

First Enantioselective Synthesis of Vinyl Oxiranes
from Aldehydes and Ylides
generated from Allyl Halides and Chiral Sulfides

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Supporting Information

HPLC Enantiomer Analyses

Waters apparatus M996 detector with diode array detector (200 – 300 nm) Daicel Chiralpack AD column (L = 250 mm, ID = 4.6 mm), Millennium Software.

3-Ethenyl-2-phenyloxirane 10a: 100% n-hexane, rate = 0.5 mL.min⁻¹, T = 20 °C.
t-Butylbenzene as t₀ reference.

	Retention Time (min)	λ_{\max} (nm)	k'	α	Rs
<i>cis</i>	9.2	215	2.29	$\alpha_{1,2} = 1.09$	Rs _{1,2} = 0.78
<i>cis</i>	9.8		2.50		
<i>trans</i> -(2R,3R)	12.0		2.84	$\alpha_{3,4} = 1.48$	Rs _{3,4} = 2.31
<i>trans</i> -(2S,3S)	15.5		4.21		

Racemic 3-(1-methylethenyl)-2-phenyloxirane 10c: 100% n-hexane, rate = 1 mL.min⁻¹, room temperature.

	Retention Time (min)	λ_{\max} (nm)
<i>cis</i>	9.19	221.6
<i>cis</i>	9.88	
<i>trans</i> -(2R,3R)	11.70	
<i>trans</i> -(2S,3S)	16.28	

Racemic 2-phenyl-3-(1-cyclohexenyl)oxirane 10d: 90% n-hexane / 10% 2-propanol, rate = 1 mL.min⁻¹, room temperature.

	Retention Time (min)	λ_{\max} (nm)
<i>cis</i>	7.2	222.3
<i>cis</i>	7.5	
<i>trans</i> -(2R,3R)	8.8	
<i>trans</i> -(2S,3S)	26.4	

Racemic 2-phenyl-3-(1-phenylethenyl)oxirane 10e: Chirosebond n°2 C1 type column, 99% *n*-hexane / 1% 2-propanol, rate = 0.5 mL.min⁻¹, T = 6 °C.

	Retention Time (min)	λ_{\max} (nm)
<i>trans</i> -(2 <i>R</i> ,3 <i>R</i>)	21.5	225.2
<i>trans</i> -(2 <i>S</i> ,3 <i>S</i>)	19.5	

Racemic 2-(2-naphthyl)-3-(1-methylethenyl)oxirane 10f: Chirosebond n°2 C1 type column, 99% *n*-hexane / 1% 2-propanol, rate = 0.5 mL.min⁻¹, room temperature.

	Retention Time (min)	λ_{\max} (nm)
<i>trans</i> -(2 <i>R</i> ,3 <i>R</i>)	26.0	240.4
<i>trans</i> -(2 <i>S</i> ,3 <i>S</i>)	32.3	

Racemic 2-(4-chlorophenyl)-3-(methylethenyl)oxirane 10g: Chirosebond n°2 C1 type column, 99% *n*-hexane / 1% 2-propanol, rate = 0.5 mL.min⁻¹, T = 10 °C.

	Retention Time (min)	λ_{\max} (nm)
<i>cis</i>	17.5 (unseparated enantiomers)	230.0
<i>trans</i> -(2 <i>R</i> ,3 <i>R</i>)	19.9	
<i>trans</i> -(2 <i>S</i> ,3 <i>S</i>)	21.2	

Racemic 2-(2-thienyl)-3-(1-methylethenyl)oxirane 10h: Chirosebond n°2 C1 type column, 99% *n*-hexane / 1% 2-propanol, rate = 0.5 mL.min⁻¹, T = 6 °C.

	Retention Time (min)	λ_{\max} (nm)
<i>trans</i> -(2 <i>R</i> ,3 <i>R</i>)	17.4	240.4
<i>trans</i> -(2 <i>S</i> ,3 <i>S</i>)	19.5	

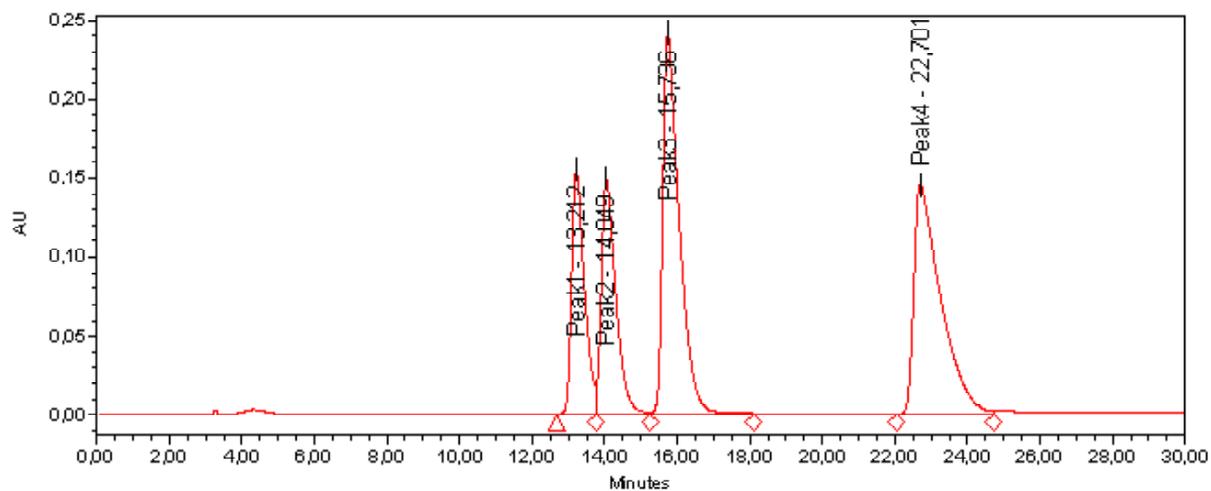
HPLC Chromatograms

3-Ethenyl-2-phenyloxirane 10a

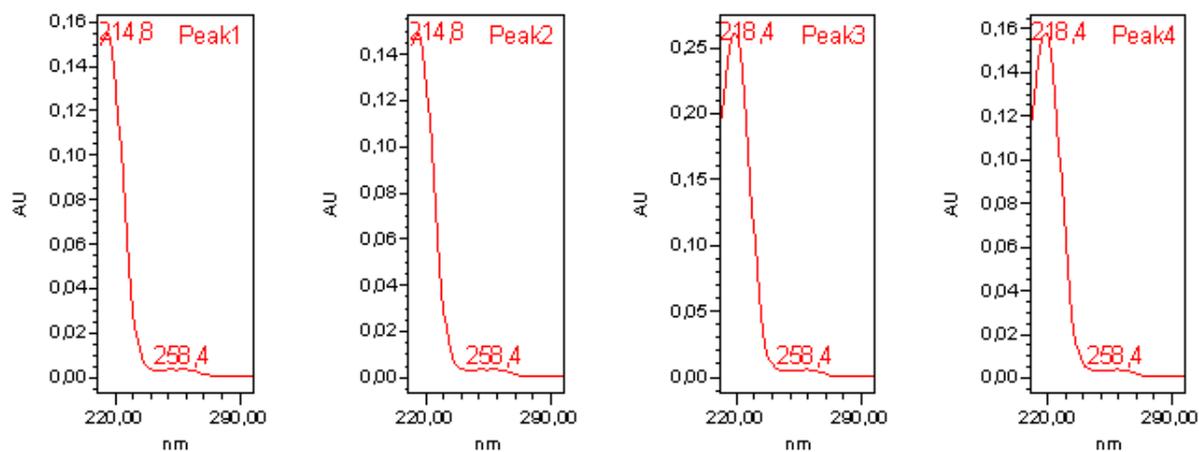
Racemic oxirane

Chromatogram

Room temperature



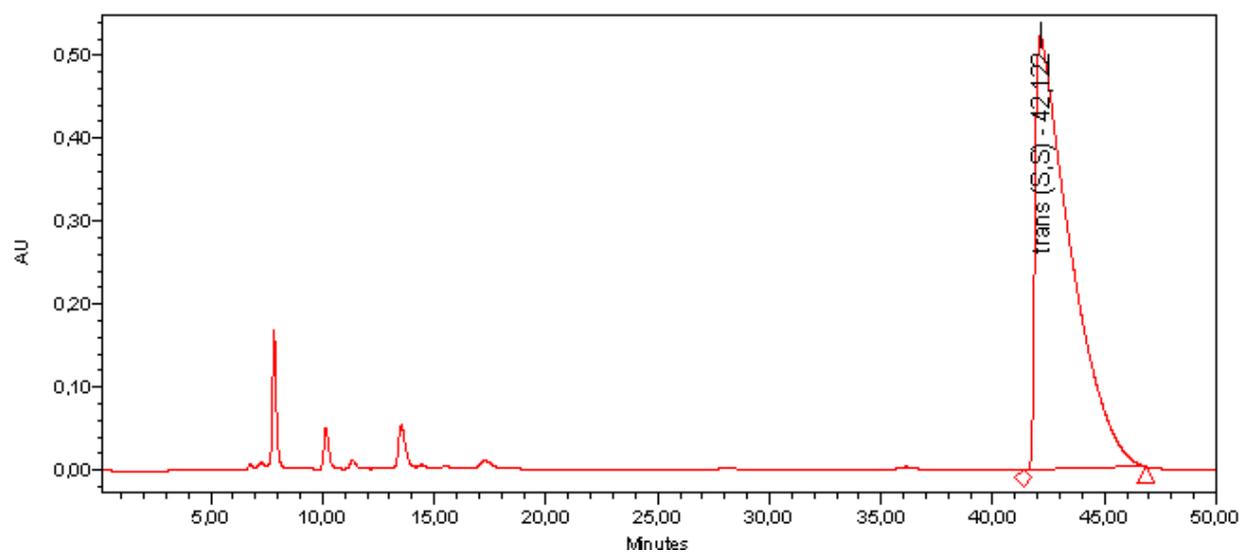
UV-VIS absorptions



Enantiopure oxirane 10a from the transformation of the oxirane generated by Sharpless epoxidation of trans-cinnamyl alcohol

Chromatogram

T = 20°C

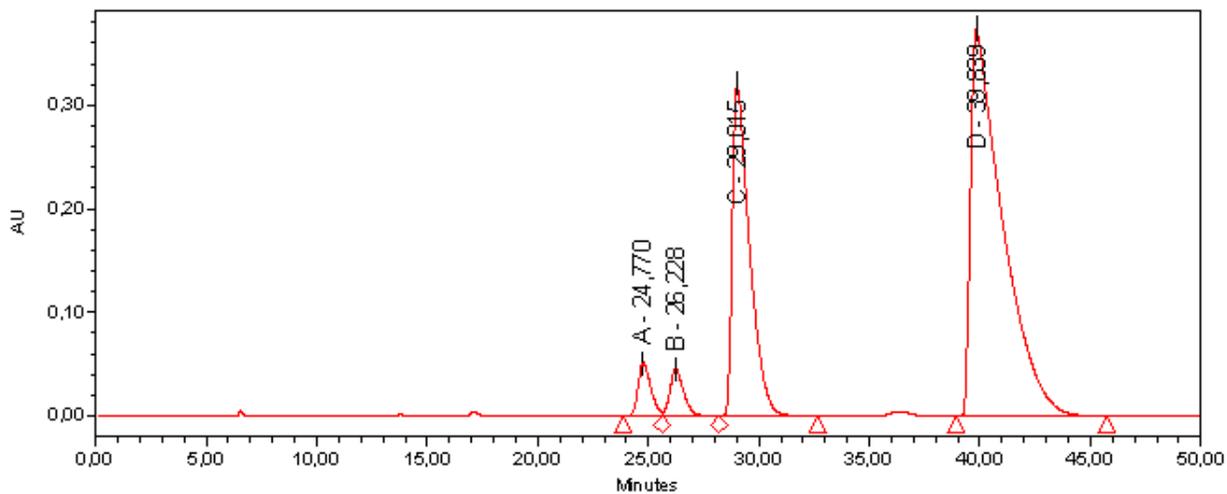


Enantioenriched oxirane from vinylidenation

Preparation : according to ref.¹

Chromatogram

T = 20°C

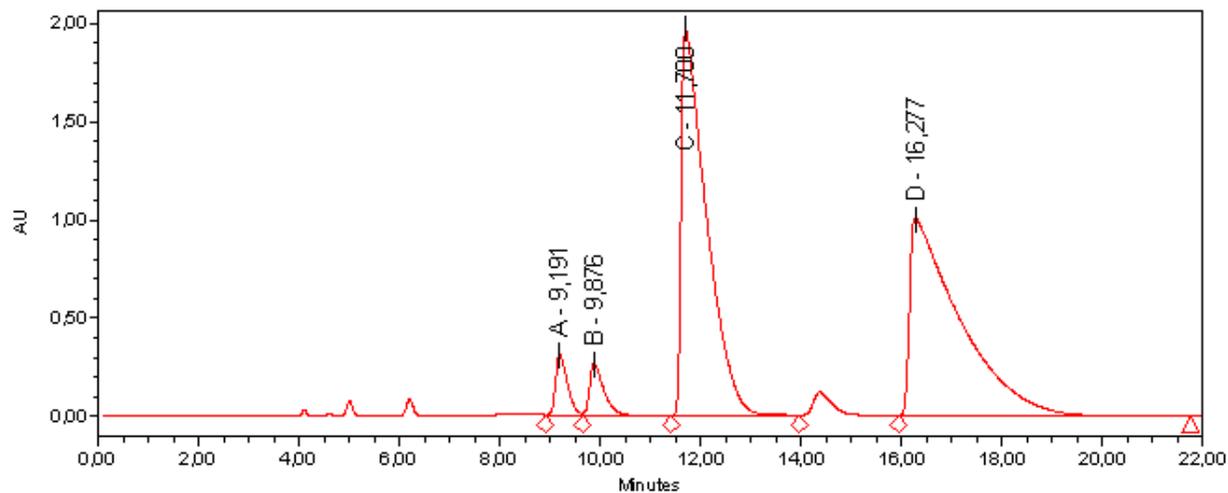


(2S,3S)-3-(1-Methylethenyl)-2-phenyloxirane 10c

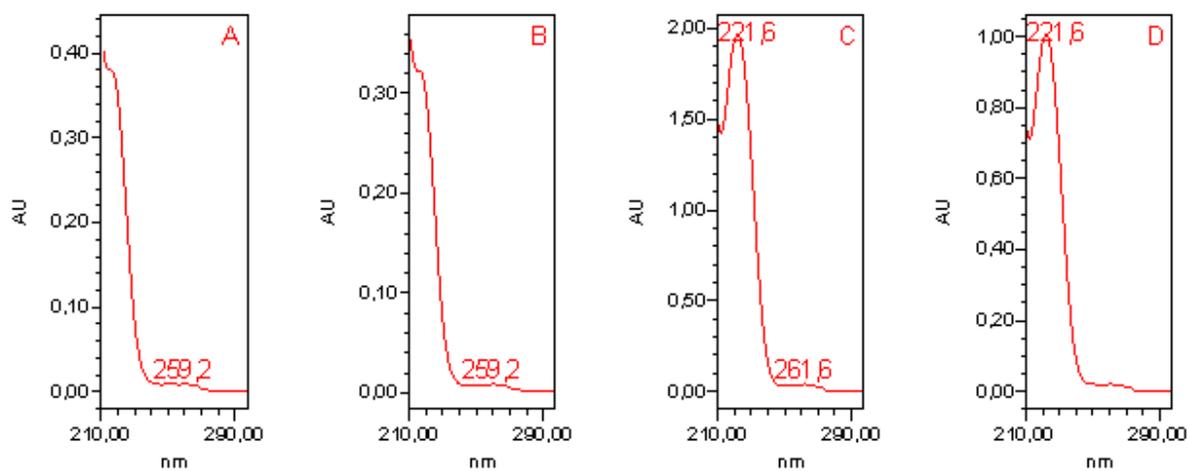
Racemic oxirane

Chromatogram

Room temperature



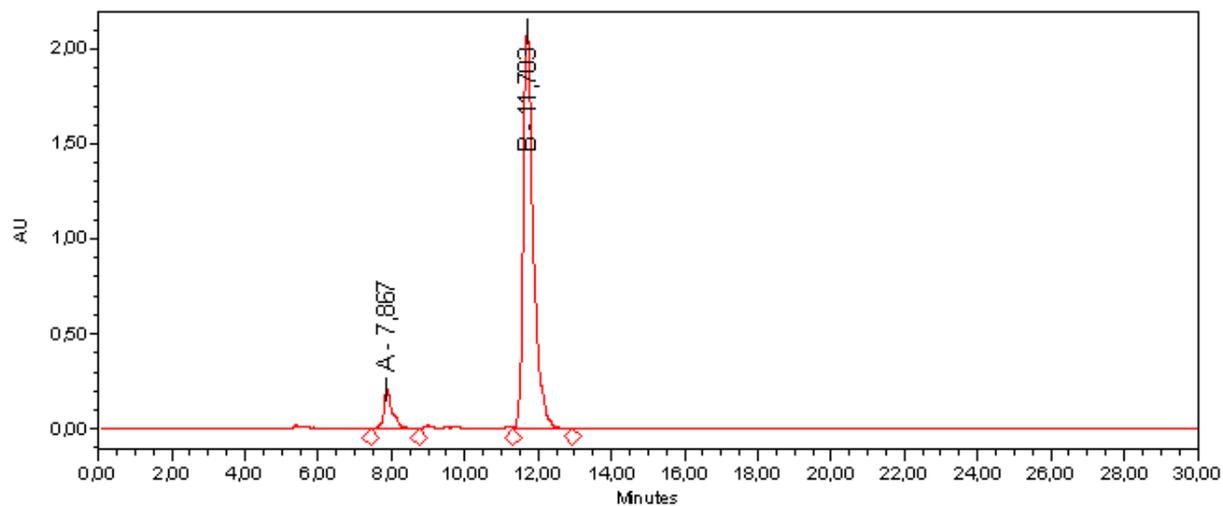
UV-VIS absorptions



Enantioenriched oxirane from vinylidenation

Chromatogram

Room temperature



Reference

- 1 Lindström, U. M., Somfai, P., *Synthesis* **1998**, 109-117.