Supporting information Available

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General Experimental Methods.

Melting points were determined using a hot-stage melting point apparatus and are uncorrected. Infrared spectra were recorded on a FT-IR spectrophotometer as potassium bromide disks of solids (KBr) or as thin films of liquids (neat) between sodium chloride plates. Nuclear magnetic resonance spectra (¹H, ³¹P and ¹³C NMR) were recorded on either 300 or 400 MHz spectrometers. ¹H and ¹³C NMR assignments were confirmed using standard 2D COSY, NOESY, HSQC, and HMBC experiments. Low resolution electrospray ionisation (ESI) were recorded in the positive mode (ESI⁺) on a QMS-quadrupole mass spectrometer. Accurate mass measurements were obtained at high resolution with a FTMS and a 4.7T superconducting magnet. The instrument was externally calibrated with FC5311. Analytical thin-layer chromatography (TLC) was performed on plastic slides coated with silica gel (Polygram SIL g/uv254). Flash chromatography was performed using Merck silica gel 60 (Merck no. 9385), 0.063-0.200 mm (230-400 mesh). Solvents were purified according to standard procedures. Chloroform used for optical rotations was of analytical purity. Degassed methanol and benzene were used in all hydrogenation reactions. Degassed dichloromethane was used in metathesis reactions. Deuterated chloroform was used as supplied. Deuterated dichloromethane was degassed by three freeze-pump-thaw cycles. Grubbs' catalyst refers to bis(tricyclohexylphosphine)benzylidene ruthenium dichloride.¹⁸ Second generation Grubbs' catalyst refers to tricyclohexylphosphine[1,3-bis(2,4,6-trimethylphenyl)-4,5-dihydroimidazol-2ylidene][benzylidene]ruthenium dichloride.¹⁹ Wilkinson's catalyst refers to chlorotris(triphenylphosphine)rhodium (I).²³ Rh(I)-(S,S)-Et-DuPHOS refers to (+)-1,2-bis[(2S,5S)-2,5tetrafluoroborate.²⁰ diethylphospholano]benzene(1,5-cyclooctadiene)rhodium(I) Rh(I)-(*R*,*R*)-Et-DuPHOS refers to (-)-1,2-bis[(2R,5R)-2,5-diethylphospholano]benzene(1,5-cyclooctadiene)rhodium(I)tetrafluoroborate.²⁰ All ruthenium-catalysed metathesis reactions were performed using standard Schlenk techniques under an atmosphere of nitrogen or in an argon filled drybox. Solvents were dried and degassed using standard procedures. In all rhodium-phosphine hydrogenations, high purity (<10 ppm of

oxygen) hydrogen and argon were used and purified by passage through a series of traps to remove water, oxygen and hydrocarbons. Ethylene and *cis*-2-butene were used as supplied. The enantiomeric excess of hydrogenation products was determined *via* analytical gas chromatography (GC) using a chiral column Model C-024 (column: 0.25 mm x 50 cm, 50CP2/XE60-SVALSAPEA) using helium as the carrier gas. Optical rotations were measured with a polarimeter (in a cell length of 1 dm) at a wavelength of 589 nm (sodium D line) at a temperature of 22°C.

(2*R*)-Methyl 2*N*-acetylaminopent-4-enoate (*ent*-4).¹⁶ (2*Z*)-Methyl 2*N*-acetylaminopenta-2,4dienoate 3 (40.0 mg, 0.24 mmol), benzene (5 ml), [(COD)Rh(*R*,*R*)-Et-DuPHOS]OTf (3 mg), 30 psi H₂, 3 h, 88% yield, 96% ee (2*R*-4), $t_{\rm R} = 16.4$ min (*R*) (GC chiral column Model C-024, 100°C for 1 min, 5°C min⁻¹ to 280°C for 9 min). $[\alpha]_{\rm D}^{22}$ -43.0° (c = 0.47, CHCl₃). Spectral data were consistent with literature data.¹⁶

(2*S*)-Methyl 2*N*-benzoylaminopent-4-enoate (6).³² (2*Z*)-Methyl 2*N*-benzoylaminopenta-2,4-dienoate (100.0 mg, 0.43 mmol), benzene (8 ml), [(COD)Rh(*S*,*S*)-Et-DuPHOS]OTf (3 mg), 30 psi H₂, 3 h, 99% yield, 100% ee (2*S*-6), $t_{\rm R} = 27.0$ min (GC chiral column Model C-024, 180°C for 1 min, 2°C min⁻¹ to 210°C for 20 min). [α]²²_D +49.3° (c = 1.12, CHCl₃). Spectral data were consistent with literature data.³²

(2*R*)-Methyl 2*N*-benzoylaminopent-4-enoate (*ent-6*).³² (2*Z*)-Methyl 2*N*-benzoylaminopenta-2,4dienoate (100.0 mg, 0.43 mmol), benzene (8 ml), [(COD)Rh(*R*,*R*)-Et-DuPHOS]OTf (3 mg), 30 psi H₂, 3 h, 93% yield, 100% ee (2*R*-6), $t_{\rm R}$ = 26.4 min (GC chiral column Model C-024, 180°C for 1 min, 2°C min⁻¹ to 210°C for 20 min). [α]²²_D -49.7° (c = 0.64, CHCl₃). Spectral data were consistent with literature data.³²

(2*S*)-Methyl 2*N*-acetylamino-5-phenylpent-4-enoate (13).³⁰ (2*Z*)-Methyl 2*N*-acetylamino-5-phenylpenta-2,4-dienoate 12 (28.0 mg, 0.11 mmol), methanol (5 ml), [(COD)Rh(*S*,*S*)-Et-DuPHOS]OTf (1 mg), 75 psi H₂, 2 h, 97% yield, 99% ee (2*S*-13). $[\alpha]_{D}^{22}$ +90.0° (c = 0.64, CHCl₃). Spectral data were consistent with literature data.³⁰

(2*R*)-Methyl 2*N*-acetylamino-5-phenylpent-4-enoate (*ent*-13).³⁰ (2*Z*)-Methyl 2*N*-acetylamino-5-phenylpenta-2,4-dienoate 12 (27.4 mg, 0.11 mmol), methanol (5 ml), [(COD)Rh(*R*,*R*)-Et-DuPHOS]OTf (1 mg), 75 psi H₂, 2 h, 92% yield, 99% ee (2*R*-13). $[\alpha]_{D}^{22}$ –89.8° (c = 1.03, CHCl₃). Spectral data were consistent with literature data.³⁰

(2S)-Methyl 2*N*-acetylamino-5-methylhex-4-enoate (15). (2*Z*)-Methyl 2*N*-acetylamino-5methylhexa-2,4-dienoate 16 (74.0 mg, 0.38 mmol), methanol (5 ml), [(COD)Rh(*S*,*S*)-Et-DuPHOS]OTf (2 mg), 75 psi H₂, 2 h, 98% yield, 100% ee (2*S*-15), $t_{\rm R}$ = 24.2 min (GC chiral column Model C-024, 100°C for 1 min, 5°C min⁻¹ to 210°C for 7 min). [α]²²_D +58.2° (c = 0.79, CHCl₃), m.p. 46-48°C. $\nu_{\rm max}$ (neat): 3288m, 2955w, 1746s, 1660s, 1538m, 1436m, 1377m, 1274w, 1210w, 1126w, 1030w, 736w cm⁻¹. ¹H NMR (300 MHz, CDCl₃): δ 1.59 (s, 3H, H6a), 1.69 (d, *J* 0.9 Hz, 3H, H6b), 2.00 (s, 3H, CH₃CO), 2.39-2.60 (m, 2H, H3), 3.72 (s, 3H, OCH₃), 4.63 (dt, *J* 7.9, 5.6 Hz, 1H, H2), 4.99 (t, *J* 7.5 Hz, 1H, H4), 6.02 (bs, 1H, NH). ¹³C NMR. (100 MHz, CDCl₃): δ 18.0, 26.0, ((CH₃)₂), 23.3 (CH₃CO), 30.8 (C3), 52.2, 52.4 (C2, OCH₃), 117.6 (C4), 136.6 (C5), 169.8, 172.8 (C1, CONH). HRMS (ESI⁺, MeOH): *m/z* calcd for C₁₀H₁₇NO₃Na [(M+Na)⁺] 222.1106, found 222.1105.

(2*R*)-Methyl 2*N*-acetylamino-5-methylhex-4-enoate (*ent*-15). (2*Z*)-Methyl 2*N*-acetylamino-5-methylhexa-2,4-dienoate 16 (24.5 mg, 0.12 mmol), methanol (5 ml), [(COD)Rh(*R*,*R*)-Et-DuPHOS]OTF (1 mg), 75 psi H₂, 2 h, 100% yield, 100% ee (2*R*-15), $t_R = 23.9$ min (GC chiral column Model C-024, 100°C for 1 min, 5°C min⁻¹ to 210°C for 7 min). $[\alpha]_{D}^{22}$ -58.3° (c = 0.53, CHCl₃). Spectral data were consistent with that previously obtained.

(2S,7S)-Dimethyl 2,7-N,N'-dibenzoylamino-oct-4-enedioate (7). (2S)-Methyl 2N-

benzoylaminopent-4-enoate 6 (49.0 mg, 0.21 mmol), dichloromethane (4 ml), 2nd generation Grubbs' S5

catalyst (18.0 mg, 0.02 mmol, 10 mol%), 12 h, 50°C, 100% conversion. Purification by flash chromatography (SiO₂, dichloromethane : light petroleum : ethyl acetate, 1:1:1) gave pure dimer **7** (32.8 mg, 36%) as a pale brown solid, m.p. 140-142°C. $t_{\rm R}$ (*E/Z*) = 13.5, 13.9 min (GC column 30QC5/BPX5), 150°C for 1 min, 10°C min⁻¹ to 280°C for 6 min). $[\alpha]^{22}_{\rm D}$ +56.4° (c = 0.27, CHCl₃). $v_{\rm max}$ (neat): 3322bm, 2953m, 2358w, 1742s, 1644s, 1603w, 1580w, 1538m, 1488m, 1436m, 1267w, 1218m, 1027w, 973w, 802w, 736m cm⁻¹. ¹H NMR (300 MHz, CDCl₃): δ 2.57-2.69 (m, 4H, H3,6), 3.67 (s, 6H, OCH₃), 4.85-4.98 (m, 2H, H2,7), 5.49 (t, *J* 4.1 Hz, 2H, H4,5), 6.86 (bd, *J* 7.4 Hz, 2H, NH), 7.40-7.44 (m, 4H, H3',5'), 7.48-7.52 (m, 2H, H4'), 7.81-7.83 (m, 4H, H2',6'). ¹³C NMR (100 MHz, CDCl₃): δ 35.2 (C3,6), 52.5 (C2,7), 52.6 (OCH₃), 127.2 (C2',6'), 128.7 (C3',5'), 128.8 (C4,5), 131.9 (C4'), 133.9 (C1'), 167.1, 172.4 (C1,8, CONH). HRMS (ESI⁺, MeOH): *m/z* calcd for C₂₄H₂₆N₂O₆Na [(M+Na)⁺] 461.1689, found 461.1695.

Attempted dimerisation of (2S)-methyl 2N-acetylamino-5-phenylpent-4-enoate (13). *Trial 1*: (2S)-Methyl 2N-acetylamino-5-phenylpent-4-enoate 13 (59.3 mg, 0.24 mmol), dichloromethane (10 ml), Grubbs' catalyst (19.8 mg, 0.02 mmol, 10 mol%), 13 h, 50°C, 0% conversion of 13 into dimer 5. *Trial* 2: (2S)-Methyl 2N-acetylamino-5-phenylpent-4-enoate 13 (59.3 mg, 0.24 mmol), dichloromethane (7 ml), 2nd generation Grubbs' catalyst (10.2 mg, 0.01 mmol, 5 mol%), 20 h, 50°C, 33% conversion of 13 into dimer 5.

Dimerisation of (2S)-Methyl 2N-acetylaminohex-4-enoate (17). (2S)-Methyl 2N-acetylaminohex-4enoate **17** (17.0 mg, 0.09 mmol), dichloromethane (4 ml), 2nd generation Grubbs' catalyst (4.2 mg, 0.005 mmol, 5 mol%), 17 h, 50°C, 100% conversion into dimer **5**. Spectroscopic data were in agreement with that previously obtained. Cross metathesis of (2*S*)-methyl 2*N*-acetylaminopent-4-enoate (4) in the presence of (2*S*)-methyl 2*N*-acetylaminopenta-2,4-dienoate (3). (2*S*)-Methyl 2*N*-acetylamino-pent-4-enoate 4 (34.0 mg, 0.20 mmol), (2*Z*)-methyl 2*N*-acetylamino-penta-2,4-dienoate 3 (33.6 mg, 0.20 mmol), dichloromethane (4 ml), Grubbs' catalyst (16.3 mg, 0.02 mmol, 10 mol%), 18 h, 50°C. The ¹H NMR spectrum displayed peaks characteristic of the starting allylglycine derivative 4 and dienamide 3 but no peaks characteristic of expected dimer 5. The mass spectrum displayed peaks attributed to allylglycine derivative 4 and tricyclohexylphosphine-dienamide conjugate addition adduct. Mass Spectrum (ESI⁺, DCM/MeOH): m/z 194.1 C₈H₁₃NO₃Na [(M+Na)⁺], 4. m/z 450.4 C₂₆H₄₅NO₃P⁺ [M⁺], tricyclohexylphosphine-dienamide adduct.

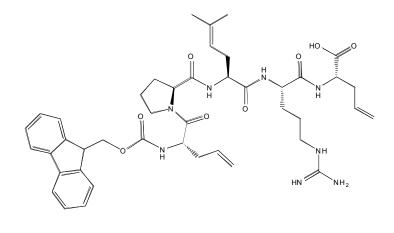
Cross metathesis of (2S)-methyl 2N-acetylaminopent-4-enoate (4) in the presence of (2S)-methyl 2N-acetylamino-5-phenylpenta-2,4-dienoate (12). (2S)-Methyl 2N-acetylaminopent-4-enoate 4 (18.1 mg, 0.11 mmol), (2Z)-methyl 2N-acetylamino-5-phenylpenta-2,4-dienoate 12 (26.1 mg, 0.11 mmol), dichloromethane (4 ml), Grubbs' catalyst (8.7 mg, 0.01 mmol, 10 mol%), 18 h, 50°C, 28% conversion of 4 into dimer 5. Dienamide 12 did not react under these conditions.

Cross metathesis of (2S)-Methyl 2N-acetylaminopent-4-enoate (4) in the presence of (2S)-Methyl 2N-acetylamino-5-methylhex-4-enoate (15). (2S)-Methyl 2N-acetylaminopent-4-enoate 4 (12.7 mg, 0.07 mmol), (2S)-methyl 2N-acetylamino-5-methylhex-4-enoate 15 (12.0 mg, 0.07 mmol), dichloromethane (4 ml), Grubbs' catalyst (11.5 mg, 0.01 mmol, 20 mol%), 18 h, 50°C, 100% conversion of 4 into dimer 5. 15 did not react under these conditions.

Wilkinson's hydrogenation of Dimer (7). (2*S*,7*S*)-Dimethyl 2,7-*N*,*N*'-dibenzoylaminooctanedioate (18). (2*S*,7*S*)-Dimethyl 2,7-*N*,*N*'-dibenzoylamino-octa-4-enedioate 7 (20.0 mg, 0.05 mmol), benzene (5 ml), Wilkinson's catalyst (2 mg), 50 psi H₂, 4 h, 100% of **18** as a brown oil. $t_{\rm R}$ = 17.2 min (GC: Column 30QC5/BPX5, 150°C for 1 min, 10°C min⁻¹ to 280°C for 6 min). $v_{\rm max}$ (neat): 3055m, 2986w, 2955w, 1741s, 1662s, 1603w, 1580w, 1518m, 1486m, 1438s, 1359w, 1286s, 1182m, 1120m, 1028w, 896m cm⁻¹. ¹H NMR (300 MHz, CDCl₃): δ 1.35-1.53 (m, 4H, H4,5), 1.80-2.02 (m, 4H, H3,6), 3.78 (s, 3H, OCH₃), 4.82 (dt, *J* 7.4, 5.3 Hz, 1H, H2,7), 6.73 (bd, *J* 7.7 Hz, 2H, NH), 7.40-7.49 (m, 6H, H3',4',5'), 7.78-7.82 (m, 4H, H3',5'). ¹³C NMR (100 MHz, CDCl₃): δ 24.9 (C4,5), 32.6 (C3,6), 52.5, 52.7 (C2, OCH₃), 127.2 (C2',6'), 128.6 (C3',5'), 131.9 (C4'), 134.1 (C1'), 167.2, 173.2 (C1,8, CONH). HRMS (ESI⁺, MeOH): m/z calcd for C₂₄H₂₈N₂O₆Na [(M+Na)⁺] 463.1845, found: 463.1842

Wilkinson's hydrogenation of (2*S*)-methyl 2*N*-acetylamino-5-phenylpenta-2,4-dienoate (12). (2*S*)-Methyl 2*N*-acetylamino-5-phenylpenta-2,4-dienoate 12 (11.5 mg, 0.05 mmol), benzene (5 ml), Wilkinson's catalyst (1 mg), 50 psi H₂, 4 h, 99% yield of a 1:4 mixture of 13:14 as a brown oil. This mixture was subjected to the hydrogenation conditions previously described which led to 100% conversion into 14.³⁴. $t_{\rm R}$ = 10.8 min (GC column 30QC5/BPX5, 150°C for 1 min, 10°C min⁻¹ to 280°C for 6 min). $v_{\rm max}$ (neat): 3054m, 2956m, 1736m, 1676m, 1509w, 1438m, 1372w, 1265s, 1174w, 1120m, 1028w, 738s cm⁻¹. ¹H NMR (300 MHz, CDCl₃): δ 1.53-1.65 (m, 4H, H3,4), 1.94 (s, 3H, CH₃CO), 2.52-2.59 (m, 2H, H5), 3.65 (s, 3H, OCH₃), 4.54-4.63 (m, 1H, H2), 5.90 (bd, *J* 7.2 Hz, 1H, NH), 7.07-7.29 (m, 5H, AromCH). ¹³C NMR (75 MHz, CDCl₃): δ 23.3 (*C*H₃CO), 27.2 (C4), 32.3 (C3), 35.5 (C5), 52.1, 52.5 (C2, OCH₃), 126.1, 128.5, 132.2 (Arom CH), 141.7, (Arom C), 169.9, 173.2 (C1, CONH). Mass Spectrum (ESI⁺, MeOH): *m/z* 272.2 C₁₄H₁₉NO₃Na [(M+Na)⁺].

Linear Pentapeptide: Fmoc-Hag-Pro-Pre-Arg-Hag-OH



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The procedure outlined in the General Section was used to attach the first amino acid, Fmoc-Hag-OH, to Wang resin. Quantities of the resin and coupling reagents are presented in Table 1. The first coupling reaction was shaken for 14 h.

Table 1 Ouantities	of Reagents used	in the Synthesis of Fm	10c-Hag-Pro-Pro	e-Arg-Hag-OH

Reagent	Mass (mg) or Volume (µl)	Mole (mmol)
Wang Resin	212 mg	0.19
Fmoc-L-Hag-OH	195 mg	0.58
DIC	90.6µ1	0.58
DMAP	7.1 mg	0.06

The procedure outlined in the General Section was used for subsequent coupling reactions in the synthesis of pentapeptide Fmoc-Hag-Pro-Pre-Arg-Hag-OH. Quantities of the coupling reagents HATU and NMM are tabulated below (Table 2) and remained constant throughout the synthesis. The quantities of successive amino acids and their reaction durations are detailed in Table 3.

Coupling Reagent	Mass (mg) or Volume (ml)	Mole (mmol)
HATU	147 mg	0.39
NMM	128 µl	1.16

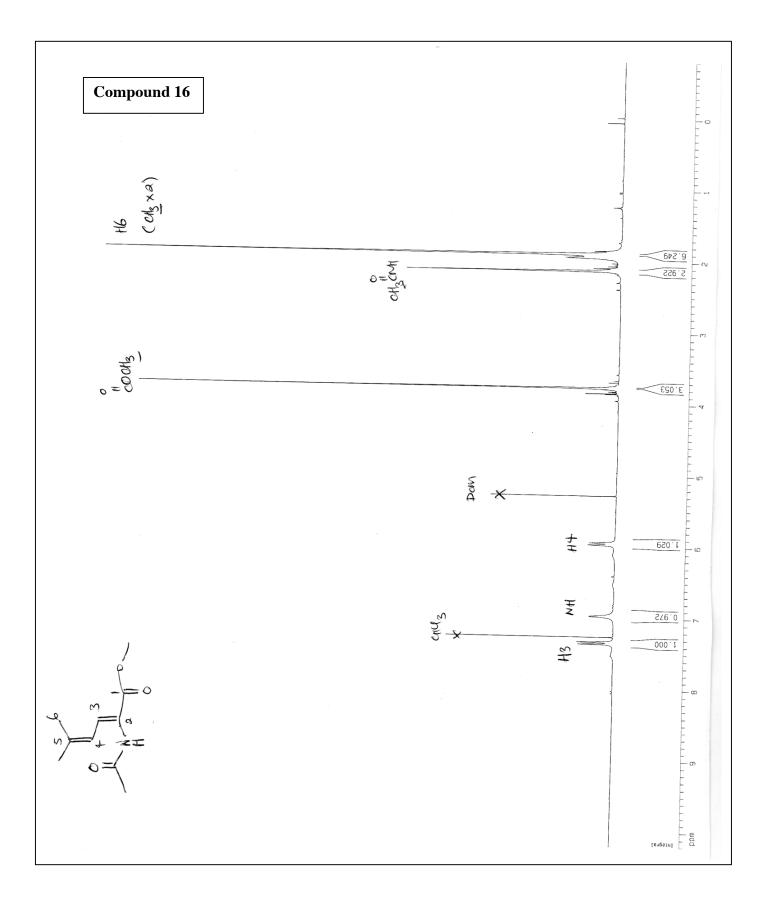
Table 2 Quantities of Coupling Reagents used in the Synthesis of Fmoc-Hag-Pro-Pre-Arg-Hag-OH

Table 3 Quantities of Amino Acids used in the Synthesis of Fmoc-Hag-Pro-Pre-Arg-Hag-OH

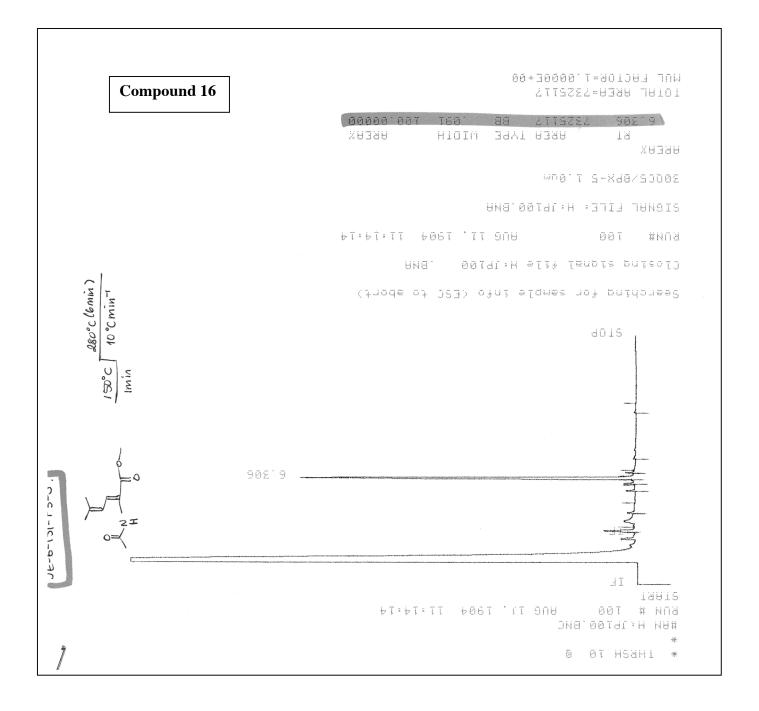
Amino Acid	Mass (mg)	Mole (mmol)	Reaction Time (h)*
Fmoc-L-Arg(Pbf)-OH	376	0.58	2
Fmoc-L-Pre-OH	211	0.58	3
Fmoc-L-Pro-OH	196	0.58	6
Fmoc-L-Hag-OH	195	0.58	2

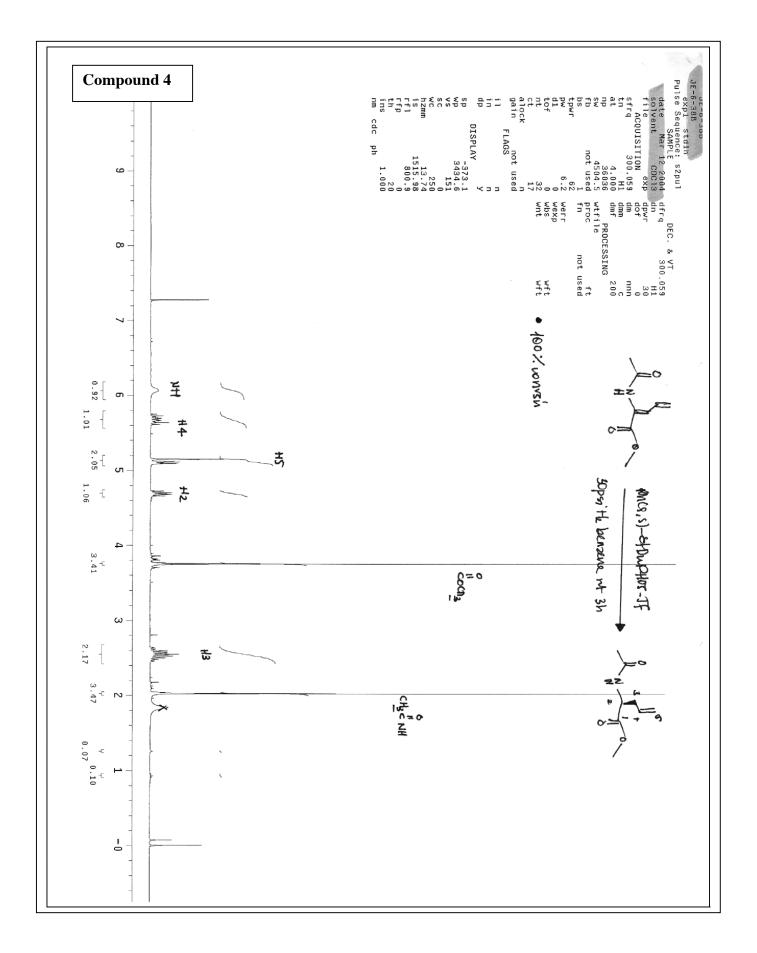
* *Note:* Reaction times have not been optimised.

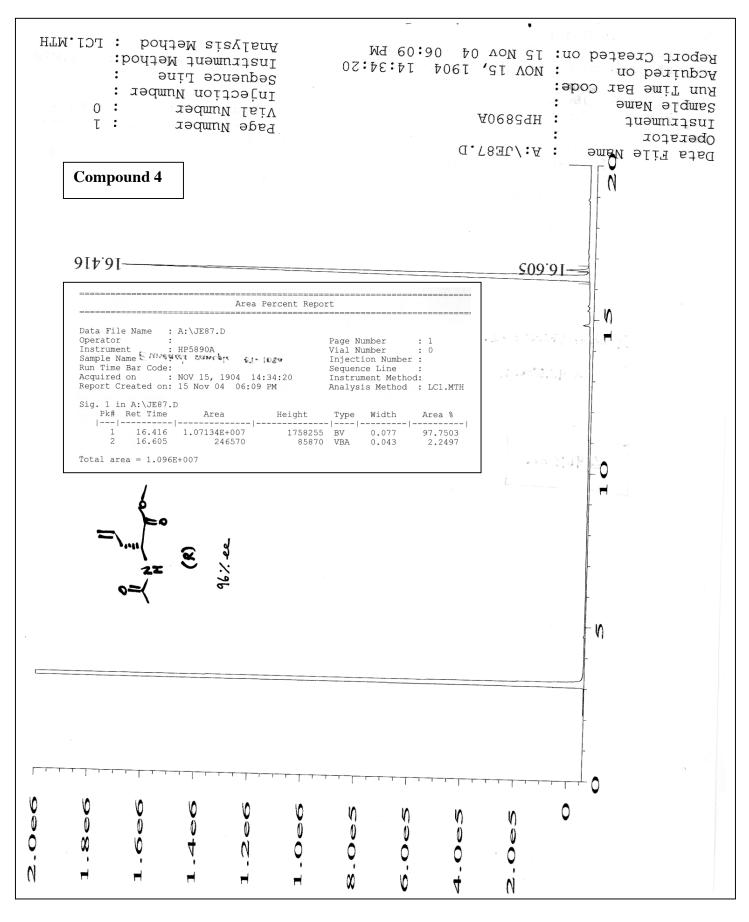
After the final amino acid coupling, a small aliquot of peptidyl-resin was subjected to the TFA-mediated cleavage procedure described in the General Section. Mass spectral analysis of the isolated residue confirmed formation of the linear pentapeptide (**19**). Mass spectrum (ESI⁺, MeCN/H₂O): m/z 813.6 (M+H)⁺, C₄₃H₅₇N₈O₈.requires 813.4; m/z 831.5 (M+H₂O+H)⁺, C₄₃H₅₉N₈O₉ requires 831.4; m/z 927.6 (M+TFA+H)⁺, C₄₅H₅₈ F₃N₈O₁₀ requires 927.4.



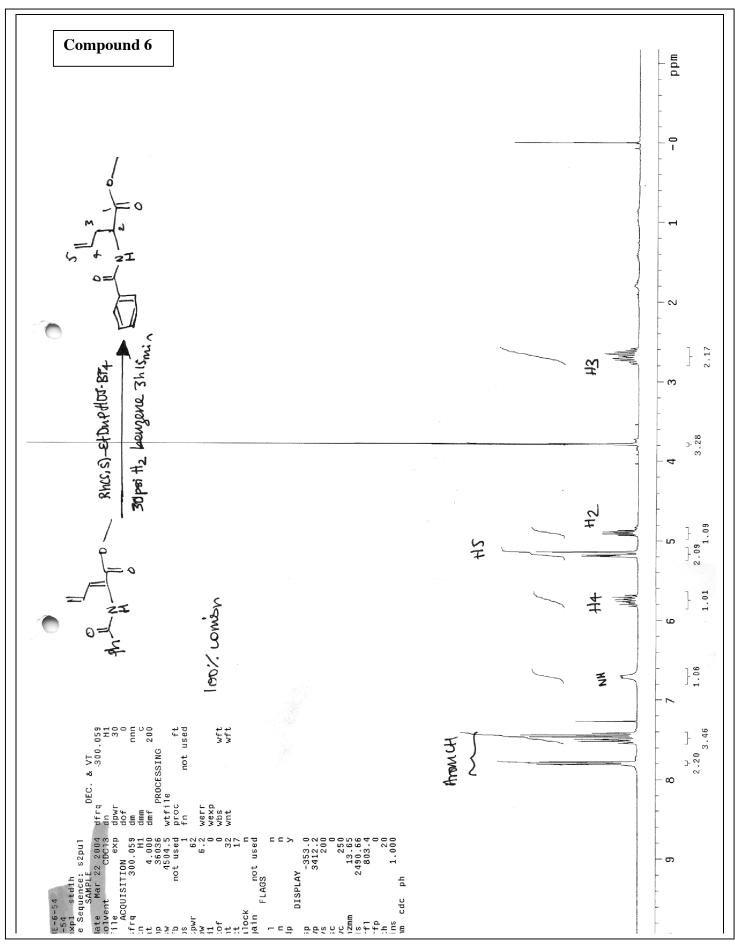
S12

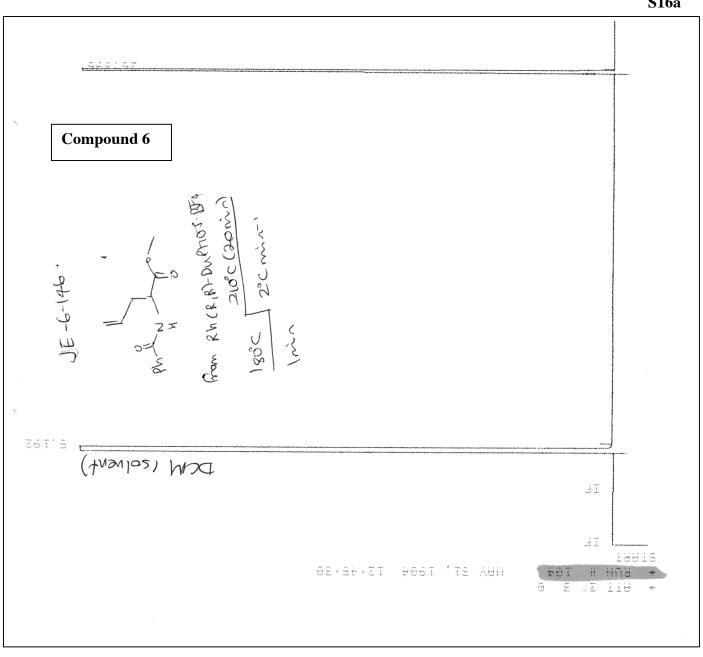


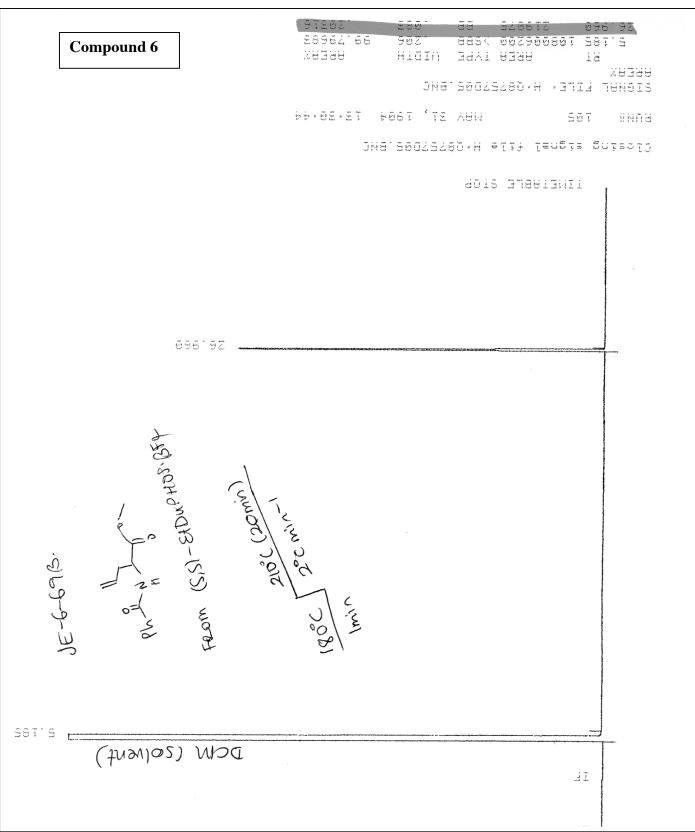




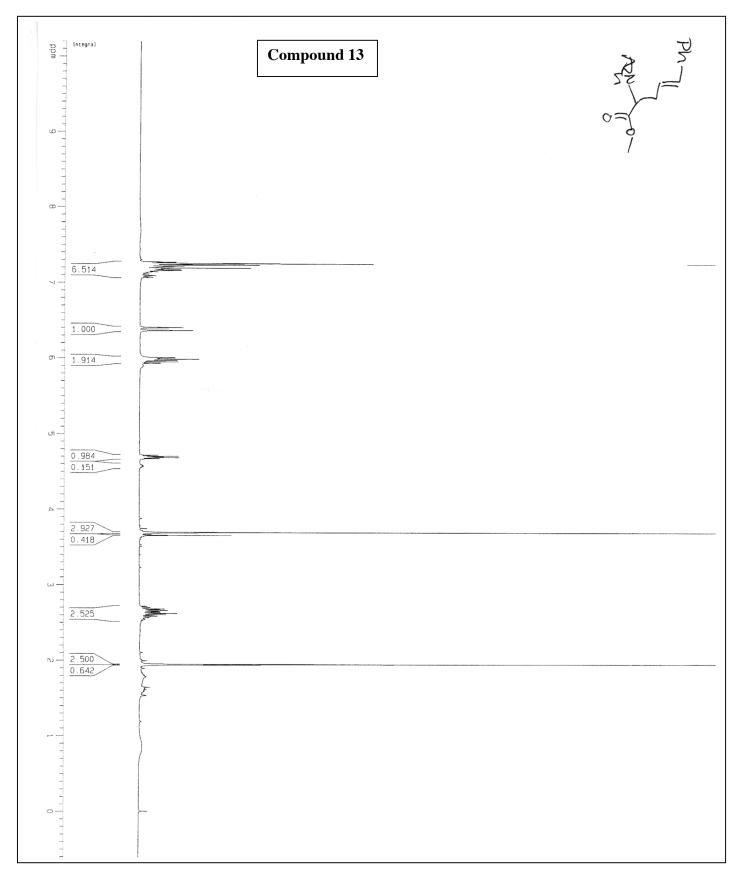
Report Created on: 15 Nov 04 06:13 PM HTM.IJJ : bodjeM sizylsnA Acquired on : NOV 15, 1904 16:58:33 :bodtaM tnamurtani Run Time Bar Code: : əuid əpres sample Name : Injection Number : 0: redmuN leiv Justrument A06829H : Page Number : Operator т: : A:\JE89.D Data File Name Area Percent Report 0 Data File Name : A:\JE89.D Operator Page Number : 1 Ν Instrument : HP5890A Vial Number : 0 Injection Number : Sample Name Run Time Bar Code: Sequence Line Instrument Method: Analysis Method : LC1.MTH Acquired on : NOV 15, 1904 16:58:33 Report Created on: 15 Nov 04 06:13 PM Sig. 1 in A:\JE89.D Pk# Ret Time Area Height Type Width Area % 22578 6514 BB 0.051 1623691 536841 BBA 0.047 |----|------| 0 1 16.213 1.3715 N 2 16.533 98.6285 Total area = 1646270££\$.01 ---16.213 0 **Compound 4** -0 17% ee S UIUII 0 0 0 5 5 0 0 5 5 5 0 0 0 0 0 0 0 0 Ŏ 0 0 1 1 00 0 4 N M Ν N

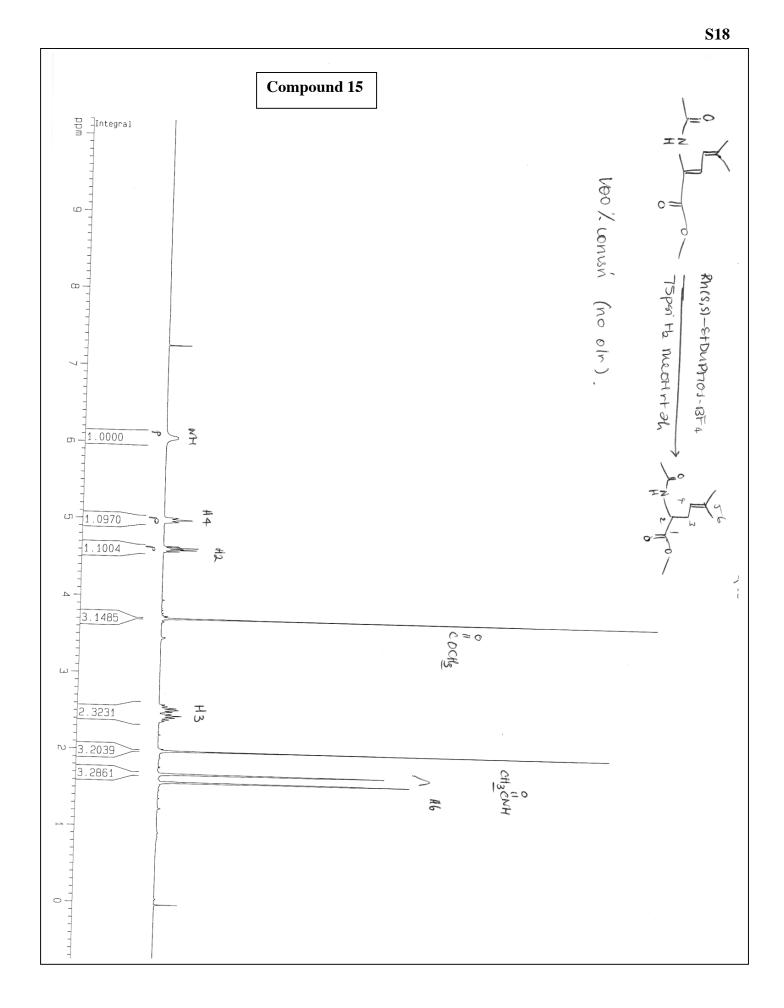




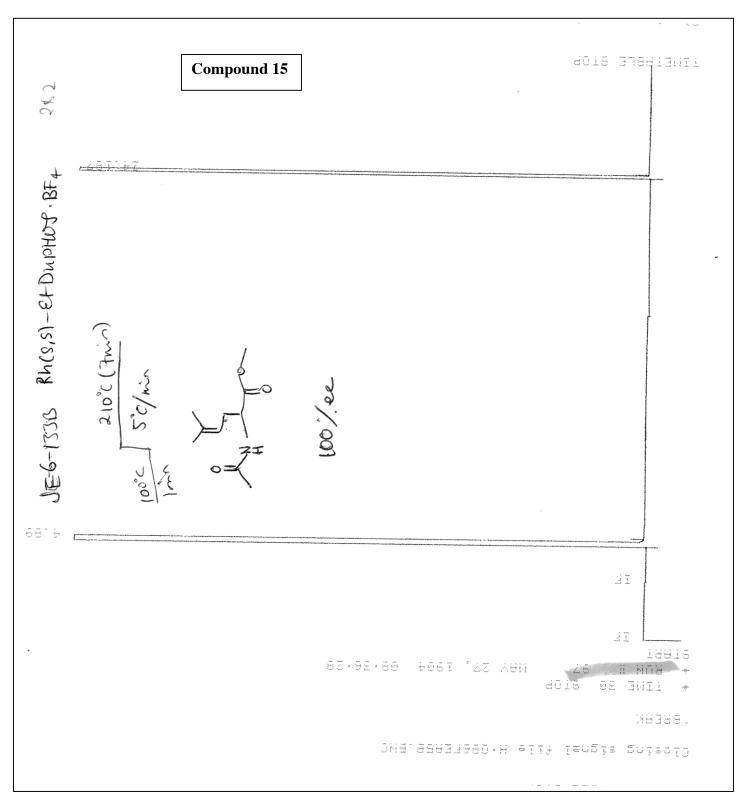


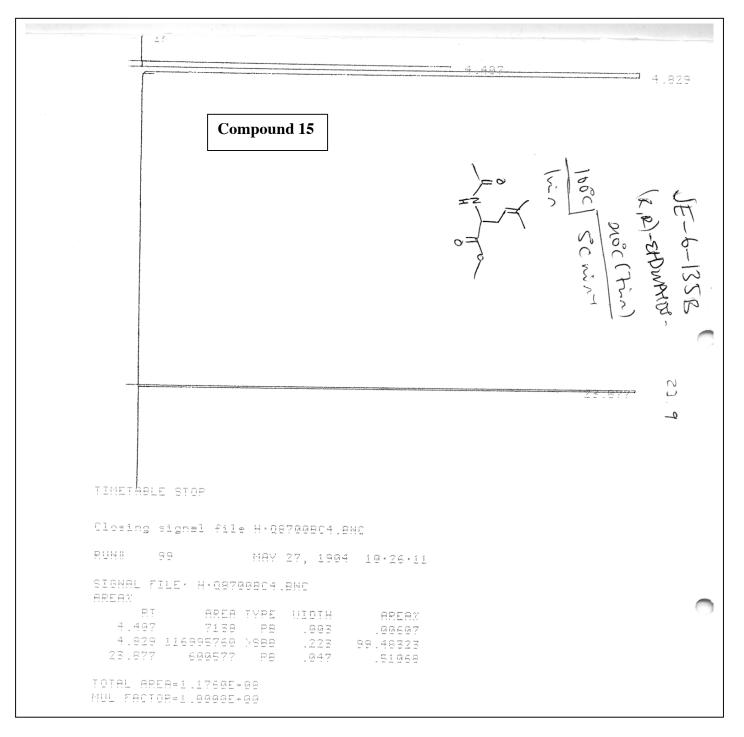
S16b

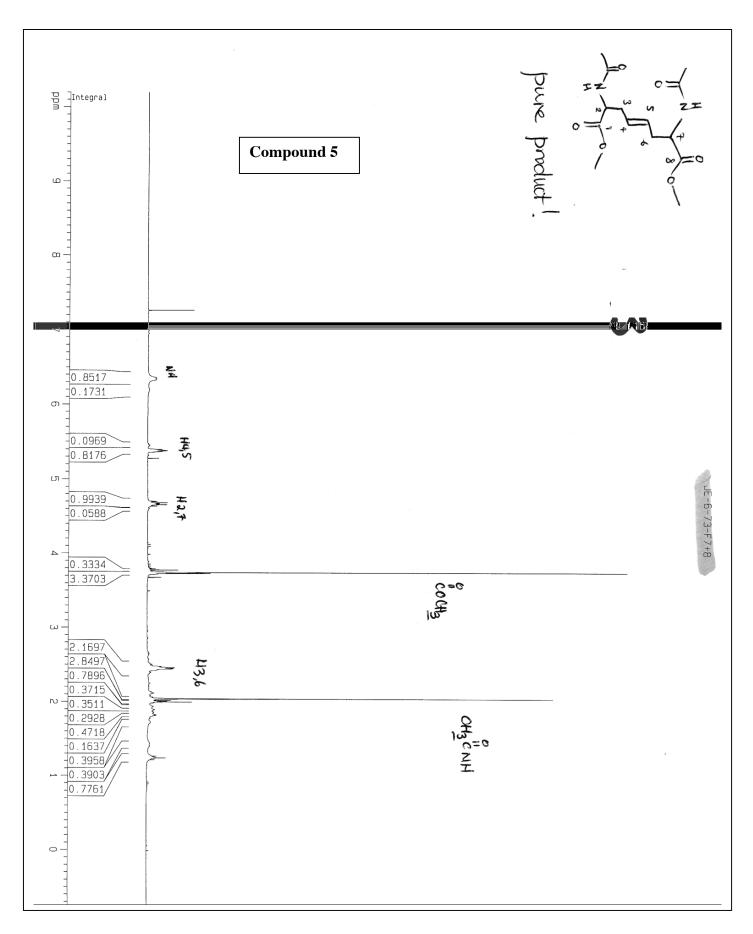


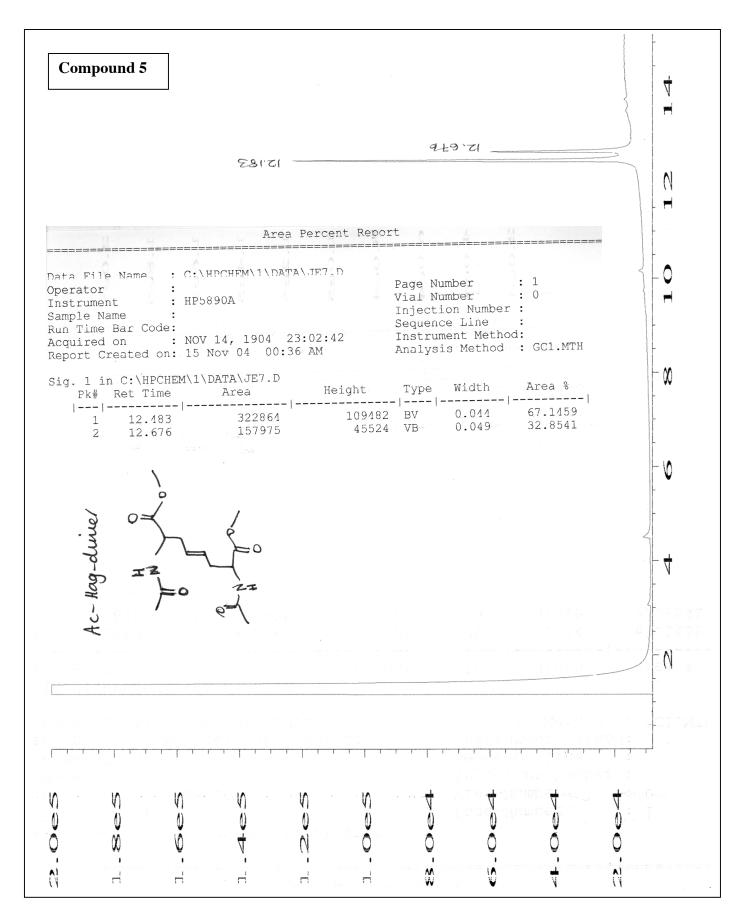


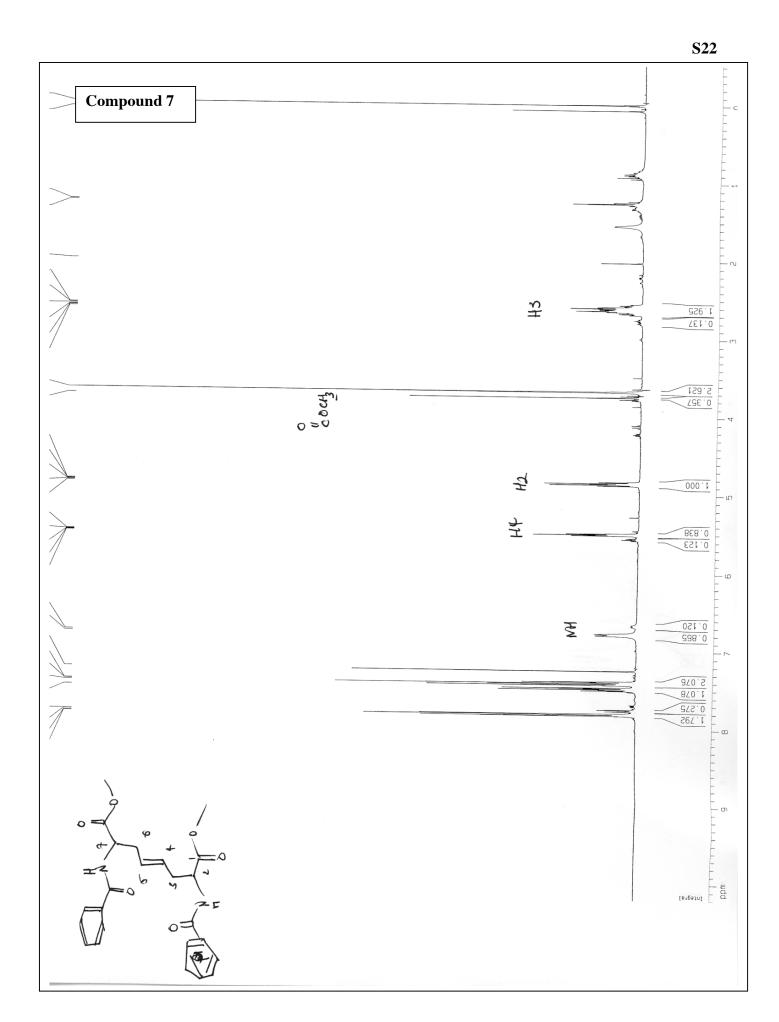
S19a

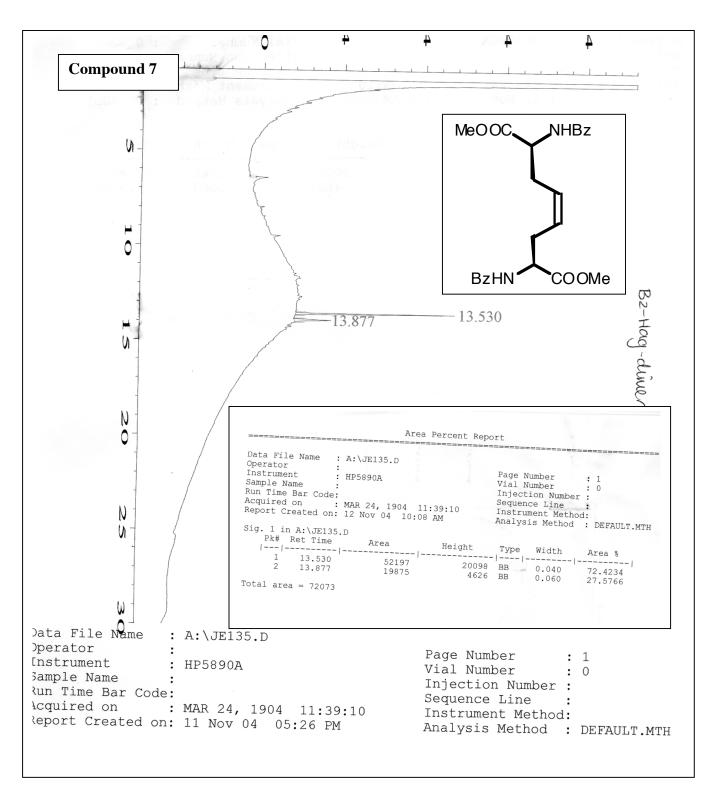






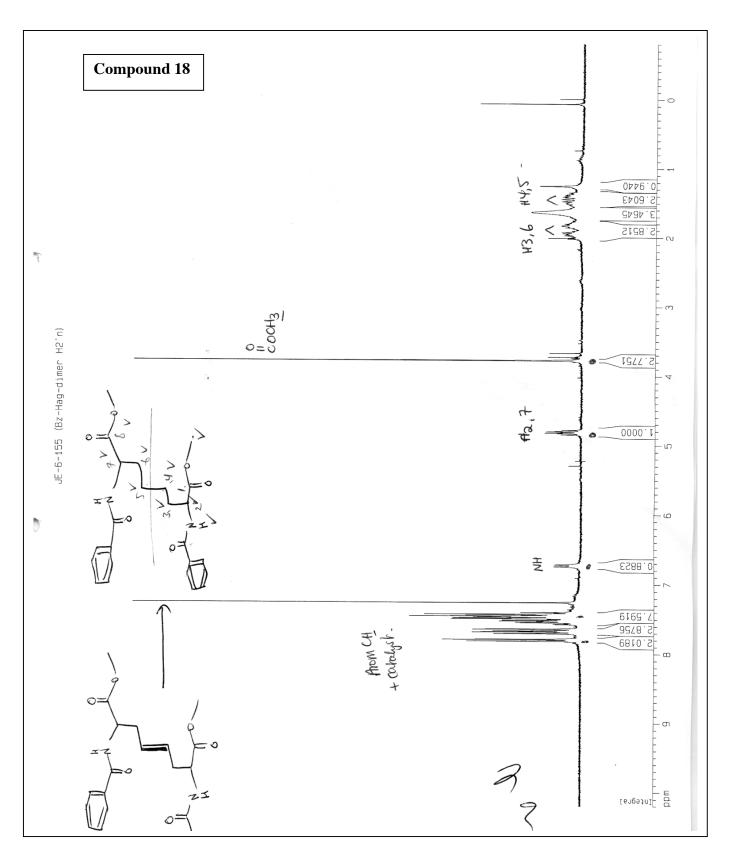


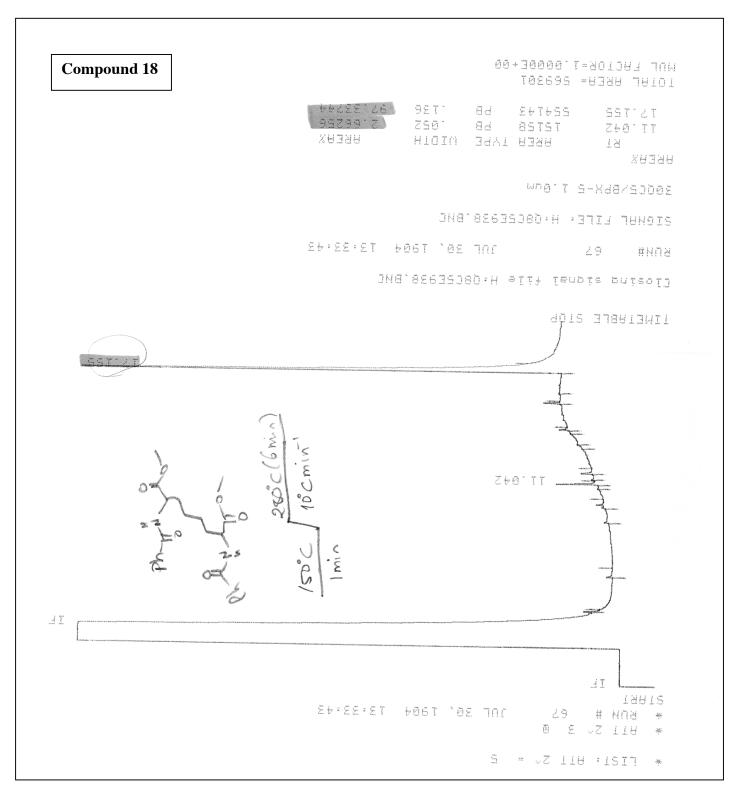




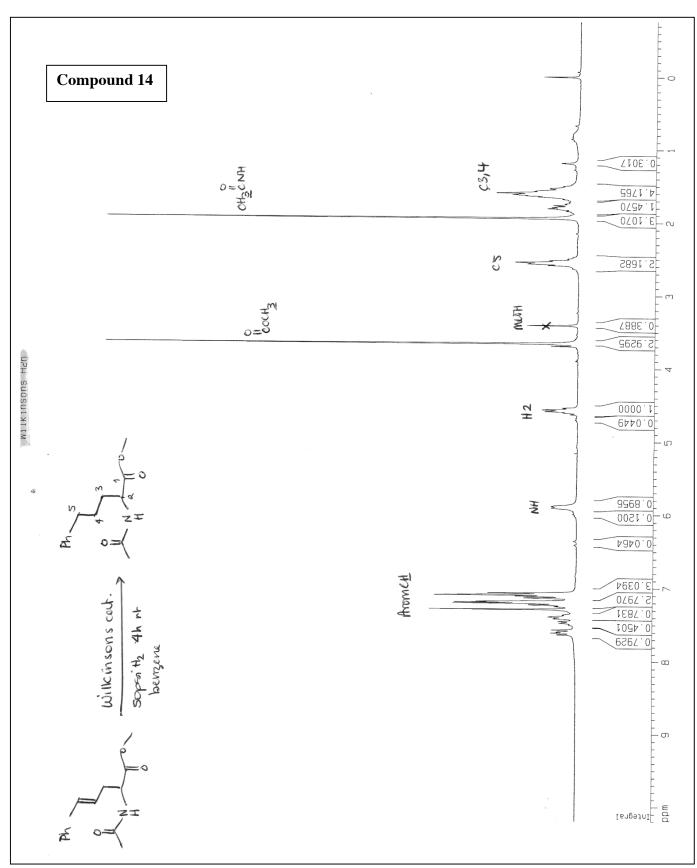
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S24





S26



MA 84:01 40 VON S1 :no belserd tropA HTM.131 : bodjeM sizylanA JUL 28, 1904 11:38:51 : Acquired on Instrument Method: Run Time Bar Code: Sequence Line : Sample Name : Injection Number : Jusmurlani A06829H : Yial Number : 0 Operator : Page Number τ: Data File Name : C:/HPCHEM/1/DATA/JE61.D **Compound 14** M Area Percent Report Data File Name : C:\HPCHEM\1\DATA\JE61.D Operator Instrument Page Number : 1 Vial Number : 0 Injection Number : Sequence Line : Instrument Method: Analysis Method : LC1.MTH Instrument : HrsosuA Sample Name : Run Time Bar Code: Acquired on : JUL 28, 1904 11:38:51 Report Created on: 12 Nov 04 10:48 AM : HP5890A Area Height Type Width Area % 1639190 28509 98.2905 1.7095 545977 BB 10214 BV --1 0.044 0.042 Ph 0 N AcHN COOMe 172.1月 982.01-0 Ň N 5 5 5 5 5 5

