Isomeric composition control of phenylisobutylketone oximes and their Pd-complexes measured by SFC

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Supercritical fluid chromatography (SFC) on chiral polysaccharide and polytartramide columns has been used to monitor isomeric purity of phenylisobutylketone oximes and their Pd-complexes. Chiral arylalkylketone oximes are considered as one of promising ligand types for Pd-based asymmetric hydrolysis catalysts. Crucial point of such objects synthesis is chiral purity control. Not only it's essential to use single enantiomers as ligands but the final product needs to be tested afterwards because in general racemization can occur during complex formation. Chromatography is reckoned to be the most effective way to measure enantiomeric composition of metal-organic compounds although some chromatographic techniques can be time-consuming and require plenty of expensive and hazardous solvents. In case of phenylisobutylketone oximes SFC has proved to be a competitive alternative to RP-HPLC providing the opportunity of simultaneous Z/E and chiral isomer separation. Pure CO₂ is capable to elute phenylisobutylketone oximes from polytartramide columns while significant amount of isopropanol as a cosolvent is required when using polysaccharide ones. Ab initio calculations have been exploited to illuminate reasons for very differing chromatographic behaviour of Z- and E-oximes. It was found that dipole moments of these two molecules depend very differently on various intrinsic movements. There is also an indirect evidence of intramolecular interaction between phenyl and oxime groups only in Z-form.