Supplementary Materials

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Marfey’s Analysis of Compounds 3 and 4

The stereochemistry of the proline residue was determined by the advanced Marfey’s method.

A solution of 2 or 3 (1.5 mg) in 6 N HCl (1 mL) was heated to 105 °C for 19 h. The solution was then evaporated to dryness and the residue redissolved in H2O (250 μL). A 50 μL portion of the acid hydrolysate solution was then placed in a 1 mL reaction vial and treated with a 1% solution of 1-fluoro-2,4-dinitrophenyl-5-L-alaninamide (L-FDAA) (200 μL) in acetone followed by 1.0 M NaHCO3 (40 μL). The reaction mixture was heated at 45 °C for 1 h, cooled to room temperature and then acidified with 2.0 M HCl (20 μL). In a similar fashion, the standard D- and L-Pro were derivatized with FDAA separately. The FDAA derivatives of the hydrolysates and standard amino acids were subjected to RP-HPLC analysis (Waters C18 column; 5 μm, 4.6 mm × 250 mm; 1.0 mL/min) at 30 °C using the following gradient program: solvent A, H2O + 0.1% TFA; solvent B, MeCN; linear gradient: 0 min, 25% B, 40 min, 60% B, 30 min, 100% B; UV detection at 340 nm. The retention times for the FDAA derivatives of hydrolysates of 2 or 3 were 14.5 min; standard L-Pro, D-Pro, and were 14.5 and 19.7 min (Figure S19), respectively.

![Figure S19. HPLC profile of Marfey’s Analysis of Compounds 3 and 4.](image-url)