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Andrew J. Clark, Joanna V. Geden, Stephen Thom, Paul Wilson.

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^1H NMR spectra were recorded in CDCl_3 , unless otherwise stated, at either 300, 400 or 500 MHz. Chemical shifts (δ) are quoted in parts per million (ppm) with residual protic solvent CHCl_3 ($\delta_{\text{H}}=7.26$ ppm) used as internal reference. Coupling constants were measured in hertz. ^{13}C NMR spectra were recorded in CDCl_3 , unless otherwise stated, at 75.5, 100.6 and 125.8 MHz. Melting points are uncorrected. Flash chromatography was performed on silica gel. Analytical thin layer chromatography was carried out using aluminium backed plates pre-coated with silica (0.2 mm, 60F₂₅₄) and visualised by ultra-violet radiation (254 nm), acidic ammonium molybdate or potassium permanganate. Preparative thin layer chromatography was performed on glass backed silica gel plates (60 Å, 1000 μm , 20 x 20 cm). All reactions were carried out under nitrogen unless otherwise stated. Petrol refers to petroleum ether b.p. 40-60°C.

2-Bromo-N-buta-1,3-dienyl-N-(4-methoxy-benzyl)-2-methyl-propionamide (11a)

Pale yellow oil (12%), R_f (9:1 petrol:ethyl acetate) 0.27; ν_{max} (film)/ cm^{-1} 2932, 2836, 1660, 1630, 1512, 1460, 1426, 1391, 1362, 1311, 1246, 1175, 1150, 1109, 1034, 998, 817, 647; δ_{H} (400 MHz, CDCl_3) 7.68 (1H, d, J 13.8 Hz), 7.09 (2H, d, J 8.5 Hz), 6.84 (2H, d, J 8.5 Hz), 6.31 (1H, ddd, J 17.0, 10.4, 10.4 Hz), 5.72 (1H, dd, J 13.8, 10.4 Hz), 5.06 (1H, d, J 17.0 Hz), 4.97 (1H, d, J 10.4 Hz), 4.91 (2H, br s), 3.76 (3H, s), 2.03 (6H, s); δ_{C} (100.6 MHz, CDCl_3) 169.1, 158.7, 134.9, 132.8, 128.7, 127.6, 115.2, 114.2, 114.1, 56.7, 55.2, 48.9, 32.6; m/z (FAB) 338 (MH)⁺, 307, 289, 206, 154, 137, 121; [Found: (MH)⁺, 338.0759, $\text{C}_{16}\text{H}_{20}\text{NO}_2\text{Br}$ requires (MH)⁺, 338.0756].

2-Bromo-N-(4-methoxy-benzyl)-2-methyl-N-(3-methyl-buta-1,3-dienyl-propionamide (11b)

Orange oil (52%), R_f (9:1 petrol:ethyl acetate) 0.41; ν_{max} (film)/ cm^{-1} 2935, 2836, 1659, 1628, 1512, 1456, 1392, 1381, 1299, 1246, 1175, 1160, 1108, 1033, 1001, 946, 878, 816, 778, 644, 610; δ_{H} (400 MHz, CDCl_3) 7.70 (1H, d, J 14.3 Hz), 7.10 (2H, d, J 8.8 Hz), 6.83 (2H, d, J 8.8 Hz), 5.83 (1H, d, J 14.3 Hz), 4.91 (2H, br s), 4.85 (1H, br s), 4.83 (1H, br s), 3.72 (3H, s), 2.04 (6H, s), 1.89 (3H, s); δ_{C} (100.6 MHz, CDCl_3) 169.2, 158.7, 140.4, 130.1, 128.9, 127.6, 115.8, 115.1, 114.2, 56.9, 55.2, 48.6, 32.6, 19.2; m/z (FAB) 354 (MH)⁺, 352 (MH)⁺, 324, 307, 286, 234, 206, 190, 154, 136, 121; [Found: (MH)⁺, 352.0897, $\text{C}_{17}\text{H}_{22}\text{NO}_2\text{Br}$ requires (MH)⁺, 352.0912].

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2:1 *E*- and *Z*-2-bromo-*N*-(4-methoxy-benzyl)-2-methyl-*N*-penta-1,3-dienyl-propionamide (11c)

Pale brown oil (41%), R_f (9:1 petrol:ethyl acetate) 0.27; $\nu_{\max}$ (film)/ cm^{-1} 2933, 2837, 1664 (C=O), 1611, 1513, 1460, 1358, 1299, 1248, 1177, 1111, 1032, 826; δ_H (400 MHz, CDCl_3) 7.61 (0.5 H, d, J 13.1 Hz, *Z*), 7.53 (1H, d, J 13.8 Hz, *E*), 7.12 (1H, d, J 8.8 Hz, *Z*), 7.09 (2H, d, J 8.8 Hz, *E*), 6.86 (1H, d, J 8.8 Hz, *Z*), 6.84 (2H, d, J 8.8 Hz, *E*), 6.06-5.93 (2H, m, *E* and *Z*), 5.70 (1H, dd, J 13.8, 10.5 Hz, *E*), 5.55 (1H, dq, J 13.6, 6.8 Hz, *E*), 5.42 (0.5H, dq, J 9.5, 6.5 Hz, *Z*), 4.94 (1H, br s, *Z*), 4.89 (2H, br s, *E*), 3.77 (1.5 H, s, *Z*), 3.76 (3H, s, *E*), 2.04 (3H, s, *Z*), 2.03 (6H, s, *E*), 1.72 (3H, dd, J 6.8, 1.5 Hz, *E*), 1.62 (1.5 H, br d, J 6.5 Hz, *Z*); δ_C (100.6 MHz, CDCl_3) 169.1 and 169.0 (*E* and *Z*), 158.7 and 158.6 (*E* and *Z*), 132.5 (*Z*), 130.2 (*E*), 129.1 (*E*), 129.0 (*E* and *Z*), 127.8 (2 x *E* and *Z*), 127.1 (*Z*), 124.7 (*Z*), 114.5 (*E*), 114.1 (*E* and *Z*), 109.5 (*Z*), 56.9 (*E*), 56.8 (*Z*), 55.2 (*E* and *Z*), 49.1 (*E* and *Z*), 32.6 (*E* and *Z*), 18.2 (*E*), 13.4 (*Z*); m/z (FAB) 307, 206, 154, 136, 121.

2-Bromo-*N*-(4-methoxy-benzyl)-2-methyl-*N*-(4-methyl-penta-1,3-dienyl)-propionamide (11d)

Pale yellow oil (58%), R_f (9:1 petrol:ethyl acetate) 0.37; $\nu_{\max}$ (film)/ cm^{-1} 2965, 2929, 2837, 1651, 1612, 1513, 1456, 1395, 1372, 1340, 1310, 1248, 1173, 1145, 1109, 1036, 997, 946, 814; δ_H (400 MHz, CDCl_3) 7.50 (1H, d, J 13.5 Hz), 7.11 (2H, d, J 8.8 Hz), 6.83 (2H, d, J 8.8 Hz), 5.90 (1H, dd, J 13.3, 11.0 Hz), 5.78 (1H, br d), 4.92 (2H, br s), 3.73 (3H, s), 2.03 (6H, s), 1.74 (3H, s), 1.60 (3H, s); δ_C (100.6 MHz, CDCl_3) 168.9, 158.7, 133.8, 130.0, 129.2, 127.8, 122.5, 114.1, 111.5, 57.0, 55.2, 49.2, 32.7, 26.0, 17.9; m/z (FAB) 206, 121.

2-Bromo-*N*-(4-methoxy-benzyl)-2-methyl-propionamide (13a)

White solid, mp 48-50°C, yield (51%), R_f (9:1 petrol:ethyl acetate) 0.23; $\nu_{\max}$ (film)/ cm^{-1} 3299, 2974, 2935, 2836, 1638, 1532, 1250, 1108, 1030, 821; δ_H (300 MHz, CDCl_3) 7.20 (2H, d, J 8.4 Hz), 6.99 (1H, br s), 6.87 (2H, d, J 8.4 Hz), 4.37 (2H, d, J 5.7), 3.76 (3H, s), 1.97 (6H, s); δ_C (100.6 MHz, CDCl_3) 171.9, 159.0, 129.8, 128.9, 114.2, 62.8, 55.3, 43.9, 32.6;

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N-Buta-1,3-dienyl-2,2-dichloro-*N*-(4-methoxy-benzyl)-acetamide (**13b**)

Impure yellow oil, 17% (based upon NMR). δ_{H} Discernible data (400 MHz, CDCl_3) 7.10 (2H, d, J 8.3 Hz), 6.88 (2H, d, J 8.3 Hz), 6.78 (1H, br s), 5.96 (1H, s), 4.44 (2H, d, J 5.2 Hz), 3.81 (3H, s); discernible data δ_{C} (100.6 MHz, CDCl_3) 164.0, 159.4, 129.2, 128.8, 114.4, 66.4, 55.3, 43.8

4-(3-Bromo-propenyl)-1-(4-methoxy-benzyl)-3,3-dimethyl-azetidin-2-one (**14**)

Colourless oil, yield 60%, R_{f} (1:1 petrol:ethyl acetate) 0.49; ν_{max} (film)/ cm^{-1} 2961, 2927, 1740, 1612, 1585, 1512, 1461, 1397, 1360, 1302, 1243, 1205, 1175, 1109, 1032, 970, 827, 627; δ_{H} (300 MHz, CDCl_3) 7.15 (2H, d, J 8.6 Hz), 6.86 (2H, d, J 8.6 Hz), 5.85 (1H, dt, J 15.3, 7.4 Hz), 5.59 (1H, dd, J 15.3, 8.4 Hz), 4.56 (1H, d, J 14.7 Hz), 3.94 (1H, d, J 14.7 Hz), 3.92 (2H, d, J 7.4 Hz), 3.80 (3H, s), 3.54 (1H, d, J 8.4 Hz), 1.25 (3H, s), 1.11 (3H, s); δ_{C} (75.5 MHz, CDCl_3) 173.7, 159.5, 132.3, 131.3, 130.2, 128.3, 114.5, 63.8, 55.8, 55.7, 44.2, 31.6, 22.3, 18.1; m/z (FAB) 338 (MH)⁺, 258, 154, 121; [Found: (MH)⁺, 338.0749, $\text{C}_{16}\text{H}_{20}\text{NO}_2\text{Br}$ requires (MH)⁺, 338.0756]; [Found: C, 57.1; H, 6.1; N, 4.0. $\text{C}_{16}\text{H}_{20}\text{NO}_2\text{Br}$ requires C, 56.8; H, 6.0; N, 4.1].

2:1 Mixture of E-4-(3-bromo-2-methyl-propenyl)-1-(4-methoxy-benzyl)-3,3-dimethyl-azetidin-2-one (**15**) and *Z*-4-(3-bromo-2-methyl-propenyl)-1-(4-methoxy-benzyl)-3,3-dimethyl-azetidin-2-one (**15**)

Inseparable 2:1 mixture of *E*-4-(3-bromo-2-methyl-propenyl)-1-(4-methoxy-benzyl)-3,3-dimethyl-azetidin-2-one (**15**) and *Z*-4-(3-bromo-2-methyl-propenyl)-1-(4-methoxy-benzyl)-3,3-dimethyl-azetidin-2-one (**15**) as a pale yellow oil (57%), R_{f} (1:1 petrol:ethyl acetate) 0.50; ν_{max} (film)/ cm^{-1} 2962, 2924, 2836, 1746, 1612, 1513, 1440, 1400, 1304, 1246, 1177, 1111, 1033; δ_{H} (400 MHz, CDCl_3) 7.13 (3H, d, J 8.5 Hz, *E* and *Z*), 6.86 (3H, d, J 8.5 Hz, *E* and *Z*), 5.46

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(1H, d, J 8.3 Hz, *E*), 5.24 (0.5H, dd, J 9.8, 1.5 Hz, *Z*), 4.54 (1.5H, d, J 14.8 Hz, *E* and *Z*), 3.92 (1.5H, d, J 14.8 Hz, *E* and *Z*), 3.91 (3H, s, *E* and *Z*), 3.79 (4.5H, s, *E* and *Z*), 3.77 (1H, d, J 9.3 Hz, *E*), 3.74 (0.5H, d, J 9.3 Hz, *Z*), 1.88 (1.5H, d, J 1.5 Hz, *Z*), 1.66 (3H, d, J 1.5 Hz, *E*), 1.30 (1.5H, s, *Z*), 1.28 (3H, s, *E*), 1.11 (1.5H, s, *Z*), 1.10 (3H, s, *E*); δ_C (100.6 MHz, CDCl₃) 173.2, 159.1, 138.6, 129.7, 128.0, 127.9, 126.8, 126.2, 114.1, 59.9, 59.7, 55.3, 55.2, 43.7, 39.5, 22.4, 22.2, 22.1, 18.0, 17.9, 15.2; *m/z* (FAB) 352 (MH)⁺, 272, 244, 188, 162, 154, 121; [Found: (MH)⁺, 352.0903, C₁₇H₂₂NO₂Br requires (MH)⁺, 352.0912]; [Found: C, 58.3; H, 6.3; N, 3.9. C₁₇H₂₂NO₂Br requires C, 58.0; H, 6.3; N, 4.0].

1:1 Mixture of 4-(3-bromo-but-1-enyl)-1-(4-methoxy-benzyl)-3,3-dimethyl-azetidin-2-one (16), 4-Buta-1,3-dienyl-1-(4-methoxy-benzyl)-3,3-dimethyl-azetidin-2-one (17), and 1-(4-methoxy-benzyl)-3,3-dimethyl-(3-oxo-but-1-enyl)-azetidin-2-one (18)

A 1:1 mixture of diastereomers of 4-(3-bromo-but-1-enyl)-1-(4-methoxy-benzyl)-3,3-dimethyl-azetidin-2-one (**16**) as a pale yellow oil (30%), *R_f* (3:1 petrol:ethyl acetate) 0.15; $\nu_{\max}$ (film)/cm⁻¹ 2961, 2924, 2837, 1742, 1639, 1611, 1586, 1512, 1460, 1397, 1302, 1244, 1175, 1131, 1032, 970, 825, 760, 626; δ_H (400 MHz, CDCl₃) 7.16 (2H, d, J 8.6 Hz, isomer A or B), 7.14 (2H, d, J 8.6 Hz, isomer A or B), 6.87 (2H, d, J 8.6 Hz, isomer A or B), 6.86 (2H, d, J 8.6 Hz, isomer A or B), 5.91-5.82 (2H, m, isomers A and B), 5.52-5.43 (2H, m, isomers A and B), 4.64 (2H, dq, J 15.3, 8.5 Hz isomers A and B), 4.52 (1H, d, J 14.6 Hz, isomer A or B), 4.51 (1H, d, J 14.6 Hz, isomer A or B), 4.00 (1H, d, J 14.6 Hz, isomer A or B), 3.97 (1H, d, J 14.6 Hz, isomer A or B), 3.80 (6H, s, isomers A and B), 3.53 (1H, d, J 8.0 Hz, isomer A or B), 3.51 (1H, d, J 8.3 Hz, isomer A or B), 1.74 (3H, d, J 6.8 Hz, isomer A or B), 1.73 (3H, d, J 6.8 Hz, isomer A or B), 1.26 (3H, s, isomer A or B), 1.25 (3H, s, isomer A or B), 1.11 (3H, s, isomer A or B) and 1.10 (3H, s, isomer A or B); δ_C (100.6 MHz, CDCl₃) 173.3 (isomer A or B), 173.2 (isomers A or B), 159.1 (isomers A and B), 138.4 (isomer A or B), 138.1 (isomer A or B), 129.9 (isomers A and B), 127.9 (isomers A and B), 126.9 (isomers A and B), 114.1 (isomers A and B), 63.5 (isomers A and B), 55.3 (isomers A and B), 55.2 (isomers A and B), 47.6 (isomer A or B), 47.4 (isomer A or B), 43.8 (isomers A and B), 25.7 (isomer A or B), 25.5 (isomer A or B), 21.9 (isomers A and B), 17.4 (isomers A and B); *m/z* (FAB) 352 (MH)⁺, 307, 289, 272, 242, 176, 162, 154, 136, 121; [Found: (MH)⁺ 352.0896, C₁₇H₂₂NO₂Br requires (MH)⁺ 352.0912]; [Found: C, 58.3; H, 6.3; N, 3.8. C₁₇H₂₂NO₂Br requires C, 58.0; H, 6.3; N, 4.0].

4-Buta-1,3-dienyl-1-(4-methoxy-benzyl)-3,3-dimethyl-azetidin-2-one (**17**) as a pale yellow oil (19%), *R_f* (3:1 petrol:ethyl acetate) 0.20; $\nu_{\max}$ (film)/cm⁻¹ 2961, 2928, 2837, 1741, 1612, 1586, 1512, 1461, 1397, 1348, 1302, 1244,

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1175, 1129, 1110, 1032, 1009, 954, 906, 827, 765, 630; δ_{H} (400 MHz, CDCl_3) 7.14 (2H, d, J 8.8 Hz), 6.85 (2H, d, J 8.8 Hz), 6.33 (1H, ddd, J 17.0, 10.3, 10.3 Hz), 6.16 (1H, dd, J 15.2, 10.3 Hz), 5.52 (1H, dd, J 15.2, 8.6 Hz), 5.22 (1H, dd, J 17.0, 1.5 Hz), 5.14 (1H, dd, J 10.3, 1.5 Hz), 4.59 (1H, d, J 14.8 Hz), 3.89 (1H, d, J 14.8 Hz), 3.80 (3H, s), 3.56 (1H, d, J 8.6 Hz), 1.25 (3H, s), 1.11 (3H, s). δ_{C} (100.6 MHz, CDCl_3) 173.6, 159.0, 135.9, 135.8, 127.7, 129.0, 128.2, 118.5, 114.1, 64.4, 55.4, 55.3, 43.6, 21.9, 18.0; m/z (FAB) 543 (2MH)⁺, 272 (MH)⁺, 154, 136, 121; [Found: (MH)⁺ 272.1652, $\text{C}_{17}\text{H}_{21}\text{NO}_2$ requires (MH)⁺ 272.1651].

1-(4-Methoxy-benzyl)-3,3-dimethyl-(3-oxo-but-1-enyl)-azetidin-2-one (18) as a pale yellow oil (8%) contaminated with trace amount of impurity, R_{f} (1:1 petrol:ethyl acetate) 0.23; ν_{max} (film)/ cm^{-1} 2962, 2928, 2838, 1744, 1675, 1612, 1585, 1513, 1462, 1399, 1358, 1302, 1247, 1176, 1111, 1031, 985, 839, 761; δ_{H} (400 MHz, CDCl_3) 7.13 (2H, d, J 8.8 Hz), 6.85 (2H, d, J 8.8 Hz), 6.47 (1H, dd, J 16.0, 7.8 Hz), 6.11 (1H, d, J 16.0 Hz), 4.54 (1H, d, J 14.8 Hz), 4.05 (1H, d, J 14.8 Hz), 3.80 (3H, s), 3.68 (1H, d, J 7.8 Hz), 2.19 (3H, s), 1.30 (3H, s), 1.13 (3H, s); δ_{C} (100.6 MHz, CDCl_3) 197.2, 173.0, 159.3, 142.2, 133.6, 129.9, 127.6, 114.2, 63.2, 56.5, 55.3, 44.3, 27.2, 22.0, 17.9. m/z (FAB) 288 (MH)⁺, 154, 137, 121; [Found: (MH)⁺ 288.1594, $\text{C}_{17}\text{H}_{21}\text{NO}_3$ requires (MH)⁺ 288.1600].

1-(4-Methoxy-benzyl)-3,3-dimethyl-4-(3-methyl-buta-1,3-dienyl)-azetidin-2-one (19) and *1-(4-methoxy-benzyl)-3,3-dimethyl-4-(3-methyl-but-1-enyl)-azetidin-2-one dimer (20)*

1-(4-Methoxy-benzyl)-3,3-dimethyl-4-(3-methyl-buta-1,3-dienyl)-azetidin-2-one (19) as a pale yellow oil (32%), R_{f} (3:1 petrol:ethyl acetate) 0.27; ν_{max} (film)/ cm^{-1} 2962, 2929, 2838, 1730, 1675, 1611, 1585, 1512, 1461, 1401, 1357, 1302, 1245, 1175, 1109, 1031, 977, 836, 762, 735, 632; δ_{H} (400 MHz, CDCl_3) 7.14 (2H, d, J 8.8 Hz), 6.85 (2H, d, J 8.8 Hz), 6.23 (1H, d, J 15.8 Hz), 5.41 (1H, dd, 15.8, 8.5 Hz), 5.01 (1H, br s), 4.97 (1H, br s), 4.52 (1H, d, J 14.8 Hz), 3.98 (1H, d, J 14.8 Hz), 3.78 (3H, s), 3.61 (1H, d, J 8.8 Hz), 1.79 (3H, s), 1.26 (3H, s), 1.11 (3H, s); δ_{C} (100.6 MHz, CDCl_3) 173.5, 159.0, 141.0, 137.8, 129.7, 128.3, 125.0, 117.6, 114.0, 64.8, 55.3, 55.2, 43.6, 21.9, 18.3, 17.5; m/z (FAB) 286 (MH)⁺, 218, 180, 154, 136, 121; [Found: (MH)⁺ 286.1810, $\text{C}_{18}\text{H}_{23}\text{NO}_2$ requires (MH)⁺ 286.1807].

1-(4-Methoxy-benzyl)-3,3-dimethyl-4-(3-methyl-but-1-enyl)-azetidin-2-one dimer (20) as a pale yellow oil 1:1 mixture of diastereomers (14%), R_{f} (1:1 petrol:ethyl acetate) 0.24; ν_{max} (film)/ cm^{-1} 2962, 2926, 2838, 1741, 1660, 1612, 1586, 1512, 1462, 1399, 1359, 1302, 1244, 1175, 1135, 1106, 1032, 983, 827, 762, 633; δ_{H} (400 MHz,

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CDCl₃) 7.03 (8H, d, J 8.3 Hz, isomers A and B), 6.76 (8H, d, J 8.3 Hz isomers A and B), 5.58 (2H, d, J 15.8 Hz, isomer A), 5.57 (2H, d, J 15.8 Hz, isomer B), 5.09 (4H, dd, J 15.8, 8.5 Hz, isomers A and B), 4.45 (4H, d, J 14.8 Hz, isomers A and B), 3.86 (2H, d, J 14.8 Hz, isomer A or B), 3.83 (2H, d, J 14.8 Hz, isomer A or B), 3.71 (12H, s, isomers A and B), 3.44 (2H, d, J 8.5 Hz, isomer A or B), 3.43 (2H, d, J 8.5 Hz, isomer A or B), 1.18 (6H, s), 1.17 (6H, s), 1.02 (6H, s), 1.01 (6H, s), 0.84 (12H, s), 0.80 (6H, s), 0.79 (6H, s); δ_C (100.6 MHz, CDCl₃) 172.6, 158.0, 142.9, 142.8, 128.6, 127.2, 122.6, 113.0, 64.2, 54.2, 53.7, 42.5, 40.0, 21.0, 20.9, 20.0, 15.8; m/z (FAB) 573 (MH)⁺, 286, 154, 121; [Found: (MH)⁺, 573.3681, C₃₆H₄₈N₂O₄ requires (MH)⁺, 573.3692].

N-Buta-1,3-dienyl-2,2,2-trichloro-*N*-(4-methoxy-benzyl)-acetamide (21a)

Yield 16%, pale yellow oil, R_f (9:1 petrol:ethyl acetate) 0.31; $\nu_{\max}$ (film)/cm⁻¹ 2935, 2837, 1684, 1638, 1613, 1513, 1431, 1368, 1298, 1247, 1202, 1177, 1034, 997, 922, 811, 666; δ_H (400 MHz, CDCl₃) 7.40 (1H, d, J 13.6 Hz), 7.13 (2H, d, J 8.8 Hz), 6.86 (2H, d, J 8.8 Hz), 6.27 (1H, ddd, J 16.8, 10.7, 10.3 Hz), 5.83 (1H, dd, J 13.6, 10.7 Hz), 5.12 (1H, d, J 16.8 Hz), 5.05 (1H, d, J 10.3 Hz), 4.97 (2H, br s), 3.76 (3H, s); δ_C (100.6 MHz, CDCl₃) 159.1, 159.0, 134.2, 130.9, 127.8, 127.5, 117.1, 116.9, 114.3, 92.5, 55.3, 50.6; m/z (FAB) 307, 154, 136, 121.

N-Buta-1,3-dienyl-2,2-dichloro-*N*-(4-methoxy-benzyl)-acetamide (21b)

Yellow oil as a 5:1 mixture of rotamers. R_f (9:1 petrol:ethyl acetate) 0.38; $\nu_{\max}$ (film)/cm⁻¹ 3005, 2934, 2836, 1680, 1639, 1612, 1513, 1431, 1386, 1356, 1293, 1247, 1176, 1114, 1033, 998, 918, 808, 684; δ_H Discernible data for major rotamer (400 MHz, CDCl₃) 7.14 (2H, d, J 8.3 Hz), 6.91 (1H, d, J 13.6 Hz), 6.86 (2H, d, J 8.3 Hz), 6.42 (1H, s), 6.28 (1H, ddd, J 17.0, 10.8, 10.0 Hz), 5.91 (1H, dd, J 13.6, 10.8 Hz), 5.16 (1H, d, J 17.0 Hz), 5.09 (1H, d, J 10.0 Hz), 4.84 (2H, s), 3.78 (3H, s); discernible data for major rotamer δ_C (100.6 MHz, CDCl₃) 162.5, 159.0, 133.7, 128.9, 128.3, 127.7, 119.2, 117.6, 114.2, 65.2, 55.3, 48.5; m/z (FAB) 248, 212, 176, 154, 121.

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2,2,2-Trichloro-N-(4-methoxy-benzyl)-N-(4-methyl-penta-1,3-dienyl)-acetamide (21c)

Pale yellow gum (71%), R_f (9:1 petrol:ethyl acetate) 0.47; $\nu_{\max}$ (film)/ cm^{-1} 2962, 2911, 2837, 1680 1614, 1514, 1445, 1393, 1345, 1301, 1248, 1205, 1178, 1036, 925, 835, 807, 690, 664; δ_H (400 MHz, d^8 -toluene) 7.28 (1H, d, J 13.6 Hz), 7.16 (2H, d, J 8.3 Hz), 6.69 (2H, d, J 8.3 Hz), 5.99 (1H, br t, J 11.3 Hz), 5.72 (1H, d, J 10.8 Hz), 4.80 (2H, br s), 3.35 (3H, s), 1.61 (3H, s), 1.48 (3H, s). δ_C (100.6 MHz, d^8 -toluene) 159.4, 158.9, 137.5, 135.0, 128.6, 128.5, 122.7, 114.5, 114.2, 94.0, 54.7, 50.9, 25.9, 20.3. m/z (FAB) 363 (MH)⁺, 329, 307, 154, 121.

3,3-Dichloro-4-(3-chloro-propenyl)-1-(4-methoxy-benzyl)-azetidin-2-one (22a) and 3-[3,3-dichloro-1-(4-methoxy-benzyl)-4-oxo-azetidin-2-yl]-propenal (23)

(22)

(23)

3,3-Dichloro-4-(3-chloro-propenyl)-1-(4-methoxy-benzyl)-azetidin-2-one (22) as a pale yellow gum (23%), R_f (3:1 petrol:ethyl acetate) 0.33; $\nu_{\max}$ (film)/ cm^{-1} 3005, 2932, 2838, 1784, 1611, 1513, 1439, 1393, 1360, 1304, 1247, 1177, 1144, 1032, 971, 852, 678; δ_H (400 MHz, CDCl_3) 7.14 (2H, d, J 8.5 Hz), 6.88 (2H, d, J 8.5 Hz), 5.95 (1H, dt, J 15.2, 6.5 Hz), 5.62 (1H, ddt, J 15.2, 8.8, 1.2 Hz), 4.62 (1H, d, J 14.8 Hz, $\text{CH}_a\text{H}_b\text{N}$), 4.23 (1H, d, J 8.8 Hz, CCl_2CHN), 4.06 (1H, d, J 14.8 Hz, $\text{CH}_a\text{H}_b\text{N}$), 4.08-4.05 (2H, m), 3.81 (3H, s); δ_C (100.6 MHz, CDCl_3) 160.8, 159.7, 135.8, 130.0, 126.7, 125.5, 114.5, 83.5, 70.5, 55.3, 44.7, 43.0; m/z (FAB) 334 (MH)⁺, 307, 289, 163, 154, 137; [Found: (M-H)⁺, 332.0004, $\text{C}_{14}\text{H}_{14}\text{NO}_2\text{Cl}_3$ requires (M-H)⁺, 332.0012].

3-[3,3-Dichloro-1-(4-methoxy-benzyl)-4-oxo-azetidin-2-yl]-propenal (23) as a pale yellow gum (12%), R_f (3:1 petrol:ethyl acetate) 0.17; $\nu_{\max}$ (film)/ cm^{-1} 2934, 2838, 1788, 1735, 1694., 1611, 1514, 1394, 1351, 1249, 1179, 1128, 1031, 978, 867; δ_H (400 MHz, CDCl_3) 9.55 (1H, d, J 7.4 Hz), 7.12 (2H, d, J 8.8 Hz), 6.88 (2H, d, J 8.8 Hz), 6.47 (1H, dd, J 15.8, 8.3 Hz), 6.24 (1H, dd, J 15.8, 7.4 Hz), 4.64 (1H, d, J 14.8 Hz), 4.43 (1H, d, J 8.3 Hz), 4.16 (1H, d, J 14.8 Hz), 3.81 (3H, s); δ_C (100.6 MHz, CDCl_3) 191.5, 160.3, 160.0, 145.6, 137.6, 130.0, 125.0, 114.6, 83.4, 69.5, 55.4, 45.3; m/z (FAB) 314 (MH)⁺, 313 (M)⁺, 307, 289, 154, 136, 121; [Found: (M)⁺, 313.0269, $\text{C}_{14}\text{H}_{13}\text{NO}_3\text{Cl}_2$ requires (M)⁺, 313.0272].

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3,3-Dichloro-1-(4-methoxy-benzyl)-4-(3-methyl-buta-1,3-dienyl)-azetidin-2-one (24) and 3,3-dichloro-1-(4-methoxy-benzyl)-4-(3-methyl-but-1-enyl)-azetidin-2-one dimer (25)

3,3-Dichloro-1-(4-methoxy-benzyl)-4-(3-methyl-buta-1,3-dienyl)-azetidin-2-one (**24**) as a pale yellow oil (41%), R_f (9:1 petrol:ethyl acetate) 0.22; $\nu_{\max}$ (film)/ cm^{-1} 3082, 2927, 2838, 1780, 1644, 1610, 1586, 1512, 1436, 1392, 1351, 1303, 1245, 1176, 1150, 1109, 1032, 970, 902, 866, 834, 805, 755, 680, 635; δ_H (400 MHz, CDCl_3) 7.14 (2H, d, J 8.8 Hz), 6.87 (2H, d, J 8.8 Hz), 6.37 (1H, d, J 15.6 Hz), 5.38 (1H, dd, J 15.6, 8.8 Hz), 5.13 (1H, br s), 5.08 (1H, br s), 4.56 (1H, d, J 14.8 Hz), 4.30 (1H, d, J 8.8 Hz), 4.09 (1H, d, J 14.8 Hz), 3.79 (3H, s), 1.82 (3H, s); δ_C (100.6 MHz, CDCl_3) 160.8, 159.9, 141.7, 140.5, 129.9, 126.0, 121.2, 120.1, 114.4, 84.5, 71.9, 55.3, 44.5, 18.0; m/z (FAB) 326 (MH)⁺, 289, 163, 154, 136, 121; [Found: (MH)⁺, 326.0696, $\text{C}_{16}\text{H}_{17}\text{NO}_2\text{Cl}_2$ requires (MH)⁺, 326.0715].

1:1 Mixture 3,3-Dichloro-1-(4-methoxy-benzyl)-4-(3-methyl-but-1-enyl)-azetidin-2-one dimer (**25**) as a pale yellow oil (14%), R_f (2:1 petrol:ethyl acetate) 0.41; $\nu_{\max}$ (film)/ cm^{-1} 2969, 2837, 1783, 1704, 1659, 1611, 1586, 1513, 1461, 1394, 1355, 1303, 1247, 1176, 1111, 1032, 983, 918, 832, 732, 684, 645; δ_H (400 MHz, CDCl_3) 7.10 (8H, d, J 8.8 Hz, isomers A and B), 6.86 (8H, d, J 8.8 Hz, isomers A and B), 5.84 (2H, d, J 15.8 Hz, isomer A), 5.82 (2H, d, J 15.8 Hz, isomer B), 5.18 (2H, dd, J 15.8, 8.8 Hz, isomer A or B), 5.17 (2H, dd, J 15.8, 8.8 Hz, isomer A or B), 4.57 (2H, d, J 14.8 Hz, isomer A or B), 4.55 (2H, d, J 14.8 Hz, isomer A or B), 4.20 (4H, d, J 8.8 Hz, isomers A and B), 4.06 (2H, d, J 14.8 Hz, isomer A or B), 4.05 (2H, d, J 14.8 Hz, isomer A or B), 3.80 (12H, s, isomers A and B), 0.96 (6H, s, isomer A or B), 0.95 (6H, s, isomer A or B), 0.91 (12H, s, isomer A and B); δ_C (100.6 MHz, CDCl_3) 160.9, 159.6, 148.0, 129.7, 125.8, 121.3, 114.4, 84.1, 72.2, 55.3, 44.5, 41.5, 22.7, 22.4, 22.2, 21.6; m/z (FAB) 653 (MH)⁺, 617, 391, 327, 292, 256, 162, 154; [Found: (MH)⁺, 655.1460, $\text{C}_{32}\text{H}_{36}\text{N}_2\text{O}_4$ $^{35}\text{Cl}_3$ ^{37}Cl requires (MH)⁺, 655.1478].

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2,2,2-Trichloro-N-(4-methoxy-benzyl)-N-(2,6,6-trimethyl-cyclohex-2-enyldenemethyl)-acetamide (26)

Colourless oil, yield 76%, R_f (9:1 petrol:ethyl acetate) 0.40; $\nu_{\max}$ (film)/ cm^{-1} 3010, 2953, 2922, 2837, 1680, 1612, 1510, 1460, 1437, 1377, 1314, 1301, 1247, 1172, 1154, 1107, 1084, 1031, 926, 857, 811, 803, 780, 758, 686, 661; δ_H (400 MHz, CDCl_3) 7.19 (2H, d, J 8.5 Hz), 6.83 (2H, d, J 8.5 Hz), 6.27 (1H, s), 5.74 (1H, br s), 5.70-5.00 (1H, br s), 4.40-3.60 (1H, br s), 3.78 (3H, s), 2.25-2.15 (2H, m), 1.99 (3H, s), 1.48 (2H, t, J 6.3 Hz), 1.03 (6H, s); δ_C (100.6 MHz, CDCl_3) 159.3, 159.2, 143.1, 131.9, 129.9, 129.3, 128.3, 120.5, 113.8, 93.0, 55.9, 55.2, 40.5, 36.6, 27.8, 23.8, 21.2; m/z (FAB) 416 (MH^+), 346, 121; [Found: (M-H^+), 414.0784, $\text{C}_{20}\text{H}_{24}\text{NO}_2\text{Cl}_3$ requires (M-H^+), 414.0794]; [Found: C, 58.0; H, 6.0; N, 3.1. $\text{C}_{20}\text{H}_{24}\text{NO}_2\text{Cl}_3$ requires C, 57.6; H, 5.8; N, 3.4].

4,4,5-Trichloro-2-(4-methoxy-benzyl)-4a,8,8-trimethyl-4,4a,5,6,7,8-hexahydro-2H-isoquinoline-3-one (27) and 3,3-dichloro-1-(4-methoxy-benzyl)-4-(2,6,6-trimethyl-cyclohexa-1,3-dienyl)-azetidin-2-one (28)

(27)

(28)

4,4,5-Trichloro-2-(4-methoxy-benzyl)-4a,8,8-trimethyl-4,4a,5,6,7,8-hexahydro-2H-isoquinoline-3-one (27) as a pale yellow oil, yield 18%, R_f (5:1 petrol:ethyl acetate) 0.34; $\nu_{\max}$ (film)/ cm^{-1} 2955, 1785, 1692, 1612, 1512, 1459, 1411, 1386, 1331, 1298, 1245, 1175, 1113, 1032, 968, 819, 762, 734, 640, 615; δ_H (400 MHz, CDCl_3) 7.21 (2H, d, J 8.8 Hz), 6.86 (2H, d, J 8.8 Hz), 5.96 (1H, s), 4.81 (1H, dd, J 10.8, 5.5 Hz), 4.73 (1H, d, J 14.6 Hz), 4.64 (1H, d, J 14.6 Hz), 3.80 (3H, s), 2.20-2.03 (2H, m), 1.52-1.45 (2H, m), 1.31 (3H, s), 1.13 (3H, s), 1.10 (3H, s); δ_C (125.8 MHz, CDCl_3) 160.6, 159.4, 132.1, 129.3, 127.8, 123.4, 114.2, 93.2, 61.0, 55.3, 51.8, 50.7, 37.1, 34.1, 31.5, 31.3, 31.2, 18.7; m/z (FAB) 416 (MH^+), 380, 307, 154, 121; [Found: (M-H^+), 414.0774, $\text{C}_{20}\text{H}_{24}\text{NO}_2\text{Cl}_3$ requires (M-H^+), 414.0794],

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3,3Dichloro-1-(4-methoxy-benzyl)-4-(2,6,6-trimethyl-cyclohexa-1,3-dienyl)-azetidin-2-one (28) as a Pale yellow oil, yield 13%, R_f (5:1 petrol:ethyl acetate) 0.45; $\nu_{\max}$ (film)/ cm^{-1} 3037, 2925, 2856, 1785, 1612, 1513, 1462, 1362, 1304, 1249, 1176, 1150, 1111, 1033, 871, 822, 658; δ_H (400 MHz, CDCl_3) 7.15 (2H, d, J 8.5 Hz), 6.88 (2H, d, J 8.5 Hz), 5.83 (1H, ddd, J 9.5, 5.5, 3.0 Hz), 5.75 (1H, dd, J 9.5, 3.0 Hz), 4.90 (1H, d, J 14.6 Hz), 4.36 (1H, s), 3.81 (3H, s), 3.65 (1H, d, J 14.6 Hz), 2.20 (1H, dt, J 17.2, 3.0 Hz), 1.93 (1H, dd, J 17.2, 5.5 Hz), 1.86 (3H, s), 1.00 (3H, s), 0.71 (3H, s); δ_C (125.8 MHz, CDCl_3) 161.3, 159.7, 132.5, 130.6, 130.4, 128.5, 126.8, 125.4, 114.5, 85.1, 69.3, 55.3, 45.0, 39.2, 33.7, 27.0, 24.8, 18.7; m/z (FAB) 380 (MH^+), 344, 307, 289, 154, 121; [Found: (M-H^+), 378.1024, $\text{C}_{20}\text{H}_{23}\text{NO}_2\text{Cl}_2$ requires (M-H^+), 378.1028].

N-Allyl-2-bromo-N-buta-1,3-dienyl-2-methyl-propionamide (29)

Yellow oil, R_f (9:1 petrol:ethyl acetate) 0.45; $\nu_{\max}$ (film)/ cm^{-1} 3086, 2980, 2932, 1662, 1631, 1603, 1459, 1426, 1391, 1362, 1310, 1257, 1220, 1153, 1109, 996, 928, 891, 780, 649; δ_H (400 MHz, CDCl_3) 7.63 (1H, d, J 13.8 Hz), 6.34 (1H, ddd, J 16.8, 10.6, 10.6 Hz), 5.86-5.73 (2H, m), 5.19 (1H, d, J 10.8 Hz), 5.15 (1H, d, J 16.8 Hz), 5.12 (1H, d, J 17.1 Hz), 5.02 (1H, d, J 10.6 Hz), 4.38 (2H, br s), 2.01 (6H, s); δ_C (100.6 MHz, CDCl_3) 168.7, 134.9, 132.5, 132.0, 116.2, 115.1, 113.7, 56.6, 48.2, 32.6; m/z (FAB) 258 (MH^+), 257 (M^+), 232, 194, 180, 154, 136, 121; [Found: (M^+) 257.0431, $\text{C}_{11}\text{H}_{16}\text{NOBr}$ requires (M^+) 257.0415].

N-Allyl-2-bromo-2-methyl-N-(2-methyl-propenyl)-propionamide (30)

Pale yellow oil, yield 75%, R_f (9:1 petrol:ethyl acetate) 0.23; $\nu_{\max}$ (film)/ cm^{-1} 3079, 2974, 2929, 1637, 1463, 1389, 1365, 1342, 1276, 1180, 1107, 1053, 991, 918, 829, 778, 751, 635; δ_H (400 MHz, CDCl_3) 6.40 (1H, br s), 5.83 (1H, ddt, J 17.3, 10.3, 5.6 Hz), 5.16 (1H, dq, J 17.3, 1.8 Hz), 5.12 (1H, dq, J 10.3, 1.8 Hz), 4.06 (2H, br d, J 5.6 Hz), 1.93 (6H, s), 1.76 (3H, d, J 1.5 Hz), 1.64 (3H, d, J 1.5 Hz); δ_C (100.6 MHz, CDCl_3) 170.3, 134.5, 132.6, 125.8, 116.5, 58.2, 54.1, 32.0, 21.7, 18.6; m/z (FAB) 262 (MH^+), 260 (MH^+), 180, 154, 136; [Found: (MH^+), 260.0636,

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C₁₁H₁₈NOBr requires (MH)⁺, 260.0650]; [Found: C, 50.5; H, 7.0; N, 5.3. C₁₁H₁₈NOBr requires C, 50.8; H, 7.0; N, 5.4].

4-Bromomethyl-1-buta-1,3-dienyl-3,3-dimethyl-pyrrolidin-2-one (31)

White solid (91%), R_f (9:1 petrol:ethyl acetate) 0.22; m.pt. 77.0-78.0°C; $\nu_{\max}$ (film)/cm⁻¹ 3084, 3032, 2965, 2925, 2868, 1700 (C=O), 1641, 1604, 1480, 1424, 1392, 1378, 1360, 1328, 1278, 1234, 1151, 1038, 996, 935, 888, 746, 680, 656; δ_{H} (400 MHz, CDCl₃) 7.07 (1H, d, J 14.3 Hz), 6.35 (1H, ddd, J 16.8, 10.6, 10.6 Hz), 5.71 (1H, dd, J 14.3, 10.6 Hz), 5.16 (1H, d, J 16.8 Hz), 5.01 (1H, d, J 10.6 Hz), 3.77 (1H, dd, J 10.5, 7.8 Hz), 3.55 (1H, dd, J 10.4, 4.8 Hz), 3.34 (1H, dd, J 10.4, 10.4 Hz), 3.20 (1H, dd, J 10.5, 8.5 Hz), 2.53 (1H, dddd, J 10.4, 8.5, 7.8, 4.8 Hz), 1.26 (3H, s), 1.04 (3H, s); δ_{C} (100.6 MHz, CDCl₃) 176.8, 134.7, 126.4, 115.1, 113.3, 47.6, 45.6, 44.6, 31.2, 24.2, 18.3. m/z (FAB) 258 (MH)⁺, 257 (M)⁺, 154, 136; [Found: (M)⁺ 257.0407, C₁₁H₁₆NOBr requires (M)⁺ 257.0415].

4-Bromomethyl-3,3-dimethyl-1-(2-methyl-propenyl)-pyrrolidin-2-one (32)

Conditions, toluene, reflux. Colourless oil, yield 95%, R_f (3:1 petrol:ethyl acetate) 0.26; $\nu_{\max}$ (film)/cm⁻¹ 2967, 2929, 2871, 1697, 1408, 1361, 1279, 1239, 1165; δ_{H} (400 MHz, CDCl₃) 5.89 (1H, qq, J 1.5, 1.5 Hz), 3.71 (1H, dd, J 10.0, 7.6 Hz), 3.54 (1H, dd, J 10.0, 4.5 Hz), 3.34 (1H, dd, J 10.8, 10.0 Hz), 3.29 (1H, dd, J 10.0, 8.5 Hz), 2.50 (1H, dddd, J 10.8, 8.5, 7.6, 4.5 Hz), 1.75 (3H, d, J 1.2 Hz), 1.68 (3H, d, J 1.2 Hz), 1.24 (3H, s), 1.04 (3H, s); δ_{C} (100.6 MHz, CDCl₃) 177.8, 128.9, 119.1, 51.4, 46.5, 43.7, 31.4, 24.3, 23.1, 18.1, 18.0; m/z (FAB) 262 (MH)⁺, 260 (MH)⁺, 180, 154, 136; [Found: C, 50.7; H, 7.0; N, 5.2. C₁₁H₁₈NOBr requires C, 50.8; H, 7.0; N, 5.4].

Discernible data for **32** in mixture of **32** and **33**, colourless oil, yield 97% δ_{H} (400 MHz, CDCl₃) 5.75 (1H, m), 5.20 (2H, m), 4.19 (1H, dd, J 15.5, 5.5 Hz), 3.70 (1H, m), 3.69 (1H, s), 1.80 (6H, s), 1.30 (3H, s), 1.26 (3H, s), [Found: (MH)⁺ 260.0661, C₁₁H₁₈NOBr requires (MH)⁺ 260.0650]

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2,2,2-Trichloro-N-(1-cyclohex-1-enyl-vinyl)-N-(4-methoxy-benzyl)-acetamide (35a)

Pale yellow oil, yield 48%, R_f (9:1 petrol:ethyl acetate) 0.39; $\nu_{\max}$ (film)/ cm^{-1} 2929, 2835, 1671, 1610, 1511, 1438, 1302, 1245, 1175, 1114, 1033, 906, 842, 806, 759, 732, 662; δ_H (400 MHz, CDCl_3) 7.17 (2H, d, J 8.5 Hz), 6.73 (2H, d, J 8.5 Hz), 5.84 (1H, br), 5.40-5.10 (1H, br), 5.16 (1H, s), 4.69 (1H, s), 3.90-3.55 (1H, br), 3.68 (3H, s), 2.12-1.95 (4H, m), 1.70-1.40 (4H, m); δ_C (100.6 MHz, CDCl_3) 160.6 and 159.3 ($\text{C}=\text{O}$ and Ar quat.), 145.6 ($\text{NC}=\text{CH}_2$), 131.3, 129.0, 130.8, 127.8, 115.2, 113.6, 93.2, 55.2, 54.7, 25.6, 25.3, 22.5, 21.9; m/z (EI) 387 (M)⁺, 352, 340, 316, 281, 270, 231, 121, 91, 77, 65; [Found: (M)⁺, 387.0552, $\text{C}_{18}\text{H}_{20}\text{NO}_2\text{Cl}_3$ requires (M)⁺, 387.0560].

Mixture of 2-bromo-N-(1-cyclohex-1-enyl-vinyl)-N-(4-methoxy-benzyl)-2-methyl-propionamide (35b) and 4-bromo-1-cyclohex-1-enyl-1-(4-methoxy-benzylamino)-4-methyl-pent-1-en-3-one (B).

Colourless oil, yield 75% (as an inseparable mixture), R_f (9:1 petrol:ethyl acetate) 0.30; $\nu_{\max}$ (film)/ cm^{-1} 2927, 1635, 1592, 1510, 1459, 1388, 1365, 1305, 1244, 1173, 1094, 1034, 921, 813, 933, 630; δ_H (400 MHz, CDCl_3 , mixture) 10.8 (1H, br, **B**), 7.25 (2H, d, J 8.5 Hz, **35b**), 7.17 (2H, d, J 8.8 Hz, **B**), 6.86 (2H, d, J 8.8 Hz, **B**), 6.82 (2H, d, J 8.5 Hz, **35b**), 5.89 (1H, m, **35b**), 5.83 (1H, m, **B**), 5.46 (1H, s, **B**), 5.50-5.20 (2H, br s, **35b**), 5.21 (1H, s, **35b**), 4.91 (1H, s, **35b**), 4.36 (2H, d, J 6.3 Hz, **B**), 3.78 (3H, s, **35b**), 3.78 (3H, s, **B**), 2.20-2.08 (4H, m, **35b**), 2.20-2.08 (4H, m, **B**), 2.03-1.90 (6H, br, **35b**), 1.91 (6H, s, **B**), 1.75-1.55 (4H, m, **35b**), 1.75-1.55 (4H, m, **B**); m/z (EI) 391 (M)⁺, 312, 270, 242, 190, 121, 77; [Found: (M)⁺, 391.1152, $\text{C}_{20}\text{H}_{26}\text{NO}_2\text{Br}$ requires (M)⁺, 391.1147].

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2-Bromo-N-(1-cyclohex-1-enyl-vinyl)-N-(4-methoxy-benzyl)-propionamide (35c)

Colourless oil, yield 47%, R_f (5:1 petrol:ethyl acetate) 0.26; $\nu_{\max}$ (film)/ cm^{-1} 2927, 1659, 1607, 1510, 1441, 1400, 1244, 1174, 1033, 900, 850, 814, 730; δ_H (400 MHz, CDCl_3) 7.22 (2H, d, J 8.8 Hz), 6.82 (2H, d, J 8.8 Hz), 5.83 (1H, br m), 5.17 (1H, s), 4.73 (1H, br s), 4.50 (1H, br m), 3.78 (3H, s), 2.22-2.05 (4H, m), 1.76 (3H, d, J 6.5 Hz), 1.74-1.67 (2H, m), 1.63-1.55 (2H, m), CH_2N not visible; δ_C (100.6 MHz, CDCl_3) 169.9, 159.0, 146.2, 132.1, 29.7, 130.5, 128.2, 113.6, 113.0, 55.2, 50.5, 40.2, 25.6, 25.3, 22.5, 22.0, 22.4; m/z (EI) 378 (MH^+), 376 (MH^+), 314, 298, 242, 175, 150, 121, 77; [Found: (MH^+), 376.0906, $\text{C}_{19}\text{H}_{24}\text{NO}_2\text{Br}$ requires (MH^+), 376.0912].

Major 6-bromo-2-(4-methoxy-benzyl)-4,4-dimethyl-1-methylene-2-aza-spiro[4.5]decan-3-one (36a) and minor 6-bromo-2-(4-methoxy-benzyl)-4,4-dimethyl-1-methylene-2-aza-spiro[4.5]decan-3-one (36b)

Major product: 6-bromo-2-(4-methoxy-benzyl)-4,4-dimethyl-1-methylene-2-aza-spiro[4.5]decan-3-one (**36a**) as a pale yellow oil, yield 52%, R_f (5:1 petrol:ethyl acetate) 0.20; $\nu_{\max}$ (film)/ cm^{-1} 2934, 2865, 1715, 1660, 1630, 1513, 1444, 1398, 1347, 1298, 1246, 1175, 1108, 1031, 918, 818; δ_H (400 MHz, CDCl_3) 7.16 (2H, d, J 8.5 Hz), 6.82 (2H, d, J 8.5 Hz), 4.96 (1H, br d, J 15.1 Hz), 4.37 (1H, br s), 4.26 (1H, br d, J 15.1 Hz), 4.32-4.15 (1H, br m), 4.24 (1H, br s), 3.76 (3H, s), 2.41-2.27 (1H, br m), 2.25-2.15 (1H, br m), 2.14-2.06 (1H, br m), 1.89-1.81 (1H, br m), 1.66-1.58 (1H, br m), 1.56-1.43 (3H, br m), 1.59 (3H, br s), 1.21 (3H, s); δ_C (125.8 MHz, d^8 -toluene, 343K) 178.9, 159.9, 154.9, 137.6, 129.4, 114.8, 88.6, 61.8, 55.1, 52.3, 47.8, 44.7, 35.4, 32.3, 25.7, 22.8, 21.8, 21.7; m/z (FAB) 394 (MH^+), 392 (MH^+), 312, 154, 121; [Found: (MH^+), 394.1197, $\text{C}_{20}\text{H}_{26}\text{NO}_2^{81}\text{Br}$ requires (MH^+), 394.1204]

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Minor product: 6-bromo-2-(4-methoxy-benzyl)-4,4-dimethyl-1-methylene-2-aza-spiro[4.5]decan-3-one (**36b**) as a pale yellow oil, yield 20%, R_f (5:1 petrol:ethyl acetate) 0.13; $\nu_{\max}$ (film)/ cm^{-1} 2938, 2874, 1718, 1662, 1631, 1513, 1447, 1397, 1355, 1304, 1247, 1173, 1109, 1031, 821; δ_H (400 MHz, CDCl_3) 7.16 (2H, d, J 8.5 Hz), 6.83 (2H, d, J 8.5 Hz), 5.13 (1H, d, J 15.3 Hz), 4.56 (1H, d, J 2.5 Hz), 4.51 (1H, d, J 2.5 Hz), 4.06 (1H, d, J 15.3 Hz), 3.97 (1H, dd, J 12.0, 4.0 Hz), 3.78 (3H, s), 2.32-2.06 (3H, m), 1.82-1.68 (3H, m), 1.49-1.32 (2H, m), 1.28 (3H, s), 1.12 (3H, s); δ_C (100.6 MHz, CDCl_3) 179.7, 158.7, 150.4, 128.9, 128.5, 114.0, 91.0, 57.7, 55.2, 50.9, 47.6, 43.6, 34.1, 30.1, 27.0, 22.7, 24.8, 18.3; m/z (EI) 391 (M)⁺, 312, 121, 77; [Found: (M)⁺, 391.1151, $\text{C}_{20}\text{H}_{26}\text{NO}_2\text{Br}$ requires (M)⁺, 391.1147].

Major 6-Bromo-2-(4-methoxy-benzyl)-4-methyl-1-methylene-2-aza-spiro[4.5]decan-3-one (**37a**), minor 6-Bromo-2-(4-methoxy-benzyl)-4-methyl-1-methylene-2-aza-spiro[4.5]decan-3-one (**37b**), and 2-(4-methoxy-benzyl)-1,4-dimethyl-5,6,7,8-tetrahydro-2H-isoquinolin-3-one (**38**)

Major product: 6-bromo-2-(4-methoxy-benzyl)-4-methyl-1-methylene-2-aza-spiro[4.5]decan-3-one (**37a**) as a pale yellow oil, yield 45%, R_f (5:1 petrol:ethyl acetate) 0.15; $\nu_{\max}$ (film)/ cm^{-1} 2939, 2681, 1716, 1661, 1633, 1513, 1460, 1397, 1348, 1299, 1247, 1215, 1178, 1032, 818; δ_H (400 MHz, CDCl_3) 7.17 (2H, d, J 8.5 Hz), 6.82 (2H, d, J 8.5 Hz), 4.82 (1H, d, J 15.0 Hz), 4.38 (1H, d, J 15.0 Hz), 4.29 (1H, d, J 2.8 Hz), 4.16 (1H, d, J 2.8 Hz), 4.11 (1H, dd, J 12.6, 3.8 Hz), 3.78 (3H, s), 2.84 (1H, q, J 7.4 Hz), 2.28-2.20 (1H, m), 2.03-1.96 (1H, m), 1.96-1.87 (1H, m), 1.83-1.77 (1H, m), 1.74-1.68 (1H, m), 1.64-1.54 (1H, m), 1.50-1.40 (2H, m), 1.37 (3H, d, J 7.4 Hz); δ_C (125.8 MHz, CDCl_3) 177.9, 158.8, 154.5, 128.8, 128.1, 113.9, 84.9, 64.3, 60.4, 48.4, 43.6, 40.7, 34.9, 33.3, 27.1, 22.9, 14.5; m/z (EI) 377 (M)⁺, 298, 149, 121, 91, 78; [Found: (M -H)⁺, 376.0905, $\text{C}_{19}\text{H}_{24}\text{NO}_2\text{Br}$ requires (M -H)⁺, 376.0912].

Minor product: 2-(4-methoxy-benzyl)-4-methyl-1-methylene-2-aza-spiro[4.5]decan-3-one (**38b**) as a pale yellow oil, yield 22%, R_f (5:1 petrol:ethyl acetate) 0.13; $\nu_{\max}$ (film)/ cm^{-1} 2937, 1717, 1662, 1630, 1513, 1444, 1392, 1356, 1296, 1247, 1181, 1033, 821; δ_H (400 MHz, CDCl_3) 7.20 (2H, d, J 8.8 Hz), 6.83 (2H, d, J 8.8 Hz), 4.95 (1H, d, J 15.3 Hz), 4.57 (1H, d, J 2.5 Hz), 4.46 (1H, d, J 2.5 Hz), 4.29 (1H, d, J 15.3 Hz), 3.93 (1H, dd, J 12.3, 4.2 Hz), 3.78 (3H, s), 2.37 (1H, q, J 7.5 Hz), 2.31-2.19 (1H, m), 2.16-2.08 (1H, m), 2.06-1.99 (1H, m), 1.85-1.74 (2H, m), 1.74-1.61 (2H, m), 1.43-1.33 (1H, m), 1.34 (3H, d, J 7.5 Hz); δ_C (125.8 MHz, CDCl_3) 175.7, 158.8, 151.0, 129.1, 128.4,

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113.8, 89.7, 55.3, 55.2, 47.9, 47.6, 43.6, 36.5, 32.9, 26.7, 22.1, 7.6; m/z (EI) 377 (M)⁺, 376 (MH)⁺, 298, 162, 121, 91, 78; [Found: (M-H)⁺, 376.0920, C₁₉H₂₄NO₂Br requires (M-H)⁺, 376.0912].

2-(4-Methoxy-benzyl)-1,4-dimethyl-5,6,7,8-tetrahydro-2H-isoquinolin-3-one (**48**) as a pale yellow oil, yield 7%, R_f (1:1 petrol:ethyl acetate) 0.16; $\nu_{\max}$ (film)/cm⁻¹ 2932, 2862, 1632, 1574, 1532, 1512, 1445, 1246, 1177, 1033, 811; δ_H (300 MHz, CDCl₃) 7.10 (2H, d, J 8.6 Hz), 6.82 (2H, d, J 8.6 Hz), 5.36 (2H, br s), 3.77 (3H, s), 2.63-2.55 (2H, m), 2.50-2.42 (2H, m), 2.18 (3H, s), 2.12 (3H, s), 1.75-1.68 (4H, m); δ_C (125.8 MHz, CDCl₃) 162.6, 158.6, 146.8, 138.9, 129.5, 121.4, 113.7, 127.0, 114.1, 55.3, 48.5, 28.4, 27.0, 23.3, 22.5, 15.7, 12.4; m/z (EI) 297 (M)⁺, 242, 176, 161, 121, 91, 77; [Found: (M)⁺, 297.1740, C₁₉H₂₃NO₂ requires (M)⁺, 297.1729].

4-Chloro-2-(4-methoxy-benzyl)-1-methyl-5,6,7,8-tetrahydro-2H-isoquinolin-3-one (**39a**), 4-chloro-1-chloromethyl-2-(4-methoxy-benzyl)-5,6,7,8-tetrahydro-2H-isoquinolin-3-one (**39b**), major 4,4,6-trichloro-2-(4-methoxy-benzyl)-1-methylene-2-aza-spiro[4.5]decan-3-one (**40a**), minor 4,4,6-trichloro-2-(4-methoxy-benzyl)-1-methylene-2-aza-spiro[4.5]decan-3-one (**40b**)

4-Chloro-1-chloromethyl-2-(4-methoxy-benzyl)-5,6,7,8-tetrahydro-2H-isoquinolin-3-one (**39a**) as a pale yellow oil, yield 21%, R_f (1:1 petrol:ethyl acetate) 0.45; $\nu_{\max}$ (film)/cm⁻¹ 2935, 2864, 1640, 1578, 1511, 1446, 1247, 1177, 1033, 921, 732; δ_H (400 MHz, CDCl₃) 7.14 (2H, d, J 8.5 Hz), 6.84 (2H, d, J 8.5 Hz), 5.51 (2H, br s), 4.45 (2H, s), 3.77 (3H, s), 2.80-2.73 (2H, m), 2.67-2.60 (2H, m), 1.80-1.73 (4H, m); δ_C (100.6 MHz, CDCl₃) 159.2, 158.6, 147.6, 137.6, 128.7, 126.2, 117.5, 127.9, 114.4, 55.3, 47.5, 38.0, 28.7, 25.0, 22.3, 21.6; m/z (EI) 351 (M)⁺, 121, 77; [Found: (M)⁺, 351.0808, C₁₈H₁₉NO₂Cl₂ requires (M)⁺, 351.0793].

4-Chloro-2-(4-methoxy-benzyl)-1-methyl-5,6,7,8-tetrahydro-2H-isoquinolin-3-one (**39b**) as a pale yellow oil, yield 29%, R_f (1:1 petrol:ethyl acetate) 0.28; $\nu_{\max}$ (film)/cm⁻¹ 2936, 2865, 1721, 1644, 1586, 1512, 1441, 1344, 1248, 1178, 1033, 941, 818, 730, 653, 610; δ_H (400 MHz, CDCl₃) 7.13 (2H, d, J 8.8 Hz), 6.82 (2H, d, J 8.8 Hz), 5.38 (2H, br s), 3.76 (3H, s), 2.78-2.72 (2H, m), 2.48-2.42 (2H, m), 2.21 (3H, s), 1.77-1.69 (4H, m); δ_C (100.6 MHz, CDCl₃)

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158.9, 158.8, 147.7, 140.7, 128.5, 122.0, 114.2, 128.2, 114.2, 55.3, 48.3, 28.8, 26.3, 22.8, 21.8, 16.1; m/z (EI) 317 (M)⁺, 121, 91; [Found: (M)⁺, 317.1179, C₁₈H₂₀NO₂Cl requires (M)⁺, 317.1183].

Major diastereomer: *4,4,6-trichloro-2-(4-methoxy-benzyl)-1-methylene-2-aza-spiro[4.5]decan-3-one (40a)* as a pale yellow oil, yield 13%, R_f (5:1 petrol:ethyl acetate) 0.27; $\nu_{\max}$ (film)/cm⁻¹ 2942, 1746, 1647, 1612, 1514, 1444, 1389, 1354, 1298, 1249, 1178, 1033, 946, 833; δ_{H} (400 MHz, CDCl₃) 7.19 (2H, d, J 8.6 Hz), 6.85 (2H, d, J 8.6 Hz), 5.12 (1H, d, J 15.4 Hz), 4.71 (1H, d, J 3.0 Hz), 4.67 (1H, d, J 3.0 Hz), 4.26 (1H, d, J 15.4 Hz), 4.24 (1H, dd, J 11.6, 4.3 Hz), 3.78 (3H, s), 2.60-2.50 (1H, m), 2.10-1.95 (2H, m), 1.90-1.80 (2H, m), 1.78-1.60 (2H, m), 1.50-1.35 (1H, m); δ_{C} (100.6 MHz, CDCl₃) 166.5, 159.1, 145.5, 128.4, 127.1, 114.1, 93.9, 90.4, 63.8, 55.5, 55.3, 44.6, 32.7, 30.7, 25.4, 22.6; m/z (EI) 387 (M)⁺, 352, 318, 281, 155, 135, 121, 91, 77, 65; [Found: (M)⁺, 387.0546, C₁₈H₂₀NO₂Cl₃ requires (M)⁺, 387.0560].

Minor diastereomer: *4,4,6-trichloro-2-(4-methoxy-benzyl)-1-methylene-2-aza-spiro[4.5]decan-3-one (40b)* as a pale yellow oil, yield 7%, R_f (5:1 petrol:ethyl acetate) 0.23; $\nu_{\max}$ (film)/cm⁻¹ 2989, 1744, 1648, 1614, 1514, 1446, 1393, 1353, 1298, 1248, 1179, 1032, 827; δ_{H} (500 MHz, CDCl₃, 323 K) 7.18 (2H, d, J 8.2 Hz), 6.84 (2H, d, J 8.2 Hz), 4.87 (1H, d, J 15.2 Hz), 4.60 (1H, d, J 3.3 Hz), 4.55 (1H, d, J 15.2 Hz), 4.49 (1H, br d), 4.43-4.37 (1H, br m), 3.78 (3H, s), 2.65-2.50 (2H, br m), 2.17-2.05 (1H, m), 1.97-1.90 (1H, m), 1.82-1.69 (1H, m), 1.60-1.42 (3H, m); δ_{C} (125.8 MHz, CDCl₃, 323 K) 166.7, 159.7, 148.5, 129.1, 127.2, 114.6, 91.3, 88.3, 64.9, 56.4, 55.6, 45.3, 33.6, 33.5, 23.8, 21.1; m/z (EI) 387 (M)⁺, 352, 318, 121, 77; [Found: (M)⁺, 387.0557, C₁₈H₂₀NO₂Cl₃ requires (M)⁺, 387.0560].

2,2,2-Trichloro-N-(4-methoxy-benzyl)-N-(1-methylene-3-phenyl-allyl)-acetamide (41)

Pale yellow oil, yield 31% from *trans*-4-phenyl-3-but-2-one), R_f (9:1 petrol:ethyl acetate) 0.28; $\nu_{\max}$ (film)/cm⁻¹ 3031, 2953, 2836, 1673, 1635, 1611, 1517, 1448, 1391, 1249, 1176, 1113, 1033, 965, 908, 839, 813, 757, 693, 667; δ_{H} (400 MHz, CDCl₃) 7.43 (2H, d, J 7.3 Hz), 7.32-7.21 (5H, m), 6.83 (2H, d, J 8.5 Hz), 6.67 (1H, d, J 16.3 Hz), 6.60 (1H, d, J 16.3 Hz), 5.60-5.20 (1H, br s), 5.44 (1H, s), 4.99 (1H, s), 4.20-3.80 (1H, br s), 3.76 (3H, s); δ_{C} (100.6 MHz, CDCl₃) 161.4, 159.5, 143.6, 135.8, 128.3, 131.1, 130.9, 128.9, 128.6, 127.0, 124.6, 122.1, 113.8, 93.2, 55.2, 54.0; m/z (EI) 409 (M)⁺, 374, 338, 288, 253, 223, 197, 176, 155, 131, 121, 91, 77, 67; [Found: (M-Cl)⁺, 374.0717, C₂₀H₁₈NO₂Cl₂ requires (M-Cl)⁺, 374.0715].

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3-Chloro-4-(chloro-phenyl-methyl)-1-(4-methoxy-benzyl)-5-methylene-1,5-dihydro-pyrrol-2-one (42)

Pale yellow oil, yield 71%), R_f (5:1 petrol:ethyl acetate) 0.20; $\nu_{\max}$ (film)/ cm^{-1} 2932, 1708, 1630, 1609, 1512, 1444, 1394, 1349, 1303, 1244, 1175, 1139, 1107, 1030, 959, 813, 733, 700; δ_H (400 MHz, CDCl_3) 7.44 (2H, d, J 7.3 Hz), 7.38-7.28 (3H, m), 7.13 (2H, d, J 8.8), 6.83 (2H, d, J 8.8 Hz), 6.30 (1H, s), 5.08 (1H, d, J 2.8 Hz), 4.99 (1H, d, J 2.8 Hz), 4.81 (1H, d, J 15.6 Hz), 4.73 (1H, d, J 15.6 Hz), 3.75 (3H, s); δ_C (100.6 MHz, CDCl_3) 163.1, 159.1, 140.2, 140.1, 136.9, 128.3, 129.9, 128.8, 128.6, 128.5, 126.9, 114.2, 99.5, 55.3, 53.4, 43.2; m/z (EI) 373 (M)⁺, 337, 302, 121, 91, 77; [Found: (M)⁺, 373.0650, $\text{C}_{20}\text{H}_{17}\text{NO}_2\text{Cl}_2$ requires (M)⁺, 373.0636].

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Chemist uo Ueaeu
JVG3-110-1
PROTON.w CDC13 u JVG 22

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JV63-114-1
PROTON, w CDCl3 u JVG 3

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PROTONight, w CDCl₃ u JVG 28

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PROTONight.w CDC13 u JVG 4
JVG4-2-2

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Chemical structure 20 is a macrocyclic molecule. It consists of two 4-(p-methylbenzyl)pyrrolidin-2-one rings. These rings are connected by a long chain that includes two trans-alkene units. The chain also contains several methyl groups and a central carbon atom bonded to two methyl groups.

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C=CC=CNC(=O)C(Cl)(Cl)Cl

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JVG3-147-1

PROTON w CDCl3 u JVG 3

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Current Data Parameters
NAME JVG3-147-1
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PROCNO 1
F2 - Acquisition Parameters
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RG 327.68
PE 6.00 usec
DE 1.00000000 sec
ACQ 0.00000000 sec
RG 0.0150000 sec
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SI 32768
SF 400.024612 MHz
WDW EM
SS 0.20 Hz
GB 0.1
F2 - Acquisition Parameters
Date_ 20060613
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INSTRUM spect
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PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 16
DSH 8223.665 Hz
AQ 1.392144 sec
RG 327.68
PE 6.00 usec
DE 1.00000000 sec
ACQ 0.00000000 sec
RG 0.0150000 sec

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JV63-134-2
PROTON, w CDCl3 u JV6 28

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JVG3-134-4
PROTON.W CDCl3 u JVG 19

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JV64-55-1
PROTON: w CDCl3 u JV6 26

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Chemist Jo Geden
JVG4-35-1
PROTON.W CDCl3 u JVG 27

31

SSS

Regiochemistry of Copper(I) Mediated Cyclization Reactions of Halo-dienamides

Andrew J. Clark, Joanna V. Geden, Stephen Thom, Paul Wilson.

Regiochemistry of Copper(I) Mediated Cyclization Reactions of Halo-dienamides

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Chemist Jo Geden

JVG4-57-RM

PROTON.w CDCl3 u JVG 22

SS7

Andrew J. Clark, Joanna V. Geden, Stephen Thom, Paul Wilson.

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Regiochemistry of Copper(I) Mediated Cyclization Reactions of Halo-dienamides

Andrew J. Clark, Joanna V. Geden, Stephen Thom, Paul Wilson.

Regiochemistry of Copper(I) Mediated Cyclization Reactions of Halo-dienamides

Andrew J. Clark, Joanna V. Geden, Stephen Thom, Paul Wilson.

Chemist Jo Geden
JV63-32-1
PROTON NMR, w CDCl₃ u JV6 35

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Regiochemistry of Copper(I) Mediated Cyclization Reactions of Halo-dienamides

Andrew J. Clark, Joanna V. Geden, Stephen Thom, Paul Wilson.

Chemist Jo Geden
JVG3-55-2
PROTON, w CDCl3 u JVG 6

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Regiochemistry of Copper(I) Mediated Cyclization Reactions of Halo-dienamides

Andrew J. Clark, Joanna V. Geden, Stephen Thom, Paul Wilson.

Andrew J. Clark, Joanna V. Geden, Stephen Thom, Paul Wilson.

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563

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Andrew J. Clark, Joanna V. Geden, Stephen Thom, Paul Wilson.

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=====
Current Data Parameters
NAME          Dev05-2003
EXPNO         62
PROCNO        1

F2 - Acquisition Parameters
Date_         20031208
Time          10.04
INSTRUM       drx500
PULPROG       gauss
TD            65536
SOLVENT       CDCl3
NS            9935
DS            4
SWH           33446.541 Hz
AQ            1.06420683 sec
RG            10321.3
DE            6.00 usec
CHRG2         140.00000000
D1            5.00000000 sec
D2            0.00178751 sec
d3            0.00464629 sec
d4            0.00464629 sec

===== CHANNEL f1 =====
NUC1           13C
P2            0.00 usec
SFO1          125.7722992 MHz

===== CHANNEL f2 =====
NUC2           1H
P2            0.00 usec

=====
F2 - Processing parameters
SI            32768
MMW           2.00 Hz
LB            0
GB            0

1D NMR plot parameters
P1            7.33333 ppm/cm
PCWD          922.23282 Hz/cm

```


Regiochemistry of Copper(I) Mediated Cyclization Reactions of Halo-dienamides

Andrew J. Clark, Joanna V. Geden, Stephen Thom, Paul Wilson.

Chemist Jo Geden
JV63-57-4
PROTON.W CDCl3 u JV6 25

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Andrew J. Clark, Joanna V. Geden, Stephen Thom, Paul Wilson.

\$67

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Chemist NMR Service
JVG3-65-2-1
C13pend. w CDCl3 u ajc 3

Chemical structure of **37a** is shown. The structure is a bicyclic compound with a bromine atom (Br) and a methyl group (Me) attached to the ring. The structure is labeled **37a**.

The ¹³C NMR spectrum (CDCl₃) shows the following chemical shifts (ppm):

- 177.922 (C=O)
- 158.792 (Ar C-O)
- 154.524 (Ar C-O)
- 128.842 (Aromatic)
- 128.683 (Aromatic)
- 128.108 (Aromatic)
- 113.875 (Aromatic)
- 84.859 (CH-OH)
- 77.273 (Solvent)
- 77.193 (Solvent)
- 77.020 (Solvent)
- 76.766 (Solvent)
- 64.273 (CH-OH)
- 60.405 (CH-OH)
- 55.223 (CH-OH)
- 48.431 (CH-OH)
- 43.608 (CH₃)
- 40.737 (CH₃)
- 34.902 (CH₃)
- 33.337 (CH₃)
- 29.706 (CH₃)
- 29.368 (CH₃)
- 27.051 (CH₃)
- 22.929 (CH₃)
- 22.697 (CH₃)
- 21.062 (CH₃)
- 14.546 (CH₃)
- 14.127 (CH₃)
- 0.000 (TMS)

The spectrum is labeled **37a**.

```

Current Data Parameters
NAME          Dec05-2003
EXPNO         42
PROCNO        1

F2 - Acquisition Parameters
Date_         20031126
Time          17.54
INSTRUM       spect
PULPROG       drx500
TD            65536
SOLVENT       CDCl3
NS            8192
DS            4
AQ            1.0042083 sec
RG            4096
DE            6.00 usec
CHRG2         140.00000000
D1            5.00000000 sec
D2            0.00000000 sec
d3            0.00178571 sec
d4            0.0046629 sec

===== CHANNEL f1 =====
NUC1           13C
P2            0.00 usec
SFO1          125.7722992 MHz

===== CHANNEL f2 =====
NUC2           1H
P4            0.00 usec

F2 - Processing parameters
SI            32768
WDW           RM
GB            2.00 Hz
GB            0

1D NMN plot parameters
PCPN          1
P2PC          0.33333 gpm/ct
FREQ          922.23282 Hz/cm

```

Regiochemistry of Copper(I) Mediated Cyclization Reactions of Halo-dienamides

Andrew J. Clark, Joanna V. Geden, Stephen Thom, Paul Wilson.

Chemist Jo Geden
JVG3-65-2-2
PROTONMight w CDCl3 + JVG 28

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Regiochemistry of Copper(I) Mediated Cyclization Reactions of Halo-dienamides

Andrew J. Clark, Joanna V. Geden, Stephen Thom, Paul Wilson.

Regiochemistry of Copper(I) Mediated Cyclization Reactions of Halo-dienamides

Andrew J. Clark, Joanna V. Geden, Stephen Thom, Paul Wilson.

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Regiochemistry of Copper(I) Mediated Cyclization Reactions of Halo-dienamides

Andrew J. Clark, Joanna V. Geden, Stephen Thom, Paul Wilson.

Chemist Jo Geden
JV63-39-4-1
PROTON.W CDCl3 u JV6 17

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Regiochemistry of Copper(I) Mediated Cyclization Reactions of Halo-dienamides

Andrew J. Clark, Joanna V. Geden, Stephen Thom, Paul Wilson.

Regiochemistry of Copper(I) Mediated Cyclization Reactions of Halo-dienamides

Andrew J. Clark, Joanna V. Geden, Stephen Thom, Paul Wilson.

Regiochemistry of Copper(I) Mediated Cyclization Reactions of Halo-dienamides

Andrew J. Clark, Joanna V. Geden, Stephen Thom, Paul Wilson.

Chemist Jo Geden
JVG3-39-4-2
C13pend1.w CDC13 u JVG 22

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Andrew J. Clark, Joanna V. Geden, Stephen Thom, Paul Wilson.

$$C_{18}H_{20}O_2N_2 = 388.72$$

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input Data Parameters

[illegible]

Regiochemistry of Copper(I) Mediated Cyclization Reactions of Halo-dienamides

Andrew J. Clark, Joanna V. Geden, Stephen Thom, Paul Wilson.

Regiochemistry of Copper(I) Mediated Cyclization Reactions of Halo-dienamides

Andrew J. Clark, Joanna V. Geden, Stephen Thom, Paul Wilson.

Chemist Jo Geden
JV63-88-1-1
PROTON w CDCl3 u JV6 17

S80

Current Data Parameters
NAME Apr23-2004
EXPTNO 40
PROCNO 1
F2 - Acquisition Parameters
Date_ 20040422
Time 16.52
INSTRUM qnp400
PROBHD 1H/13C
PULPROG zgpg30
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
F2 125.761 MHz
AQ 25.4 sec
RG 6.00 usec
DE 1.0000000 sec
ACQSTRT 0.1310000 sec
RGWGT 0.1310000 sec
===== CHANNEL f1 =====
NUC1 13C
STO1 400.014000 MHz
F2 - Processing parameters
SI 32768
SF 400.136199 MHz
RGW -0.20 Hz
GB 0.1
F2 NMR plot parameters
FREQ 100.626100 MHz
PULP 102.57180 Hz/cm

Andrew J. Clark, Joanna V. Geden, Stephen Thom, Paul Wilson.

File with other

581

[illegible]

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F2 - Processing parameters	
SF	65536
WDW	EM
LB	2.00 Hz
GB	0

1D NMR plot parameters	
PPMCM	5.64103 ppm/cm
HZCM	567.41736 Hz/cm

```

Current Data Parameters
NAME          Mar12-2004
EXNO          12
PROCNO        1

F2 - Acquisition Parameters
Date          20040312
Time          16.45
INSTRUM       dpx400
PULPROG       prescand
TD             65536
SOLVENT       CDCl3
NS             512
NH            25125.629 Hz
AQ            1.3044164 sec
RG            4096
DE            6.00 usec
CNS2          140.0000000
D1            2.00000000 sec
d15           0.00178571 sec
d4            0.00464529 sec

===== CHANNEL f1 =====
NUC1          13C
P2            0.00 usec
SFO1          100.5976627 MHz

===== CHANNEL f2 =====
NUC2          1H
P4            0.00 usec

```

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