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phthaloyl chloride. The racemic product, containing carboxylic acid groups, was then condensed with enantiomerically pure amines to yield diastereomeric benzoamides that can be separated by flash chromatography. The ligands are modular in nature, easily modified, and readily assembled, Klosin adds.

Dowpharma scientists were involved in separating the new ligands and testing them in the rhodium -catalyzed asymmetric hydroformylation of styrene, allyl cyanide, and vinyl acetate. Under mild pressures (20 to 500 psig of  $CO/H_{2'}$  a mixture known as syngas) and temperatures (40 to 120

°C), they found high activities and selectivities for all three substrates, without any evidence of hydrogenation or other side reactions. At 60 °C and 500 psig of syngas, the best ligand found provides enantiomeric excesses of 87 to 95% at turnover frequencies of about 3,000 per hour.

"The rates seen are surprisingly fast," Landis says. "But many of the hydroformylation systems that have been reported before as being quite slow are, at least under the conditions we use, surprisingly fast also." The influence of pressure and temperature on enantioselectivity also varies significantly with substrate. Landis says a more detailed examination of the reaction kinetics is under way.

Although other ligands have been developed over the past 15 years, progress in enantioselective hydroformylation has lagged that in asymmetric hydrogenation, having been hindered by low turnover frequencies, ineffective control of regioselectivity, and limited applicability to ranges of substrates. The phospholane work, Landis believes, shows "significant progress toward practical catalytic production of chiral

materials in a process that is 100% atom efficient, involves gaseous reagents that are easily separated from products, and converts a simple achiral functional group into a chiral product with a more versatile functional group."



**EFFICIENT** Rhodium-catalyzed asymmetric hydroformylation of vinyl acetate, allyl cyanide, and styrene (shown top to bottom) under mild conditions (60 °C and 500 psig of syngas) using a bis-3,4-diazaphospholane ligand (0.004 mol % to 0.003 mol % Rh catalyst) offers high enantioselectivities for all three substrates.

IMAGE COURTESY OF NASA

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