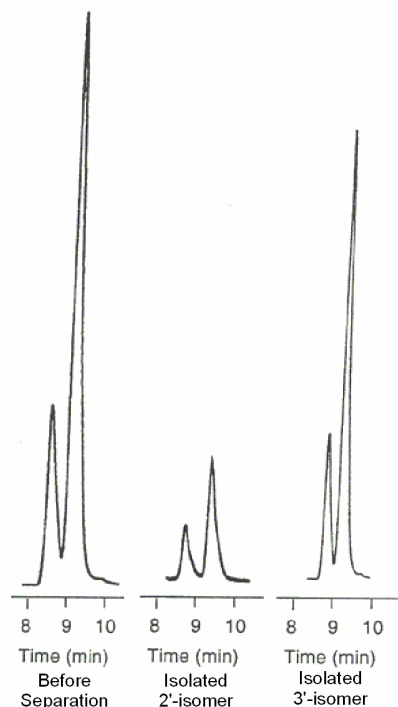


## Electronic Supplemental Information

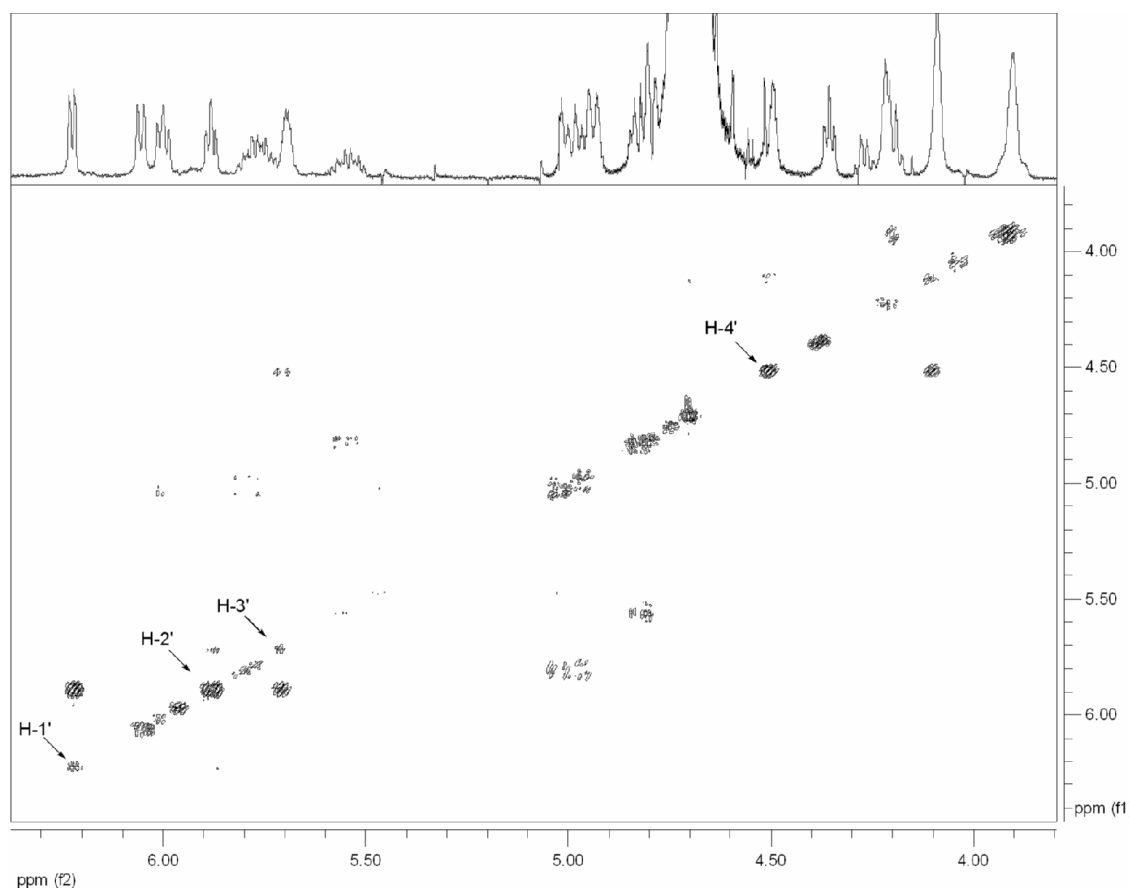
### Positional Assignment of Differentially Substituted Bisaminoacylated pdCpAs<sup>†</sup>

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**Figure S1.** Attempted separation of *N*-pentenoylated 2'- and 3'-*O*-valyl-pdCpAs (**2**) by HPLC.



**Figure S2.** gDQCOSY spectrum of 2'-*O*-[*N*-(4-pentenoyl)-(*S*)-alanyl]-3'-*O*-[*N*-(4-pentenoyl)-(*S*)-[1- $^{13}\text{C}$ ]valyl]-pdCpA (6.3 ppm to 3.8 ppm) with  $^1\text{H}$  spectrum shown on horizontal axis.