



## Direct Chromatographic Resolution of P-Chiral Phosphinoylethenes on a Chiral Stationary Phase Containing *N,N'*-(3,5-Dinitrobenzoyl)-*trans*-1,2-diaminocyclohexane as Selector

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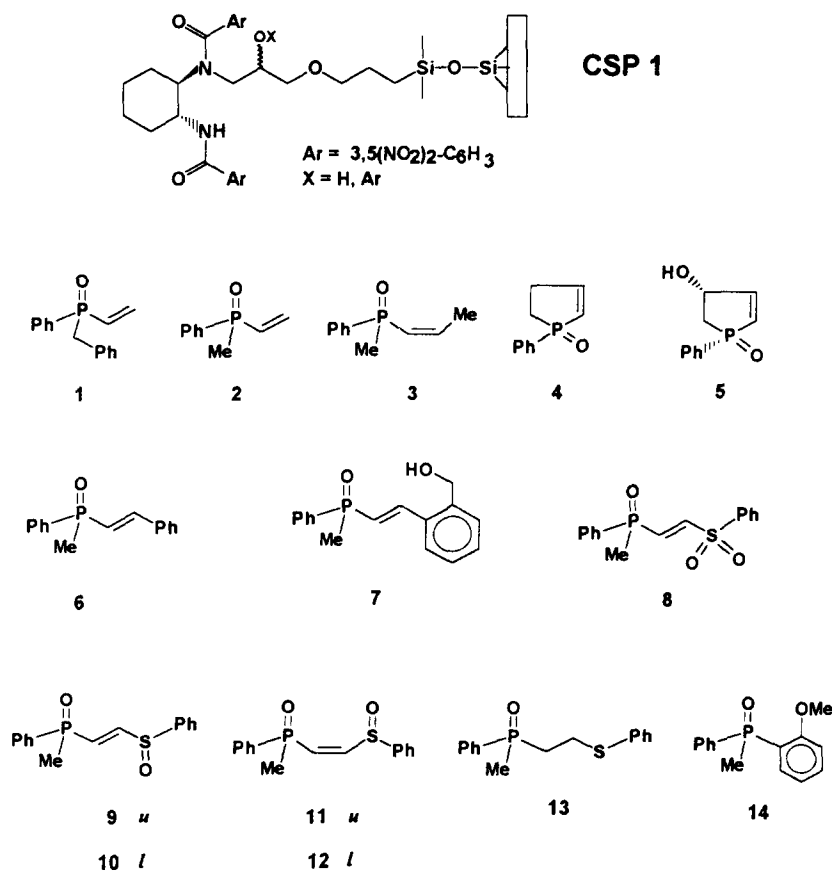
**Abstract:** *A  $\pi$ -acidic, totally synthetic HPLC chiral stationary phase is used to separate the enantiomers of a wide range of unsaturated P-chiral phosphine oxides. It allows for fast, sensitive and accurate e.e. determinations and secures easy access to optically pure enantiomers by chromatography on preparative columns.*

### Introduction

One of the most important problems in the field of enantioselective synthesis is to determine the enantiomeric excess (e.e.) of starting materials and products in an accurate and possibly straightforward manner. High-performance liquid chromatography (HPLC) utilizing chiral stationary phases (CSPs) offers many advantages in this respect: precision, reliability, speed of analysis, little amounts of the samples required, are the typical features of this technique.<sup>1</sup> Recently, P-chiral phosphine oxides bearing carbon unsaturation as the added functionality emerged as useful substrates for effecting chirality transfer from phosphorus to carbon<sup>2,3</sup> and for synthesis of other P-chiral phosphine oxides of known configuration.<sup>4,5</sup> However, such compounds usually lack the enantiopure models for comparison and they typically do not lend themselves to reliable quantitative NMR analysis with chiral shift reagents.<sup>2,6</sup> The development of a convenient and possibly general method for determination of enantiomeric purity of such compounds as well as for their resolution has become therefore important.

Recent success in HPLC resolution of a large diversity of organic as well as heteroorganic compounds on a synthetic chiral stationary phase based on *trans*-1,2-diaminocyclohexane as selector (CSP1)<sup>7</sup> prompted us to evaluate its utility also for resolution of P-chiral phosphinoylethenes. Previously reported direct

chromatographic resolutions of tertiary phosphine oxides on brush-type chiral phases were limited practically only to alkyldiarylphosphine oxides and most frequently required the presence of *P*-naphthyl substituent in the phosphine oxide structure to secure satisfactory resolution.<sup>8</sup> In this report we wish to demonstrate that a wide range of *P*-chiral phosphinoylethenes including both unsubstituted and terminally substituted ones of either *E* or *Z* configuration, cyclic and acyclic, can be readily resolved into enantiomers by means of HPLC utilizing a brush-type  $\pi$ -acidic CSP containing the *N,N'*-3,5-dinitrobenzoyl derivative of either (*R,R*)- or (*S,S*)-1,2-diaminocyclohexane covalently bound to silica microparticles (Chart I).



## Results and discussion

Chromatographic data obtained on the (*R,R*)-CSP1 for a number of phosphine oxides are listed in Table I; although we used, for comparison purposes, a single eluent throughout the whole series of compounds, baseline resolutions were observed in most of the cases,<sup>9</sup> thanks to the good selectivity and high chromatographic efficiency of our column (Fig. 1).

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