff bases under catalyst-free and solvent-free conditions occurs readily and with good yields, ranging from 48% to 95%. The direct solid-solid reductive aminations were carried out by grinding the corresponding di-Schiff base and sodium borohydride with an agate mortar and pestle at room temperature. Then, the reactions were conducted by heating at 60-90°C on a hot plate and were complete within 15 minutes, producing tetrahydro-di-Schiff bases compounds. Compared to the previously reported methodologies, our protocol offers considerable benefits, including that it has a simple procedure, is environmentally benign, produces high yields, does not require the use of a catalyst, and allows for the product to be synthesized on the gram scale.

Biography

Jaime Ríos-Motta has his expertise in synthesis of heterocyclic compounds based on the use of aminal cage as preformed Mannich reagents. He has completed his PhD from the National University of Colombia. He belongs to the group of research in synthesis of heterocycles as Senior Investigator. He has published more than 80 papers in reputed journals and has been serving as an Editorial Board Member of reputed journals.

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