Application of *bis*-acetylenic ketones in synthesis: one pot preparation of the 1,2,4-triazepine and oxa-triaza-indenone cores.

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General Experimental

^1H and ^{13}C Spectra were recorded on Brüker DPX200 (200MHz) or Brüker DQX 400 (400MHz), spectrometers at ambient temperatures. ^1H NMR spectral assignments are supported by ^1H - ^1H COSY where necessary. For ^1H NMR recorded in CDCl_3 chemical shifts (δ_{H}) are quoted in parts per million (ppm) and are referenced to the residual solvent peak. The following abbreviations are used: s, singlet, d, doublet, t, triplet, dd, doublet of doublets, ddd, doublet of doublet of doublets, dt, doublet of triplets, m, multiplet and br, broad. Coupling constants (J) were recorded in Hertz (Hz) to the nearest 0.5Hz. Carbon spectra are supported by DEPT, HMQC and COSY analyses where necessary.

Infrared (IR) spectra were recorded as thin films between NaCl plates on a Perkin-Elmer Paragon Fourier Transform spectrometer. Absorption maximum (ν_{max}) was reported in wave numbers (cm^{-1}) and only selected peaks are reported. The following abbreviations are used: w, weak, m, medium, s, strong and br, broad.

Low-resolution mass spectra (m/z) were recorded using a V.G.TRIO (GCMS) spectrometer, a Micromass Platform (APCI) spectrometer, Micromass Autospec spectrometer (CI^+) and a Micromass ZAB spectrometer (CI^+ , EI). Only molecular ion (M^+) and other major peaks are reported. High-resolution mass spectra were recorded on a Micromass Autospec spectrometer and are accurate to $\pm 5\text{ppm}$.

Melting points were obtained using a Büchi 510 Cambridge Instruments GallenTM III hot stage melting point apparatus and are uncorrected.

Flash chromatography was carried out using silica gel 60 0.040-0.063mm, 230-400 mesh as the stationary phase. Thin layer chromatography was carried out on aluminium backed plates pre-coated with Merck silica gel 60 F₂₅₄ (1.05554), which were visualized by quenching of u.v. fluorescence ($\lambda_{\text{max}} = 254\text{ nm}$) or by staining with either 10% w/v ammonium molybdate in 2M sulphuric acid or basic potassium permanganate solution (followed by heat) as appropriate. Retention factors (R_f) are reported to ± 0.5 .

General procedure for the preparation of compounds 3a/f (Scheme 1 and Table 1)

To a solution of freshly prepared ketone **1a/c** (1.0 mmol) in ethanol (103 mg / cm³) was added Boc-amidrazone **2a/b** (1.1 eq.) in one portion. The resultant deep red solutions were stirred at room temperature for 10 min before being absorbed onto silica gel and purified by flash chromatography.

1-*tert*-Butoxycarbonylamino-6-hydroxy-2-phenyl-6-phenylethynyl-1,6-dihydro-pyrimidine-4-carboxylic acid ethyl ester **3a**

Yellow oil (419 mg, 91% yield); R_f = 0.2 [light petroleum:EtOAc (3:1)]; $\nu_{\max}$ (Film)/cm⁻¹ 3450m (OH), 3353m (NH), 3256m (NH), 2203m (C≡C), 1740 (HNC=O), 1704s (CO₂Et); δ_H (200 MHz, CDCl₃) 7.84-7.79 (2H, m, Ar), 7.55-7.32 (8H, m, Ar), 6.27 (1H, s, =CH), 5.75 (2H, br, -OH and -NH), 4.36 (2H, q, J = 7 Hz, OCH₂CH₃), 1.46 (9H, s, (CH₃)₃COO), 1.34 (3H, t, J = 7 Hz, OCH₂CH₃); δ_C (50.3 MHz, CDCl₃): 174.8 (C=O), 164.4 (C=O), 163.1 (N=C=O), 150.1 (NC=N), 145.5 (CNOH), 132.9 (CH, Ar), 132.5 (C, Ar), 131.5 (CH, Ar), 130.5 (CH, Ar), 128.8 (CH, Ar), 128.5 (CH, Ar), 127.0 (CH, Ar), 120.3 (C, Ar), 111.7 (C=CH), 90.0 (C≡), 89.5 (C≡), 84.0 ((CH₃)₃C), 62.6 (CH₂), 27.9 ((CH₃)₃C), 13.5 (CH₃); HRMS found: MH⁺ 462.2038, C₂₆H₂₈N₃O₅ requires 462.2029; m/z (AP⁺) 462 (40%, MH⁺).

1-*tert*-Butoxycarbonylamino-6-hydroxy-6-pent-1-ynyl-2-phenyl-1,6-dihydro-pyrimidine-4-carboxylic acid ethyl ester **3b**

Yellow oil (368 mg, 86% yield); R_f = 0.3 [light petroleum:EtOAc (1:1)]; $\nu_{\max}$ (Film)/cm⁻¹ 3445m (OH), 3350m (NH), 3240m (NH), 2198m (C≡C), 1744s (HNC=O), 1710s (CO₂Et); δ_H (200 MHz, CDCl₃) 7.82-7.77 (2H, m, Ar), 7.52-7.46 (3H, m, Ar), 6.16 (1H, s, =CH), 5.65 (2H, br, -OH and -NH), 4.36 (2H, q, J = 7 Hz, OCH₂CH₃), 2.29 (2H, t, J = 7 Hz, CH₂C≡), 1.52 (2H, sextet, J = 7 Hz, CH₂CH₂C≡), 1.46 (9H, s, (CH₃)₃COO), 1.35 (3H, t, J = 7 Hz, OCH₂CH₃), 0.96 (3H, t, J = 7 Hz, CH₂CH₂CH₃); δ_C (50.3 MHz, CDCl₃): 175.1 (C=O), 164.6 (C=O), 163.0 (N=C=O), 150.2 (NC=N), 144.8 (CNOH), 132.5 (C, Ar), 131.6 (CH, Ar), 128.8 (CH, Ar), 126.9 (CH, Ar), 112.4 (C=CH), 93.0 (C≡), 83.9

((CH₃)₃C), 82.5 (C≡), 62.6 (CH₂), 60.4 (CH₂C≡), 27.9 ((CH₃)₃C), 21.2 (CH₂CH₂CH₃), 21.0 (CH₂CH₂CH₃), 13.5 (CH₂CH₃); HRMS found: MH⁺ 428.2179, C₂₃H₃₀N₃O₅ requires 428.2185; *m/z* (AP⁺) 428 (100%, MH⁺).

1-*tert*-Butoxycarbonylamino-6-hex-1-ynyl-6-hydroxy-2-phenyl-1,6-dihydro-pyrimidine-4-carboxylic acid ethyl ester 3c

Yellow oil (383 mg, 87% yield); *R_f* = 0.35 [light petroleum:EtOAc (1:1)]; *v*_{max} (Film)/cm⁻¹ 3455m (OH), 3356m (NH), 3240m (NH), 2199m (C≡C), 1742s (HNC=O), 1709s (CO₂Et); δ_H (200 MHz, CDCl₃) 7.81-7.76 (2H, m, Ar), 7.52-7.44 (3H, m, Ar), 6.13 (1H, s, =CH), 5.70 (2H, br, -OH and -NH), 4.33 (2H, q, *J* = 7 Hz, OCH₂CH₃), 2.30 (2H, t, *J* = 7 Hz, CH₂C≡), 1.50-1.30 (4H, m, CH₂CH₂CH₂C≡), 1.44 (9H, s, (CH₃)₃COO), 1.33 (3H, t, *J* = 7 Hz, OCH₂CH₃), 0.86 (3H, t, *J* = 7 Hz, CH₂CH₂CH₃); δ_C (50.3 MHz, CDCl₃): 175.1 (C=O), 164.4 (C=O), 163.0 (N=CC=O), 150.2 (NC=N), 144.8 (CNOH), 132.6 (C, Ar), 131.5 (CH, Ar), 128.8 (CH, Ar), 127.0 (CH, Ar), 112.1 (C=CH), 93.7 (C≡), 83.9 ((CH₃)₃C), 82.4 (C≡), 62.5 (CH₂), 60.4 (CH₂C≡), 29.7(CH₂CH₂CH₂C≡), 27.9 ((CH₃)₃C), 21.9 (CH₂CH₂CH₃), 18.8 (CH₂CH₂CH₂C≡), 13.5 (CH₂CH₃); HRMS found: MH⁺ 442.2325, C₂₄H₃₂N₃O₅ requires 442.2342; *m/z* (AP⁺) 442 (100%, MH⁺).

1-*tert*-Butoxycarbonylamino-6-hydroxy-2-methyl-6-phenylethynyl-1,6-dihydro-pyrimidine-4-carboxylic acid ethyl ester 3d

Yellow oil (351 mg, 88% yield); *R_f* = 0.3 [light petroleum:EtOAc (3:1)]; *v*_{max} (Film)/cm⁻¹ 3449m (OH), 3343m (NH), 3250m (NH), 2202m (C≡C), 1745s (HNC=O), 1706s (CO₂Et); δ_H (200 MHz, CDCl₃) 7.50-7.35 (5H, m, Ar), 6.25 (1H, s, =CH), 5.65 (2H, br, -OH and -NH), 4.34 (2H, q, *J* = 7 Hz, OCH₂CH₃), 2.02 (3H, s, (CH₃)C=N), 1.45 (9H, s, (CH₃)₃COO), 1.35 (3H, t, *J* = 7 Hz, OCH₂CH₃); δ_C (50.3 MHz, CDCl₃): 174.7 (C=O), 164.5 (C=O), 163.2 (N=CC=O), 150.1 (NC=N), 145.4 (CNOH), 132.9 (CH, Ar), 130.5 (CH, Ar), 128.5 (CH, Ar), 120.3 (C, Ar), 112.7 (C=CH), 90.0 (C≡), 89.5 (C≡), 84.0 ((CH₃)₃C), 62.6 (CH₂), 27.9 ((CH₃)₃C), 19.2 (CH₃C=N), 13.5 (CH₂CH₃); HRMS found: MH⁺ 400.1875, C₂₁H₂₆N₃O₅ requires 400.1872; *m/z* (AP⁺) 400 (100%, MH⁺).

1-tert-Butoxycarbonylamino-6-hydroxy-2-methyl-6-pent-1-ynyl-1,6-dihydro-pyrimidine-4-carboxylic acid ethyl ester 3e

Yellow oil (299 mg, 82% yield); $R_f = 0.35$ [light petroleum:EtOAc (1:1)]; $\nu_{\max}$ (Film)/ cm^{-1} 3449m (OH), 3349m (NH), 3248m (NH), 2197m ($\text{C}\equiv\text{C}$), 1741s ($\text{HNC}=\text{O}$), 1706s (CO_2Et); δ_{H} (200 MHz, CDCl_3) 6.09 (1H, s, $=\text{CH}$), 5.40 (2H, br, $-\text{OH}$ and $-\text{NH}$), 4.30 (2H, q, $J = 7$ Hz, OCH_2CH_3), 2.31 (2H, t, $J = 7$ Hz, $\text{CH}_2\text{C}\equiv$), 2.02 ($(\text{CH}_3)\text{C}=\text{N}$), 1.57 (2H, sextet, $J = 7$ Hz, $\text{CH}_2\text{CH}_2\text{C}\equiv$), 1.43 (9H, s, $(\text{CH}_3)_3\text{COO}$), 1.31 (3H, t, $J = 7$ Hz, OCH_2CH_3), 0.97 (3H, t, $J = 7$ Hz, $\text{CH}_2\text{CH}_2\text{CH}_3$); δ_{C} (50.3 MHz, CDCl_3): 175.2 ($\text{C}=\text{O}$), 164.6 ($\text{C}=\text{O}$), 163.2 ($\text{N}=\text{CC}=\text{O}$), 150.2 ($\text{NC}=\text{N}$), 144.8 (CNOH), 112.5 ($\text{C}=\text{CH}$), 93.7 ($\text{C}\equiv$), 83.9 ($(\text{CH}_3)_3\text{C}$), 82.4 ($\text{C}\equiv$), 62.5 (CH_2), 60.4 ($\text{CH}_2\text{C}\equiv$), 27.9 ($(\text{CH}_3)_3\text{C}$), 21.3 (OCH_2CH_3), 21.0 ($\text{CH}_2\text{CH}_2\text{CH}_3$), 19.1 ($\text{CH}_3\text{C}=\text{N}$), 13.5 (CH_2CH_3); HRMS found: MH^+ 366.2038, $\text{C}_{18}\text{H}_{28}\text{N}_3\text{O}_5$ requires 366.2029; m/z (AP^+) 366 (100%, MH^+).

1-tert-Butoxycarbonylamino-6-hex-1-ynyl-6-hydroxy-2-methyl-1,6-dihydro-pyrimidine-4-carboxylic acid ethyl ester 3f

Yellow oil (333 mg, 88% yield); $R_f = 0.35$ [light petroleum:EtOAc (1:1)]; $\nu_{\max}$ (Film)/ cm^{-1} 3460m (OH), 3349m (NH), 3263m (NH), 2195m ($\text{C}\equiv\text{C}$), 1735s ($\text{HNC}=\text{O}$), 1704s (CO_2Et); δ_{H} (200 MHz, CDCl_3) 6.05 (1H, s, $=\text{CH}$), 5.48 (2H, br, $-\text{OH}$ and $-\text{NH}$), 4.27 (2H, q, $J = 7$ Hz, OCH_2CH_3), 2.30 (2H, t, $J = 7$ Hz, $\text{CH}_2\text{C}\equiv$), 1.99 ($(\text{CH}_3)\text{C}=\text{N}$), 1.50-1.30 (4H, m, $\text{CH}_2\text{CH}_2\text{CH}_2\text{C}\equiv$), 1.44 (9H, s, $(\text{CH}_3)_3\text{COO}$), 1.30 (3H, t, $J = 7$ Hz, OCH_2CH_3), 0.86 (3H, t, $J = 7$ Hz, $\text{CH}_2\text{CH}_2\text{CH}_3$); δ_{C} (50.3 MHz, CDCl_3): 175.2 ($\text{C}=\text{O}$), 164.6 ($\text{C}=\text{O}$), 163.3 ($\text{N}=\text{CC}=\text{O}$), 150.2 ($\text{NC}=\text{N}$), 144.8 (CNOH), 112.2 ($\text{C}=\text{CH}$), 93.9 ($\text{C}\equiv$), 83.8 ($(\text{CH}_3)_3\text{C}$), 82.3 ($\text{C}\equiv$), 62.4 (CH_2), 60.4 ($\text{CH}_2\text{C}\equiv$), 29.7 ($\text{CH}_2\text{CH}_2\text{CH}_2\text{C}\equiv$), 27.8 ($(\text{CH}_3)_3\text{C}$), 21.9 ($\text{CH}_2\text{CH}_2\text{CH}_3$), 19.1 ($\text{CH}_3\text{C}=\text{N}$), 18.8 ($\text{CH}_2\text{CH}_2\text{CH}_2\text{C}\equiv$), 13.5 (CH_2CH_3); HRMS found: MH^+ 380.2183, $\text{C}_{19}\text{H}_{30}\text{N}_3\text{O}_5$ requires 380.2125; m/z (AP^+) 380 (100%, MH^+).

Preparation of 2-Oxo-4-phenyl-7a-phenylethynyl-2,3-dihydro-7aH-1-oxa-3,3a,5-triaza-indene-6-carboxylic acid ethyl ester 4 (Scheme 2)

Method A: A solution of **3a** (460 mg, 1 mmol) in THF (80 mg / cm³) was heated at reflux and TBAF (3 eq., 1M solution in THF) was added. After 2h, the reaction mixture was allowed to cool at room temperature, quenched with diethylether (20 mL) and washed with water (3 x 30 mL). The organic layer was dried over Na₂SO₄, absorbed onto silica gel and purified by flash chromatography, to yield **4** (348mg, 90% yield) as a colourless solid (m.p. 179-82°C Ethanol).

Method B: To a solution of freshly prepared ketone **1a** (1.0 mmol) in THF (103 mg / cm³) was added *Boc*-amidrazone **2a** (1.1 eq.) in one portion. The resultant deep red solution was stirred at room temperature for 10 min. The solution was heated at reflux and then TBAF (3 eq., 1M solution in THF) was added. After 2h, the reaction mixture was allowed to cool at room temperature, quenched with diethylether (20 mL) and washed with water (3 x 30 mL). The organic layer was dried over Na₂SO₄, absorbed onto silica gel and purified by flash chromatography to yield **4** (335mg, 87% yield).

Colourless solid, m.p. 179-82°C (Ethanol). $R_f = 0.4$ [light petroleum:EtOAc (1:1)]; (Film)/cm⁻¹ 3375m (NH), 3546m (NH), 2201m (C≡C), 1745 (HNC=O), 1710s (CO₂Et); δ_H (200 MHz, CDCl₃) 7.90-7.85 (2H, m, Ar), 7.76-7.61 (2H, m, Ar), 7.53-7.38 (6H, m, Ar), 7.13 (1H, s, =CH), 4.50 (2H, q, $J = 7$ Hz, OCH₂CH₃), 1.43 (3H, t, $J = 7$ Hz, OCH₂CH₃); δ_C (50.3 MHz, CDCl₃): 174.3 (C=O), 162.1 (C=O), 154.3 (N=C=O), 149.7 (NC=N), 140.6 (CNOH), 133.0 (CH, Ar), 131.1 (C, Ar), 130.6 (CH, Ar), 128.8 (CH, Ar), 128.6 (CH, Ar), 126.4 (CH, Ar), 125.8 (C, Ar), 120.2 (CH, Ar), 110.3 (C=CH), 90.7 (C≡), 89.6 (C≡), 62.4 (CH₂), 13.8 (CH₂CH₃); HRMS found: MH⁺ 388.1301, C₂₂H₁₇N₃O₄ requires 388.1297; m/z (AP⁺) 388 (20%, MH⁺).

Preparation of 3-Phenyl-7-phenylethynyl-2-(2,2,2-trifluoro-acetyl)-2H-[1,2,4]triazepine-5-carboxylic acid ethyl ester **6a and 3-Phenyl-7-phenylethynyl-1-(2,2,2-trifluoro-acetyl)-1H-[1,2,4]triazepine-5-carboxylic acid ethyl ester **6b** (Scheme 3)**

Method A: To a solution of **3a** (460 mg, 1 mmol) in DCM (1 mL / 100mg) was added trifluoroacetic anhydride (1 mL) and the solution obtained was heated at reflux. After 2h, the reaction mixture was allowed to cool at room temperature and the solvent removed *in vacuo*. The oil so obtained was dissolved in diethylether (10 mL), washed with water (5 mL), then saturated NaHCO₃ (5 mL) and finally with water (10 mL). The organic layer was dried over MgSO₄, absorbed onto silica gel and purified by flash chromatography to yield **6** (382 mg, 87% yield) as a yellow oil.

Method B: To a solution of freshly prepared ketone **1a** (225 mg, 1 mmol) in DCM (100 mg / cm³) was added Boc-amidrazone **2a** (260 mg, 1.1 eq.) in one portion. The resultant deep red solution was stirred at room temperature for 10 min. At this point trifluoroacetic anhydride (1 mL / 70 mg of **1a**) was added and the mixture was heated at reflux. After 2h, the reaction mixture was allowed to cool at room temperature and the solvent removed *in vacuo*. The oil so obtained was dissolved in diethylether (10 mL), washed with water (5 mL), then saturated NaHCO₃ (5 mL) and finally with water (10 mL). The organic layer was dried over MgSO₄, absorbed onto silica gel and purified by flash chromatography to yield **6** (360mg, 82% yield) as a yellow oil.

$R_f = 0.7$ [light petroleum:EtOAc (4:1)]; $\nu_{\max}$ (Film)/cm⁻¹ 2204m (C≡C), 1740s (CO₂Et); δ_H (400 MHz, (CD₃)₂SO)) 8.10-8.07 (2H, m, Ar, major isomer), 8.01-7.98 (2H, m, Ar, minor isomer), 7.94 (1H, s, =CH, major isomer), 7.78 (1H, s, =CH, minor isomer), 7.72-7.38 (8H, m, Ar), 4.38 (2H, q, $J = 7$ Hz, OCH₂CH₃, minor isomer), 4.30 (2H, q, $J = 7$ Hz, OCH₂CH₃, major isomer), 1.28 (3H, t, $J = 7$ Hz, OCH₂CH₃, minor isomer), 1.18 (3H, t, $J = 7$ Hz, OCH₂CH₃, major isomer); δ_C (100.6 MHz, (CD₃)₂SO)): 173.2 (CF₃C=O), 172.6 (CF₃C=O), 162.7 (C=O), 160.2 (C=O), 136.0 (C), 134.8 (CH, Ph), 133.7 (C), 133.2 (CH, Ph), 131.6 (CH, Ph), 131.3 (C), 130.6 (=CH), 130.3 (=CH), 129.1 (CH, Ph),

128.6 (CH, Ph), 128.5 (CH, Ph), 128.4 (CH, Ph), 126.7 (CH, Ph), 126.6 (CH, Ph), 119.0 (C), 118.3 (C), 116.3 (C), 95.5 (C≡), 87.3 (C≡), 63.7 (CH₂), 63.5 (CH₂), 13.8 (CH₂CH₃), 13.5 (CH₂CH₃); δ_F (400 MHz, CDCl₃) –61.68 (3F, s, CF₃CO, major isomer), –62.8 (3F, s, CF₃CO, minor isomer); HRMS found: M⁺ 439.1140, C₂₃H₁₆F₃N₃O₃ requires 439.1143; m/z (AP⁺) 439 (100%, M⁺).

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 Group jeb
 mfaa21 -1c

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AQ 2.5559540 sec
RG 408.4
DM 156.000 umsec
DE 1.0000000 umsec
TE 300.2 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 7.10 umsec
PL1 -1.00 dB
SFO1 500.1310007 MHz

F2 - Processing parameters
SI 32768
SF 500.1310007 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

D3 NMR plot parameters
SI 32768
SF 500.1310007 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

===== CHANNEL f2 =====
NUC2 13C
P2 12.00 umsec
PL2 -1.00 dB
SFO2 125.7611500 MHz

F2 - Processing parameters
SI 65536
SF 125.7611500 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

Group job
mfaaz6 crude

Current Data Parameters
NAME 05072001-12
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030705
Time 19:29
INSTRUM dpx200
PROBHD 5 mm Dual 13
PULPROG zgpg30
TD 32768
AQ 0.03000000 sec
RG 327.68
SR 1.00000000 sec
DS 4
SFO1 50.1277413 MHz
FIDRES 0.369570 Hz
AQ 1.3566452 sec
RG 18384
SR 4146.000000 sec
DS 100.000000 sec
F2 - Processing parameters
SI 32768
SF 50.1277413 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
C1 15.00 cm
F1P 220.000 ppm
F1 11071.00 Hz
F2P -10.000 ppm
F2 -503.23 Hz
PPH2 6.57143 ppm/cm
PPH2N 330.69226 Hz/cm

*** Current Data Parameters ***

NAME	EXPNO	PROCNO	Acquisition	Parameters
NAME	:	:	rmatrixid	:
EXPNO	:	1	51	:
PROCNO	:	1	400.1356012 MHz	:
DATE	:	Jul 15 2004	DMSO	:
NS	:	51		:
SF01	:	400.1356012 MHz		:
SOLVENT	:	DMSO		:

*** Current Data Parameters ***

NAME	EXPNO	PROCNO
rematri	13	1

*** Acquisition Parameters ***

DATE	TIME	INSTRUM	PROBHD
Jul 12 2004	22:26	NS	226
SFO1	100.6254358	MHz	
SOLVENT	CDCl3