Supporting Information

Chromatographic purification of 3en, 3ex.

The crude reaction mixture extract (1.4 g) was purified using flash liquid chromatography on a Teledyne CombiFlash Rf system equipped with a 2x24 g normal-phase RediSep Rf Gold silica column. The elution was done with an ACS grade hexanes/ethyl acetate mixture. The sample was dry packed using a minimal amount of Fisher 230-400 mesh normal-phase silica gel. The separation was done using the following parameters. Flow Rate -35 ml/min; Equilibration Volume -5.0 CV (Column Volume); Run length -20 CV; Fraction volume -7 mL; UV wavelength -254 nm & 280 nm. Gradient: 25% ethyl acetate over 2 CV; 25% - 33% over 4 CV; 33% - 66% ethyl acetate over 4 CV; 66% ethyl acetate over 4 CV; 100% ethyl acetate over 8 CV. Diastereomer *3ex* was eluted first in fractions 6-10 (480 mg) followed by *3en* fractions. 12-17 (900 mg).

Chromatographic purification of 4en, 4ex.

The crude reaction mixture extract (1.1 g) was purified using flash liquid chromatography on a Teledyne CombiFlash Rf system equipped with a 2x24 g normal-phase RediSep Rf Gold silica column. The elution was done with an ACS grade hexanes/ethyl acetate mixture. The sample was dry packed using a minimal amount of Fisher 230-400 mesh normal-phase silica gel. The separation was done using the following parameters: Flow Rate -35 ml/min; Equilibration Volume -5.0 CV (Column Volume); Run length -15 CV; Fraction volume -7 mL; UV wavelength -254 nm & 280 nm. Gradient: 33% ethyl acetate -33% over 2 CV; 33% -42% ethyl acetate over 4 CV; 42% -45% ethyl acetate over 3 CV; 100% ethyl acetate over 6 CV. Diastereomer *4ex* was eluted first in fractions 8-14 (78 mg) followed by *4en* fractions. 15-21 (1000 mg).







Figure S.3. APT of len









60









64













Figure S.27. Deconvoluted 1H NMR of 3ex









Figure S.35. ¹H NMR of *4en*





74 Oleg Sannikov *et al.*: Introducing Complex NMR Mixtures at the Undergraduate Level: Isomerization, Separation and Analysis of the Diels-Alder Adducts from the Reaction of Methylcyclopentadiene and Maleic Anhydride (Part II)











