Electronic Supplementary Information

for

Structure and reactivity of bicyclic methylene aziridines prepared by intramolecular aziridination of allenes

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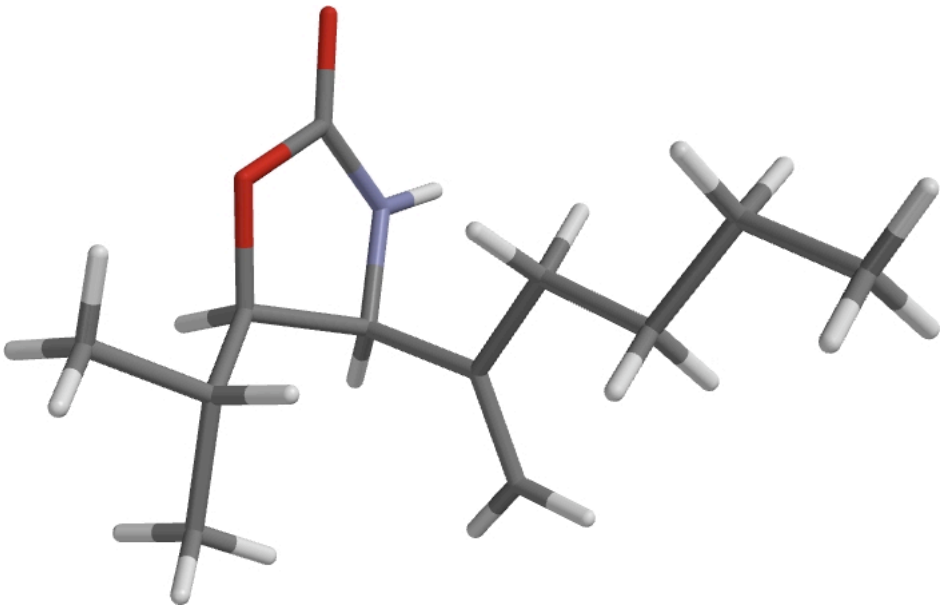
(1) Experimental procedures and characterization data for products listed in Tables 1 and 2, and Scheme 5

(2) Graphic of oxazolidinone (23) from single crystal X-ray diffraction co-ordinates

(3) NMR spectra [^{1}H, ^{13}C, HSQC, C-H HMBC, N-H HMBC] for methylene aziridine (2), ^{1}H and ^{13}C NMR spectra for oxazolidinones (21)–(25), and tricyclic carbamate (28) with NOESY spectrum

Molecular structure of oxazolidinone (23) from single crystal X-ray diffraction co-ordinates.



General procedure for the formation of methylene aziridines from *N*-tosyloxycarbamates (Table 1)

To a stirred solution of *N*-tosyloxycarbamate **8**, **9**, **11**, **13**, **15**, or **18** (1.0 eq.) in degassed acetone (8 mL/mmol) at 25 °C was added Rh_2OAc_4 (0.05 equiv.) and K_2CO_3 (7.0 equiv.). The reaction mixture was stirred vigorously for 16–18 h and was then diluted with dichloromethane, filtered through Celite® and concentrated *in vacuo* to give the crude product which was then purified as described.

(4*R,5*S**)-4-Isopropyl-6-methylene-3-oxa-1-azabicyclo[3.1.0]hexan-2-one (2)**

The reaction of tosyloxycarbamate **8** according to the general procedure, and purification by column chromatography (petrol/ether, 2:1) afforded the *title compound* (119 mg, 27%) as a yellow oil. R_f 0.30 (petrol/ether, 1:1); ν_{max} (thin film)/cm^{-1} 2967s, 2878m, 2360m, 2342m, 1792s, 1470m, 1393m, 1341m; δ_H (400 MHz, $CDCl_3$) 1.03 and 1.08 (2 × 3 H, 2 × d, *J* 6.7, $CH(CH_3)_2$), 1.77–1.86 (1 H, m, $CH(CH_3)_2$), 3.64 (1 H, d, *J* 5.6, CHN), 4.36 (1 H, dd, *J* 9.5, 5.6, CHO), 5.18 (1 H, d, *J* 3.2) and 5.39 (1 H, dt, *J* 3.2, 1.0, $=CH_2$); δ_C (100 MHz, $CDCl_3$) 17.5, 19.7, 32.1, 43.7, 83.7, 91.4, 130.2, 162.8; HRMS (FI^+) found 153.0789, $C_8H_{11}NO_2$ (M^+) requires 153.0790.

(4*R,5*S**)-4-Benzyl-6-methylene-3-oxa-1-azabicyclo[3.1.0]hexan-2-one (10)**

The reaction of tosyloxycarbamate **9** according to the general procedure, and purification by column chromatography (petrol/ether, 2:1) afforded the *title compound* (7 mg, 7%) as a yellow oil although sufficient purity for full characterization was not attained. R_f 0.24 (petrol/ether, 1:1); δ_H (500 MHz, $CDCl_3$) 2.91 (1 H, dd, *J* 13.7, 7.8) and 3.18 (1 H, dd, *J* 13.7, 6.5, $PhCH_2$), 3.54 (1 H, dt, *J* 5.5, 1.0, CHN), 4.96 (1 H, app. dt, *J* 7.8, 6.5, CHO), 5.26 (1 H, d, *J* 3.3) and 5.46 (1 H, dt, *J* 3.3, 1.0, $=CH_2$), 7.23–7.38 (5 H, m, Ph); δ_C (125 MHz, $CDCl_3$) 38.8, 44.0, 78.5, 91.9, 127.5, 129.0, 129.1, 130.2, 134.6, 162.5.

(4*R,5*S**)-6-Methylene-4-(naphthalen-1-ylmethyl)-3-oxa-1-azabicyclo[3.1.0]hexan-2-one (12)**

The reaction of tosyloxycarbamate **11** according to the general procedure, and purification by column chromatography (petrol/ether, 4:1) afforded the *title compound* (11 mg, 20%) as a yellow oil that was crystallized from ether to yield colourless plates, m.p. 84 °C. R_f 0.24 (petrol/ether, 2:1); ν_{max} (thin film)/cm^{-1} 3049s, 1791s, 1597s, 1511s, 1396s, 1175s; δ_H (500 MHz, $CDCl_3$) 3.47 (1 H, dd, *J* 14.1, 8.1, ArC*H*H′), 3.48 (1

H, dt, *J* 5.5, 1.0, CHN), 3.74 (1 H, dd, *J* 14.1, 5.8, ArCH*H*′), 5.16–5.20 (1 H, m, CHO), 5.27 (1 H, d, *J* 3.3) and 5.49 (1 H, dt, *J* 3.3, 1.0, $=CH_2$), 7.39 (1 H, d, *J* 7.0), 7.46 (1 H, dd, *J* 8.2, 6.9), 7.53–7.57 (1 H, m), 7.58–7.62 (1 H, m), 7.84 (1 H, d, *J* 8.2), 7.92 (1 H, d, *J* 8.1) and 8.00 (1 H, d, *J* 8.2, Ar); δ_H (500 MHz, $CDCl_3$) 35.6, 44.1, 77.6, 92.0, 122.9, 125.6, 126.1, 126.7, 127.6, 128.4, 129.1, 130.3, 130.5, 131.7, 134.0, 162.5; HRMS (ESI^+) not found; however, crystals were grown that proved suitable for X-ray crystallographic analysis and proof of identity and purity.

(4*R,5*S**)-6-Methylene-4-[2-(5-methylfuran-2-yl)ethyl]-3-oxa-1-azabicyclo[3.1.0]hexan-2-one (14)**

The reaction of tosyloxycarbamate **13** according to the general procedure, and purification by column chromatography (petrol/ether, 4:1) afforded the *title compound* (33 mg, 16%) as a yellow oil. R_f 0.18 (petrol/ether, 2:1); δ_H (200 MHz, $CDCl_3$) 2.05 (2 H, app. q, *J* 6.9, C*H*$_2$CHO), 2.27 (3 H, s, CH_3), 2.62–2.90 (2 H, m, C*H*$_2$furan), 3.56 (1 H, dt, *J* 5.6, 1.0, CHN), 4.77 (1 H, app. q, *J* 6.4, CHO), 5.19 (1 H, dt, *J* 3.1, 1.0) and 5.39 (1 H, dt, *J* 3.1, 0.5, $=CH_2$), 5.85–5.89 (1 H, m) and 5.94 (1 H, d, *J* 3.0, furan). Full characterization was not possible as rearrangement to carbamate **28** during isolation could not be prevented.

4-(Propen-2-yl)-3,6-dihydro-2*H*-1,3-oxazin-2-one (16) and 1,1,3,3-tetramethyl-3,7-dihydrooxazolo[3,4-*c*][1,3]oxazin-5(1*H*)-one (17)

The reaction of tosyloxycarbamate **15** according to the general procedure, and purification by column chromatography (neat petrol → petrol/ether, 2:1) afforded *oxazinone* **17** (18 mg, 4%) as a pale yellow oil. R_f 0.34 (petrol/ether, 1:1); ν_{max} (thin film)/cm^{-1} 3271m, 2957m, 1701s, 1412m, 1271m, 1146m, 928m; δ_H (500 MHz, C_6D_6) 1.25 and 1.75 (2 × 6 H, 2 × s, 2 × $C(CH_3)_2$), 3.87 (1 H, t, *J* 3.0, CH=), 4.37 (2 H, d, *J* 3.0, CH_2O); δ_C (100 MHz, C_6D_6) 27.5, 29.3, 67.4, 79.7, 86.0, 96.3, 144.5, 148.0; HRMS (ESI^+) not found. Further elution afforded *oxazinone* **16** (20 mg, 6%) as a pale yellow oil. R_f 0.08 (petrol/ether, 1:1); ν_{max} (thin film)/cm^{-1} 3364m br, 2983m, 1726s, 1379s, 1280s, 1178s, 1091m, 1009m, 841m, 814m; δ_H (500 MHz, $CDCl_3$) 1.97 (3 H, s, CH_3), 4.95 (2 H, d, *J* 4.0, CH_2O), 5.18 and 5.29 (2 × 1 H, 2 × s, $=CH_2$) overlays 5.18 (1 H, multiplicity not visible, =C*H*CH_2O), 7.70 (1 H, br s, NH); δ_C 19.7, 66.8, 96.9, 112.7, 134.6, 136.8, 153.1; HRMS (ESI^+) found 162.0528, $C_7H_9NNaO_2$ (MNa^+) requires 162.0525.

4,4-Dimethyl-7-methylene-3-oxa-1-azabicyclo[4.1.0]heptan-2-one (19)

The reaction of tosyloxycarbamate **18** according to the general procedure, and purification by column chromatography (petrol/ethyl acetate, 1:1) afforded the *title compound* (42 mg, 9%) as a yellow oil. R_f 0.17 (petrol/ether, 1:1); ν_{max} (thin film)/cm^{-1} 3288br, 2982s, 2936s, 1956s, 1722s, 1492m, 1348m, 1275s; δ_H (500 MHz, $CDCl_3$) 1.43 (3 H, s, CH_3), 1.47 (1 H, dd, *J* 14.3, 9.1, C*H*H′), 1.62 (3 H, s, CH_3), 2.23 (1 H, dd, *J* 14.3, 6.5, CH*H*′), 3.38 (1 H, dd, *J* 9.1, 6.5, CHN), 4.97 and 5.15 (2 × 1 H, 2 × d, *J* 2.9, $=CH_2$); δ_C (125 MHz, $CDCl_3$) 25.3, 29.3, 34.1, 37.5, 84.8, 85.8, 134.4, 155.5; HRMS (FI^+) found 153.0789, $C_8H_{11}NO_2$ (M^+) requires 153.0790.

Ring-opening reactions (Table 2)

***cis*-4-(But-1-en-2-yl)-5-isopropyloxazolidin-2-one (21)**

Ethylmagnesium bromide (0.29 mL of a 1.0 M solution in THF, 0.29 mmol) was added dropwise to a stirred suspension of CuI (1 mg, 0.005 mmol) in THF (1 mL) in a pear-shaped flask at −40 °C. After 10 min, $BF_3{\cdot}OEt_2$ (0.018 mL, 0.147 mmol) was added followed by a solution of methylene aziridine **2** (15 mg, 0.098 mmol) in THF (0.5 mL) *via* canula. The mixture was allowed to warm to 0 °C over 1 h and was then quenched with NH_4Cl solution (saturated, aqueous, 1 mL) and warmed to RT. The organic components were extracted into ether (3 × 5 mL), the combined organic extracts were then washed with brine (5 mL), dried over $MgSO_4$ and concentrated *in vacuo*. The residue was purified by column chromatography (petrol/ether, 2:1 → 1:1) to afford the *title compound* (13 mg, 72%) as a yellow oil. R_f 0.25 (ether); ν_{max}/cm^{-1} (thin film) 3237br, 2964s, 1745s, 1730m, 1417s, 1217s; δ_H (500 MHz, $CDCl_3$) 0.90 and 1.07 (2 × 3 H, 2 × d, *J* 6.6, $CH(CH_3)_2$) 1.10 (3 H, t, *J* 7.3, CH_3CH_2), 1.76–1.86 (1 H, m, $CH(CH_3)_2$), 2.02–2.18 (2 H, m, CH_3CH_2), 4.24 (1 H, dd, *J* 9.2, 7.0, CHO), 4.30 (1 H, d, *J* 7.0, CHN), 5.07 and 5.09 (2 × 1 H, 2 × s, $=CH_2$), 5.21 (1 H, br s, NH); δ_C (125 MHz, $CDCl_3$) 11.8, 19.10, 19.14, 24.1, 28.0, 61.6, 85.8, 114.3, 146.7, 159.6; HRMS (ESI^+) found 206.1151, $C_{10}H_{17}NNaO_2$ (MNa^+) requires 206.1151.

***cis*-5-Isopropyl-4-(3-methylbut-1-en-2-yl)oxazolidin-2-one (22)**

Iso-propylmagnesium chloride (0.49 mL of a 2.0 M solution in THF, 0.98 mmol) was added dropwise to a stirred suspension of CuI (3.1 mg, 0.016 mmol) in THF (1.5 mL) in a pear-shaped flask at −40 °C. After 10 min, $BF_3{\cdot}OEt_2$ (0.030 mL, 0.243 mmol) was added followed by a solution of methylene aziridine **2** (25 mg, 0.163 mmol) in THF (0.5 mL) *via* canula. The mixture was allowed to warm to 0 °C over 1 h and was then quenched with NH_4Cl solution (saturated, aqueous, 2 mL) and warmed to RT. The organic components were extracted into ether (3 × 10 mL), the combined organic extracts were then washed with brine (5 mL), dried over $MgSO_4$ and concentrated *in vacuo*. The residue was purified by column chromatography (petrol/ether, 2:1 → 1:1) to afford the *title compound* (8 mg, 26%) as a colourless oil. R_f 0.20 (ether); ν_{max}/cm^{-1} (thin film) 3205br, 2124br, 2958s, 2927w, 1767s, 1737s, 1462m, 1406s, 1375s, 1318m; δ_H (500 MHz, $CDCl_3$) 0.90 and 1.06 (2 × 3 H, 2 × d, *J* 6.6, $CH(CH_3)_2$), 1.10 and 1.13 (2 × 3 H, 2 × d, *J* 6.7, $=CCH(CH_3)_2$), 1.85 (1 H, oct, *J* 6.6, $CH(CH_3)_2$), 2.20–2.26 (1 H, m, $=CCH(CH_3)_2$), 4.31–4.35 (2 H, m, CHO and CHN), 4.98 (1 H, br s, NH) 5.14 and 5.17 (2 × 1 H, 2 × s, $=CH_2$); δ_C (125 MHz, $CDCl_3$) 18.4, 19.3, 22.8, 23.4, 28.6, 31.2, 60.5, 85.6, 112.6, 151.9, 159.3; HRMS (ESI^+) found 220.1305, $C_{11}H_{19}NNaO_2$ (MNa^+) requires 220.1308.

***cis*-4-(Hex-1-en-2-yl)-5-isopropyloxazolidin-2-one (23)**

Butylmagnesium chloride (0.56 mL of a 2.0 M solution in THF, 1.12 mmol) was added dropwise to a stirred suspension of CuI (10.7 mg, 0.056 mmol) in THF (2 mL) in a pear-shaped flask at −40 °C. After 10 min, a solution of methylene aziridine **2** (85 mg, 0.555 mmol) in THF (1 mL) was added *via* canula. The mixture was allowed to warm to 0 °C over 1 h and was then quenched with NH_4Cl solution (saturated, aqueous, 3 mL), stirred for 30 min, and then warmed to RT. The organic components were extracted into ether (3 × 20 mL), the combined organic extracts were then washed with brine (15 mL), dried over $MgSO_4$ and concentrated *in vacuo*. The residue was purified by column chromatography (petrol/ether, 1:1 → neat ether) to afford the *title compound* (37 mg, 32%) as a colourless oil. R_f 0.20 (ether); δ_H (500 MHz, $CDCl_3$) 0.90 and 1.07 (2 × 3 H, 2 × d, *J* 6.5, $CH(CH_3)_2$), 0.93 (3 H, t, *J* 7.3, CH_3CH_2), 1.36 (2 H, app. sext, *J* 7.6, CH_3CH_2), 1.45–1.51 (2 H, m, $CH_2CH_2C=$), 1.84 (1 H, dsept, *J* 8.8, 6.5, $CH(CH_3)_2$), 1.99–2.12 (2 H, m, $CH_2C=$), 4.23–4.29 (2 H, m, CHO and CHN), 4.96 (1 H, br s, NH), 5.07–5.09 (2 H, m, $=CH_2$); δ_C (125 MHz, $CDCl_3$) 11.8, 19.10, 19.12, 22.5, 28.2, 30.2, 31.2, 61.6, 85.8, 114.9, 145.4, 159.4; HRMS (ESI^+) found 234.1469, $C_{12}H_{21}NNaO_2$ (MNa^+) requires

234.1465. The oil was crystallized by vapour diffusion of petrol into an ether solution of the sample allowing X-ray crystallographic analysis and further proof of identity and purity.

cis-5-Isopropyl-4-(1-phenylvinyl)oxazolidin-2-one (24)

Phenyllithium (0.21 mL of a 1.8 M solution in dibutyl ether, 0.38 mmol) was added dropwise to a stirred suspension of CuI (35 mg, 0.183 mmol) in ether (1 mL) in a pear-shaped flask at 0 °C. After 1.5 h, a solution of methylene aziridine **2** (28 mg, 0.183 mmol) in ether (0.5 mL) was added *via* canula. The mixture was stirred for 30 min and was then quenched with NH_4Cl solution (saturated, aqueous, 5 mL) and allowed to warm to RT. The organic components were extracted into ether (3 × 10 mL), the combined organic extracts were then washed with brine (10 mL), dried over $MgSO_4$ and concentrated *in vacuo*. The residue was purified by column chromatography (petrol/ether, 1:1) to afford the *title compound* (19 mg, 45%) as a colourless oil. R_f 0.27 (ether); ν_{max}/cm^{-1} (thin film) 3278br, 2986s, 2900m, 1751br, 1471w, 1389s, 1375s, 1256m; δ_H (500 MHz, $CDCl_3$) 0.60 and 1.00 (2 × 3 H, 2 × d, *J* 6.6, $CH(CH_3)_2$) 1.81 (1 H, app. oct, *J* 6.6, $CH(CH_3)_2$), 4.41 (1 H, t, *J* 7.5, CHO), 4.95 (1 H, d, *J* 7.5, CHN), 5.44 (1 H, d, *J* 1.3, =C*H*H′), 5.60 (1 H, s, =CH*H*′), 5.81 (1 H, br s, NH) 7.32–7.37 (5 H, m, Ph); δ_C (125 MHz, $CDCl_3$) 17.9, 19.5, 28.5, 58.4, 85.6, 116.7, 126.2, 128.3, 128.9, 139.6, 144.5, 159.8; HRMS (ESI^+) found 254.1156, $C_{14}H_{17}NNaO_2$ (MNa^+) requires 254.1151.

cis-4-(Buta-1,3-dien-2-yl)-5-isopropyloxazolidin-2-one (25)

Vinylmagnesium bromide (1.18 mL of a 1.0 M solution in THF, 1.18 mmol) was added dropwise to a stirred suspension of CuI (6 mg, 0.03 mmol) in THF (2 mL) in a pear-shaped flask at −40 °C. After 10 min, a solution of methylene aziridine **2** (90 mg, 0.59 mmol) in THF (1 mL) was added *via* canula. The mixture was allowed to warm to 0 °C over 1 h and was then quenched with NH_4Cl solution (saturated, aqueous, 3 mL), stirred for 30 min, and then warmed to RT. The organic components were extracted into ether (3 × 20 mL), the combined organic extracts were then washed with brine (15 mL), dried over $MgSO_4$ and concentrated *in vacuo*. The residue was purified by column chromatography (petrol/ether, 1:1 → neat ether) to afford the *title compound* (33 mg, 31%) as an off-white solid. R_f 0.18 (ether); m.p. 81 °C; ν_{max}/cm^{-1} (thin film) 3266br, 2362m, 1745s, 1403m, 1249s; δ_H (500 MHz, $CDCl_3$) 0.90 and 1.01 (2 × 3 H, 2 × d, *J* 6.8, $CH(CH_3)_2$) 1.90 (1 H, oct, *J* 6.8, $CH(CH_3)_2$), 4.45 (1 H, dd, *J* 8.0, 6.8, CHO), 4.65 (1 H, d, *J* 8.0, CHN), 5.13 (1 H, br s, NH), 5.19 (1 H, d, *J*

11.3, CH=C*H*H′), 5.29 (1 H, s, C=C*H*H′), 5.31 (1 H, d, *J* 17.5, CH=CH*H*′), 5.43 (1 H, s, C=CH*H*′), 6.38 (1 H, dd, *J* 17.5, 11.3, C*H*=CH_2); δ_C (125 MHz, $CDCl_3$) 17.8, 19.8, 28.4, 57.1, 84.8, 115.5, 117.9, 135.9, 141.9, 159.3; HRMS (ESI^+) found 204.0997, $C_{10}H_{15}NNaO_2$ (MNa^+) requires 204.0995.

(1*R,4*R**,5*R**,8*R**)-8-Methyl-11-oxatricyclo[6.2.1.0^{1,5}]undec-9-en-6-one-4-yl carbamate (28)**

Method 1. A sample of methylene aziridine **2** in $CDCl_3$ was monitored periodically by ^{1}H NMR spectroscopy until resonances attributed to the starting material (**14**) were no longer visible (28 d). The sample was then concentrated and the residue purified by preparative TLC (petrol/ether, 1:1) to afford the *title compound* as a colourless oil. See below for data.

Method 2. Camphorsulfonic acid (5 mg, 0.022 mmol) was added to a solution of methylene aziridine **14** (33 mg, 0.15 mmol) in chloroform (3 mL). The reaction reaction mixture was stirred at RT for 48 h and then concentrated *in vacuo*. The residue was purified by column chromatography (petrol/ethyl acetate, 1:1) to afford the *title compound* (7 mg, 22%) as a colourless oil. R_f 0.29 (ethyl acetate); ν_{max}/cm^{-1} (thin film) 3627s, 3585s, 3356br, 2970br, 1712br, 1610s, 1395s, 1337s; δ_H (500 MHz, C_6D_6) 1.32 (3 H, s, CH_3), 1.40–1.45 (1 H, m, H-2), 1.99–2.08 (2 H, m, H′-2 and H-3), 2.11–2.16 (1 H, m, H′-3), 2.20 (1 H, d, *J* 7.9, H-5), 2.28 and 2.40 (2 × 1 H, 2 × d, *J* 17.2, H-7 and H′-7), 3.76 (2 H, br s, NH_2), 5.59 (1 H, d, *J* 5.7, H-10), 5.62 (1 H, d, *J* 5.7, H-9) 5.66 (1 H, ddd, *J* 9.5, 7.9, 3.2, H-4); δ_C (125 MHz, $CDCl_3$) 24.0, 32.1, 32.8, 50.1, 60.0, 75.9, 83.0, 92.5, 134.9, 138.0, 155.8, 205.9; HRMS (ESI^+) found 260.0889, $C_{12}H_{15}NNaO_4$ (MNa^+) requires 260.0893.

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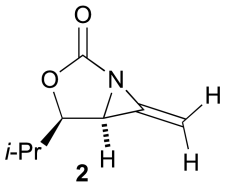

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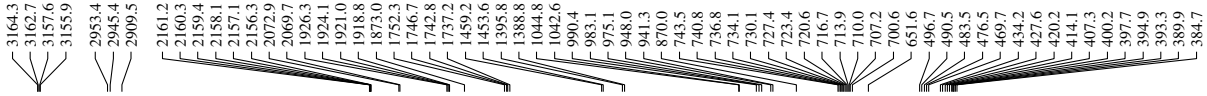

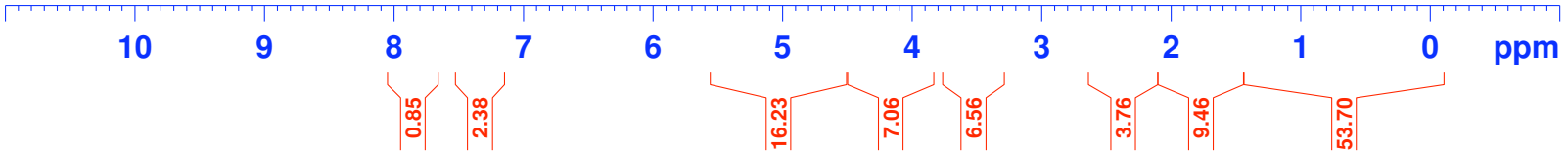

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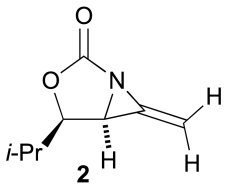

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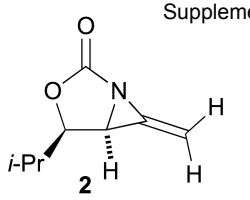
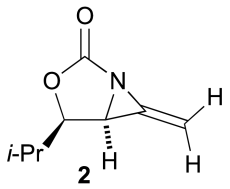
SFO2        400.2016008 MHz

F2 - Processing parameters
SI                32768
SF          100.6303718 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

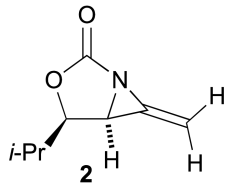


Acquisition Date: 2007-01-22 17:40:13.688
Processed by: Jeremy Robertson
Pulse Sequence: hmbcgpl2ndqf
Spectrometer Freq.: 500.30 MHz
Solvent: $CDCl_3$
Temperature: 297.97 K
Sweep Width: 8.06 ppm
Number of Scans: -1073750780
Relaxation Delay: 0.000 s



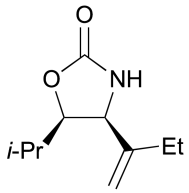
2

Acquisition Date: 2007-05-04 10:59:15.675
Processed by: Jeremy Robertson
Pulse Sequence: hmbcgpndqf
Spectrometer Freq.: 500.13 MHz
Solvent: DMSO
Temperature: 298.05 K
Sweep Width: 6.01 ppm
Number of Scans: -1073748140
Relaxation Delay: 0.000 s



2

Instrument AVC500
8683 George Feast 3/1/07



21

NMR@CHEM.OX

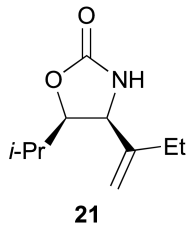
```
Current Data Parameters
NAME          gf86830301
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20080103
Time               18.54
INSTRUM          avc500
PROBHD   5 mm CPDUL 13C
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10330.578 Hz
FIDRES         0.157632 Hz
AQ            3.1719923 sec
RG                     4
DW               48.400 usec
DE                6.00 usec
TE                300.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.60 usec
PL1               -6.00 dB
SFO1        500.3030896 MHz

F2 - Processing parameters
SI                 32768
SF          500.3000240 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```


Instrument AVC500
8683 George Feast 3/1/07



21

NMR@CHEM.OX

```
Current Data Parameters
NAME          gf86830301
EXPNO                  4
PROCNO                 1

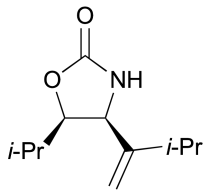
F2 - Acquisition Parameters
Date_           20080103
Time               21.24
INSTRUM          avc500
PROBHD   5 mm CPDUL 13C
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 2048
DS                    2
SWH           31250.000 Hz
FIDRES         0.476837 Hz
AQ            1.0486259 sec
RG                 1820
DW               16.000 usec
DE               20.00 usec
TE               298.0 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                8.00 usec
PL1              -4.40 dB
SFO1        125.8131151 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            80.00 usec
PL12             12.40 dB
PL13             17.00 dB
PL2              -6.00 dB
SFO2        500.3020012 MHz

F2 - Processing parameters
SI                32768
SF          125.8005438 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```


Instrument AVC500
9120 George Feast 14/2/08



22

NMR@CHEM.OX

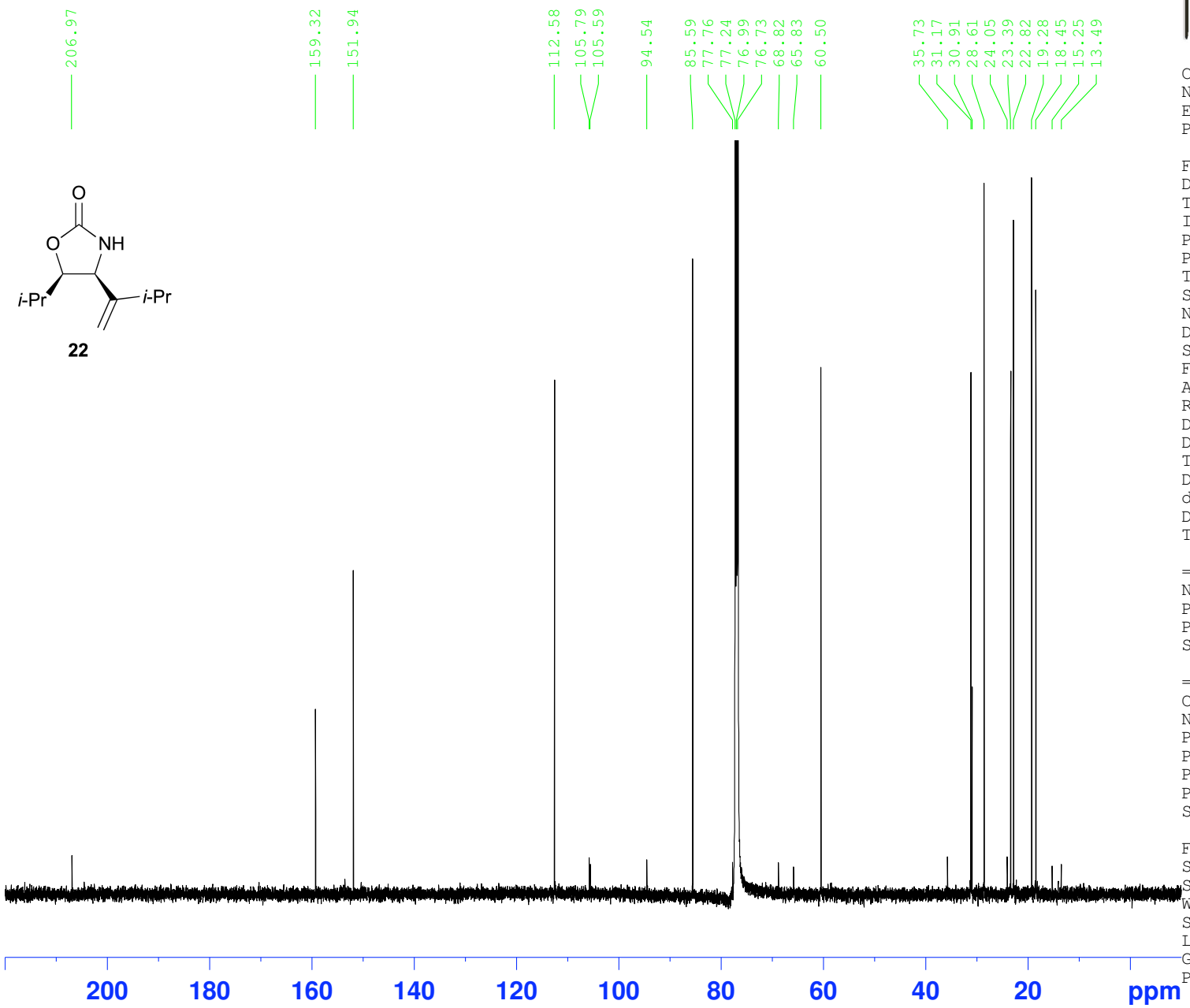
```
Current Data Parameters
NAME          gf91201402
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20080215
Time               20.37
INSTRUM          avc500
PROBHD   5 mm CPDUL 13C
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           10330.578 Hz
FIDRES         0.157632 Hz
AQ            3.1719923 sec
RG                    4
DW               48.400 usec
DE                6.00 usec
TE               300.0 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.60 usec
PL1               -6.00 dB
SFO1        500.3030896 MHz

F2 - Processing parameters
SI                32768
SF          500.3000240 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

Instrument AVC500
9120 George Feast 14/2/08



22

NMR@CHEM.OX

```
Current Data Parameters
NAME          gf91201402
EXPNO                  4
PROCNO                 1

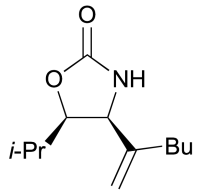
F2 - Acquisition Parameters
Date_           20080216
Time                0.01
INSTRUM          avc500
PROBHD   5 mm CPDUL 13C
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 3072
DS                    2
SWH           31250.000 Hz
FIDRES         0.476837 Hz
AQ            1.0486259 sec
RG                  912
DW               16.000 usec
DE               20.00 usec
TE               300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                8.00 usec
PL1               -4.40 dB
SFO1        125.8131151 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2            80.00 usec
PL12              12.40 dB
PL13              17.00 dB
PL2               -6.00 dB
SFO2        500.3020012 MHz

F2 - Processing parameters
SI                 32768
SF          125.8005438 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                1.40
```


Instrument AVC500
GEORGE FEAST 9893 14/4/08



23

NMR@CHEM.OX

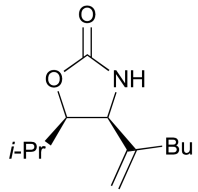
```
Current Data Parameters
NAME          gf98931404
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20080415
Time               18.36
INSTRUM          avc500
PROBHD   5 mm CPDUL 13C
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           10330.578 Hz
FIDRES         0.157632 Hz
AQ            3.1719923 sec
RG                    4
DW               48.400 usec
DE                6.00 usec
TE               300.0 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.60 usec
PL1               -6.00 dB
SFO1        500.3030896 MHz

F2 - Processing parameters
SI                32768
SF          500.3000240 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```


Instrument AVC500
GEORGE FEAST 9893 14/4/08



23

NMR@CHEM.OX

```
Current Data Parameters
NAME          gf98931404
EXPNO                  4
PROCNO                 1

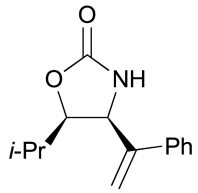
F2 - Acquisition Parameters
Date_           20080415
Time               20.14
INSTRUM          avc500
PROBHD   5 mm CPDUL 13C
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    2
SWH           31250.000 Hz
FIDRES         0.476837 Hz
AQ            1.0486259 sec
RG                 1820
DW               16.000 usec
DE               20.00 usec
TE                300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                8.00 usec
PL1               -4.40 dB
SFO1        125.8131151 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2            80.00 usec
PL12              12.40 dB
PL13              17.00 dB
PL2               -6.00 dB
SFO2        500.3020012 MHz

F2 - Processing parameters
SI                32768
SF          125.8005438 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```


Instrument AVC500
9092 George Feast 13/2/08



24

NMR@CHEM.OX

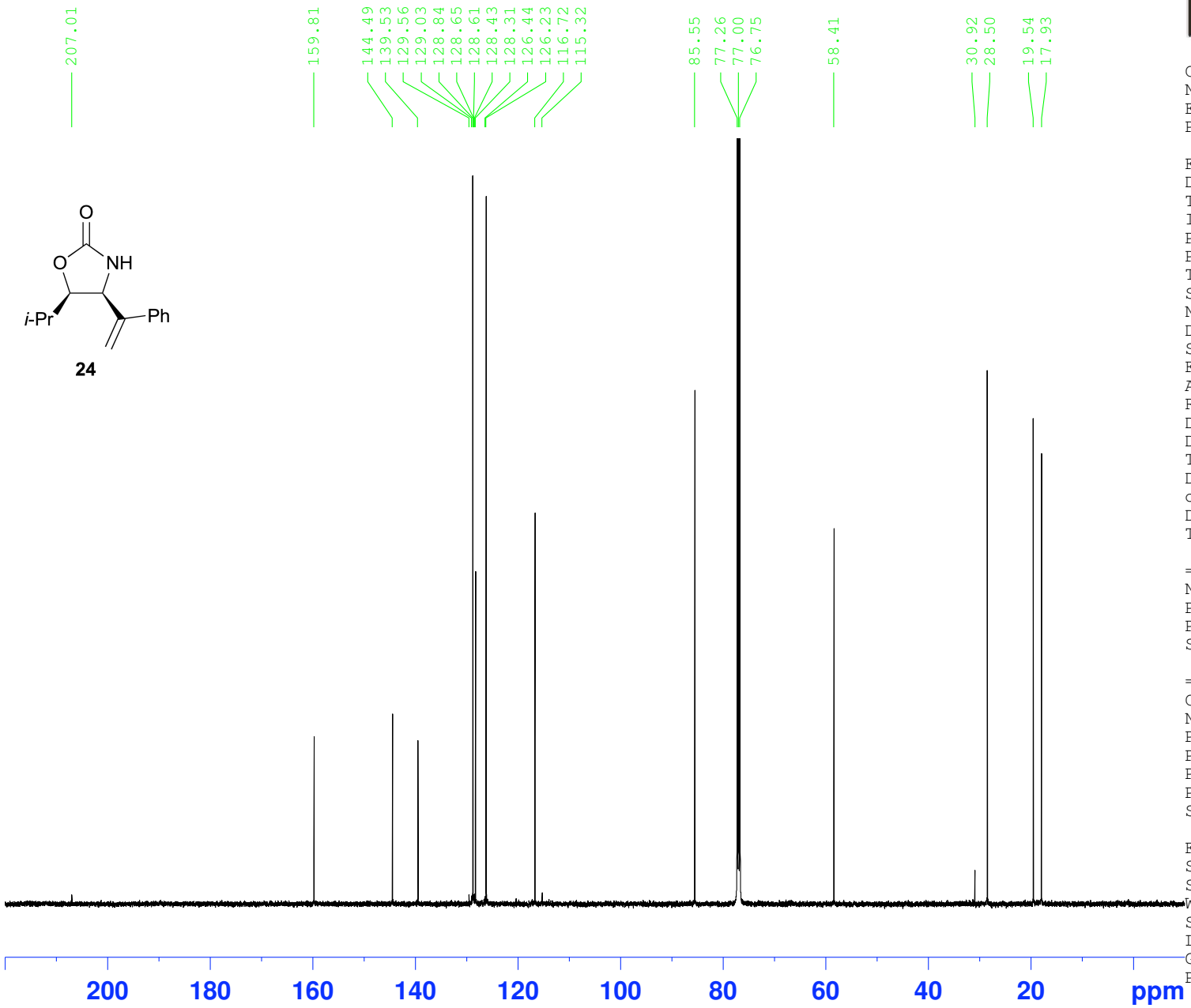
```
Current Data Parameters
NAME          gf90921302
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20080213
Time                9.15
INSTRUM          avc500
PROBHD   5 mm CPDUL 13C
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10330.578 Hz
FIDRES         0.157632 Hz
AQ            3.1719923 sec
RG                     4
DW               48.400 usec
DE                6.00 usec
TE                300.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.60 usec
PL1               -6.00 dB
SFO1        500.3030896 MHz

F2 - Processing parameters
SI                 32768
SF          500.3000240 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```

Instrument AVC500
9092 George Feast 13/2/08



24

NMR@CHEM.OX

```
Current Data Parameters
NAME          gf90921302
EXPNO                  4
PROCNO                 1

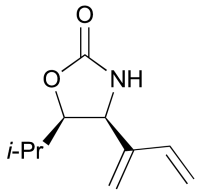
F2 - Acquisition Parameters
Date_           20080213
Time                9.44
INSTRUM          avc500
PROBHD   5 mm CPDUL 13C
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  256
DS                    2
SWH           31250.000 Hz
FIDRES         0.476837 Hz
AQ            1.0486259 sec
RG                  912
DW               16.000 usec
DE               20.00 usec
TE               300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                8.00 usec
PL1               -4.40 dB
SFO1        125.8131151 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2            80.00 usec
PL12              12.40 dB
PL13              17.00 dB
PL2               -6.00 dB
SFO2        500.3020012 MHz

F2 - Processing parameters
SI                  32768
SF          125.8005438 MHz
WDW                    EM
SSB                     0
LB                1.00 Hz
GB                      0
PC                1.40
```


Instrument AVC500
GEORGE FEAST 1829 24/10/08

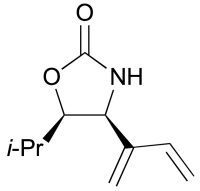


25

NMR@CHEM.OX

NAME	gf18292410
EXPNO	1
PROCNO	1
Date_	20081027
Time	17.00
INSTRUM	avc500
PROBHD	5 mm CPDUL 13C
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1719923 sec
RG	4
DW	48.400 usec
DE	6.00 usec
TE	298.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	9.60 usec
PL1	-6.00 dB
PL1W	15.19999981 W
SFO1	500.3030896 MHz
SI	32768
SF	500.3000240 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

Instrument AVC500
GEORGE FEAST 1829 24/10/08

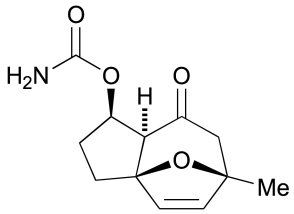


25

NMR@CHEM.OX

NAME	gf18292410
EXPNO	4
PROCNO	1
Date_	20081027
Time	19.56
INSTRUM	avc500
PROBHD	5 mm CPDUL 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	3072
DS	2
SWH	31250.000 Hz
FIDRES	0.476837 Hz
AQ	1.0486259 sec
RG	1820
DW	16.000 usec
DE	20.00 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	8.00 usec
PL1	-4.40 dB
PL1W	28.15752029 W
SFO1	125.8131151 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-6.00 dB
PL12	12.40 dB
PL13	17.00 dB
PL2W	15.19999981 W
PL12W	0.21970686 W
PL13W	0.07618046 W
SFO2	500.3020012 MHz
SI	32768
SF	125.8005438 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

Instrument AVC500
0436 George Feast 20/5/08



28

Proton NMR in CDCl3

NMR@CHEM.OX

```
Current Data Parameters
NAME          gf04362005
EXPNO                  1
PROCNO                 1

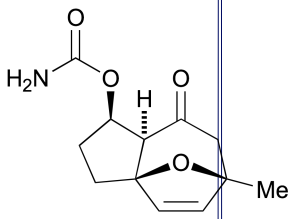
F2 - Acquisition Parameters
Date_           20080520
Time               13.14
INSTRUM          avc500
PROBHD   5 mm CPDUL 13C
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           10330.578 Hz
FIDRES         0.157632 Hz
AQ            3.1719923 sec
RG                    4
DW               48.400 usec
DE                6.00 usec
TE               300.0 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.60 usec
PL1               -6.00 dB
SFO1        500.3030896 MHz

F2 - Processing parameters
SI                32768
SF          500.3000240 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

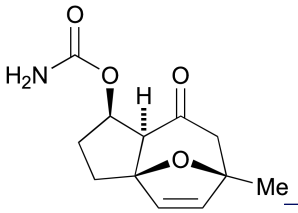
Acquisition Date: 2008-06-06 13:53:04.079
Processed by: Jeremy Robertson
Pulse Sequence: zg30
Spectrometer Freq.: 500.30 MHz
Solvent: C_6D_6
Temperature: 297.97 K
Sweep Width: 20.65 ppm
Number of Scans: -1073748140
Relaxation Delay: 0.000 s

Proton NMR in C6D6



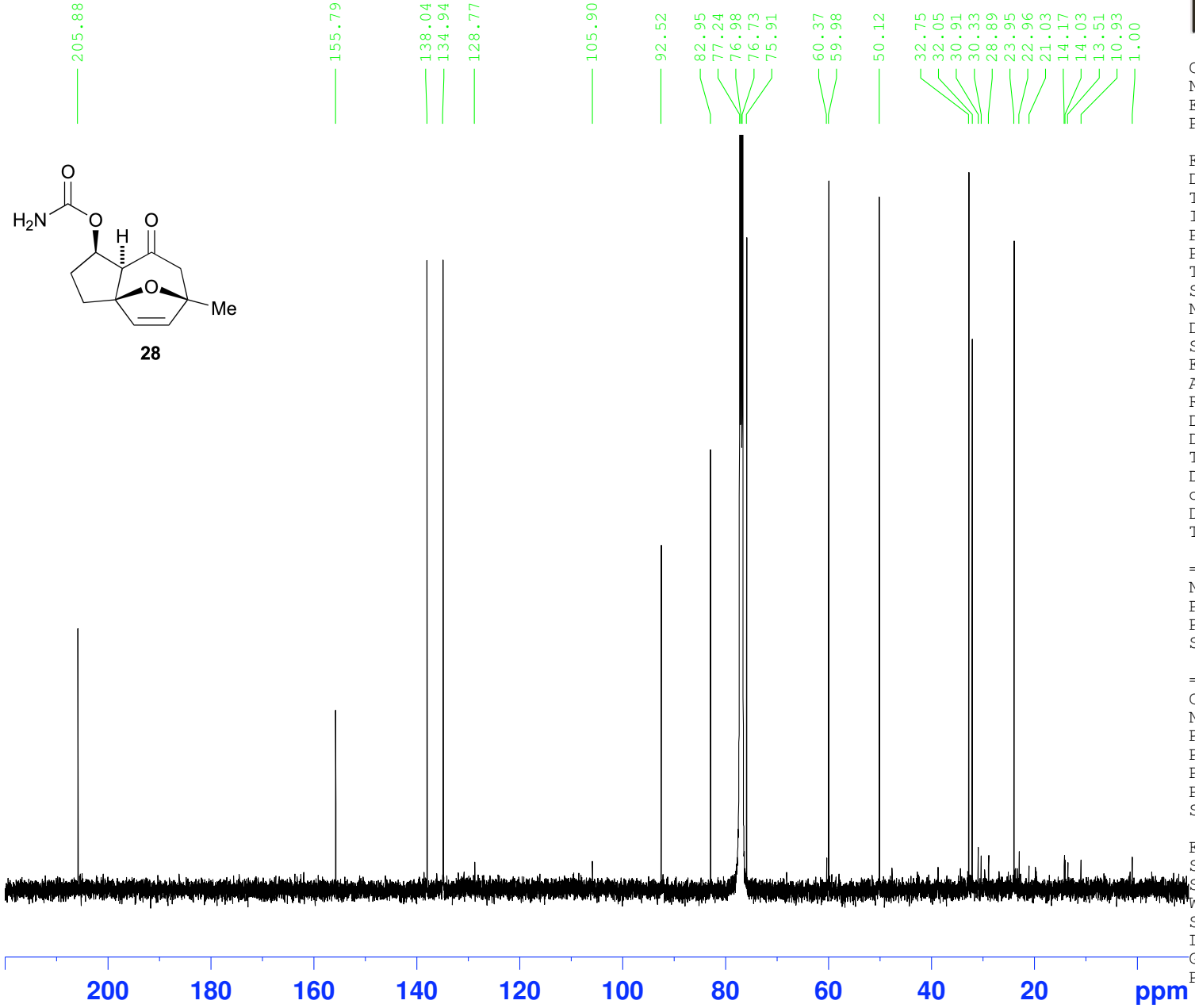
28

NOESY Spectrum



28

Instrument AVC500
0436 George Feast 20/5/08



28

NMR@CHEM.OX

```
Current Data Parameters
NAME          gf04362005
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20080520
Time               19.13
INSTRUM          avc500
PROBHD   5 mm CPDUL 13C
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 3072
DS                    2
SWH           31250.000 Hz
FIDRES         0.476837 Hz
AQ            1.0486259 sec
RG                 1820
DW               16.000 usec
DE               20.00 usec
TE               300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 8.00 usec
PL1               -4.40 dB
SFO1        125.8131151 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                  1H
PCPD2             80.00 usec
PL12              12.40 dB
PL13              17.00 dB
PL2               -6.00 dB
SFO2        500.3020012 MHz

F2 - Processing parameters
SI                 32768
SF          125.8005438 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                1.40
```