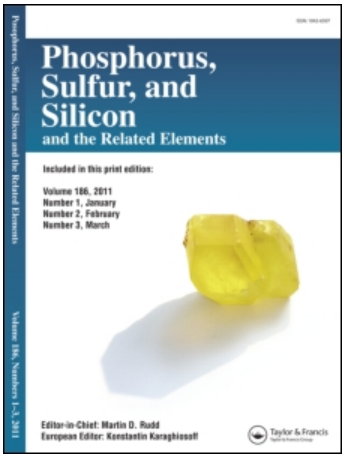

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Non-Racemic Mixtures of 1,3,2-Oxazaphosphacyclanes - Enantiomeric Composition Determination by NMR

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NON-RACEMIC MIXTURES OF 1,3,2-OXAZAPHOSPHACYCLANES - ENANTIOMERIC COMPOSITION DETERMINATION BY NMR

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The enantiomeric ratio in non-racemic mixtures of 2-anilino-2-oxo-1,3,2-oxazaphosphorinane (I) and 3-(α-methylbenzyl)-(I) [1] enantiomers was determined by measurement of the integral intensity ratio of two ^{31}P-NMR signals, assigned to enantiomers and differentiated due to the effect of statistically controlled associate-diastereoisomerism (SCADA), [2] when association occurs under conditions of fast exchange.

To prove the possible application of this analytical approach in the case of other oxazaphosphacyclanes we have synthesized the individual isomers of 2-anilino-3,4-dimethyl-5-phenyl-2-oxo-1,3,2-oxazaphospholane (II):

$$\underset{\text{(I): 1R,2S or 1S,2R}}{PhCH(OH)CH(CH_3)NHCH_3.HCl} + PhNHPOCl_2 \longrightarrow \underset{\text{mixtures: 2R,4S,5R and 2S,4S,5R or 2R,4R,5S and 2S,4R,5S}}{(II)}$$

$$\underset{1S,2R\text{-(I)}}{} \xrightarrow{1.POCl_3,\ 2.PhNH_2} \underset{2R,4R,5S\text{- and }2S,4R,5S\text{-(II)}}{}$$

Individual diastereomers of(II) have been separated from mixtures by column chromatography.Among the purified diastereomers, pairs of isomers,each of them being enantiomeric to other one, have been selected(2R,4R,5S and 2S,4S,5R, or 2S,4R,5S and 2R,4S,5R).NMR-Analysis of non-racemic mixtures of these pair isomers(II) has shown the absence of SCADA in phosphorus spectra and,in controversy,essential diastereomeric anisochronity of NH-protones, their integral intensity ratio being equal to proportion of enantiomers (II) in mixture.

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