Ru-Catalyzed Addition of Imides to Alkynes: A New Synthesis of Enimides

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General: Reactions were performed in oven-dried glassware under a nitrogen atmosphere containing a teflon-coated stirrer bar and dry septum. Solvents were freshly distilled from CaH₂. All commercially available reagents were distilled either from CaH₂ or molecular sieves under an inert atmosphere before use. A sample of (cod)Ru[met]₂ was supplied by Umicore AG. GC analyses were carried out using an HP-5 capillary column (Phenyl Methyl Siloxane 30 m x 320 x 0.25, 100/2.3-30-300/3) and a time program beginning with 2 min at 60 °C followed by 30 °C/min ramp to 300 °C, then 3 min at this temp. Column chromatography was performed using Combi Flash Companion-Chromatography-Systems (Ico-Systems) and RediSep packed column (12 g). TLC analyses were performed on commercial Kieselgel 60 F254 silica gel plates. NMR spectra were obtained on Bruker AMX 400 systems using CDCl₃ as solvent, with proton and carbon resonances at 400 MHz and 100 MHz, respectively. Mass spectral data were acquired on a GC-MS Saturn 2100 T (Varian).

Representative procedure for the catalytic hydroimidation of terminal alkynes.

Synthesis of *N*-((*Z*)-hex-1-enyl)succinimide (3a); Method A. An oven-dried flask was charged with (cod)Ru[met]₂ (6.4 mg, 0.02 mmol), scandium triflate (19.7 mg, 0.04 mmol) and succinimide (**1a**, 99.1 mg, 1.00 mmol) and flushed with argon. Subsequently, tri-*n*-butylphosphine (15 µL, 0.06 mmol), 1-hexyne (**2a**; 229 µL, 2.0 mmol), and dry DMF (3.0 mL) were added *via* syringe. The resulting colorless solution was stirred for 15 h at 60 °C, then poured into an aqueous sodium bicarbonate solution (30 mL). The resulting mixture was extracted repeatedly with 20 mL portions of ethyl acetate, the combined organic layers were washed with water and brine, dried with magnesium sulfate, and filtered, and the volatiles were removed *in vacuo*. The residue was purified by column chromatography (silica gel, ethyl acetate/hexanes 3:1) to afford the title product (177.6 mg, 98%) as a yellowish oil. ¹H-NMR (400 MHz, CDCl₃): δ = 5.82 (d, ³*J* = 8.6 Hz, 1H), 5.68 (dt, ³*J* = 15.0 Hz, 7.5 Hz, 1H), 2.74 (s, 4H), 1.89 (dt, *J* = 14.6, 7.3, 1.6 Hz, 2H), 1.25 – 1.36 (m, 4H), 0.83 (t, *J* = 7.1 Hz, 3H) ppm. ¹³C-NMR (101 MHz, CDCl₃): δ = 175.6, 133.6, 116.7, 30.7, 28.4, 27.7, 22.3, 13.8 ppm. Anal. Calcd for C₁₀H₁₅NO₂: C, 66.2; H, 8.3; N, 7.7. Found: C, 66.3; H, 8.4; N, 7.7. MS (Ion trap, EI): *m/z* (%) = 182 (13, [M⁺]), 138 (67), 100 (75), 82 (100), 56 (44).

Representative procedure for the catalytic for the hydroimidation of terminal alkynes.

Synthesis of *N*-((*E*)-hex-1-enyl)succinimide (4a); Method B. An oven-dried flask was charged with (cod)Ru[met]₂ (16 mg, 0.05 mmol), scandium triflate (19.7 mg, 0.04 mmol) and succinimide (**1a**, 99.1 mg, 1.00 mmol) and flushed with argon. Subsequently, tri-isopropylphosphine (29 µL, 0.15 mmol), 1-hexyne (**2a**; 229 µL, 2.0 mmol), and dry DMF (3.0 mL) were added *via* syringe. The resulting colorless solution was stirred for 15 h at 60 °C, then poured into an aqueous sodium bicarbonate solution (30 mL). The resulting mixture was extracted repeatedly with 20 mL portions of ethyl acetate, the combined organic layers were washed with water and brine, dried with magnesium sulfate and filtered, and the volatiles were removed *in vacuo*. The residue was purified by column chromatography (silica gel, ethyl acetate/hexanes 3:1) to afford the title product (135.5 mg, 75 %) as a yellowish oil. ¹H-NMR (400 MHz, CDCl₃): δ = 6.52 (d, *J* = 14.7 Hz, 1H), 6.36 (dt, *J* = 15.0 Hz, 14.7 Hz, 1H), 2.66 (s, 4H), 2.05 (dt, *J* = 14.6, 7.2, 2H), 1.37 (m, 2H), 1.24 – 1.31 (m, 4H), 0.83 (t, *J* = 7.2 Hz, 3H) ppm. ¹³C-NMR (101 MHz, CDCl₃): δ = 175.4, 124.5, 117.8, 31.2, 30.5, 27.6, 22.0, 13.7 ppm. Anal. Calcd for C₁₀H₁₅NO₂: C, 66.2; H, 8.3; N, 7.7. Found: C, 66.4; H, 8.5; N, 7.9. MS (Ion trap, EI): *m/z* (%) = 182 (30, [M⁺]), 138 (100), 100 (75), 82 (94), 56 (53).

***N*-[(*Z*)-hex-1-en-1-yl]glutarimide (3b)** was prepared according to method A, using the following amounts: (cod)Ru[met]₂ (6.4 mg, 0.02 mmol), scandium triflate (19.7 mg, 0.04 mmol), glutarimide (113.1 mg, 1.00 mmol), tri-*n*-butylphosphine (15 µL, 0.06 mmol), 1-hexyne (**2a**; 229 µL, 2.0 mmol). ¹H-NMR (400 MHz, CDCl₃): δ = 5.88 (d, ³*J* = 8.3 Hz, 1H), 5.70 (dt, ³*J* = 15.0 Hz, 7.5 Hz, 1H), 2.67 (t, ³*J* = 6.66 Hz, 4H), 1.96 (t, *J* = 7.3 Hz, 2H), 1.78 (dt, *J* = 14.5, 7.3, 1.56 Hz, 2H) 1.23 – 1.33 (m, 4H), 0.82 (t, *J* = 7.14 Hz, 3H) ppm. ¹³C-NMR (101 MHz, CDCl₃): δ = 171.6, 132.9, 120.0, 32.9, 30.3, 26.9, 22.2, 17.2, 13.7 ppm. Anal. Calcd for C₁₁H₁₇NO₂: C, 67.7; H, 8.8; N, 7.2. Found: C, 66.9; H, 8.7; N, 7.0. MS (Ion trap, EI): *m/z* (%) = 195 (100, [M⁺]), 138 (52), 114 (69), 86 (40), 56 (50).

***N*-[(*Z*)-hex-1-en-1-yl]-4,4-dimethylglutarimide (3c)** was prepared according to method A, using the following amounts: (cod)Ru[met]₂ (6.4 mg, 0.02 mmol), scandium triflate (19.7 mg, 0.04 mmol), 3,3-dimethylglutarimide (141.2 mg, 1.00 mmol), tri-*n*-butylphosphine (15 µL, 0.06 mmol), 1-hexyne (**2a**; 229 µL, 2.0 mmol). ¹H-NMR (400 MHz, CDCl₃): δ = 5.90 (d, ³*J* = 8.22 Hz, 1H), 5.74 (dt, ³*J* = 15.0 Hz, 8.22 Hz), 2.56 (s, 4H), 1.83 (dt, *J* = 14.5, 7.29, 1.61 Hz, 2H) 1.31 – 1.39 (m, 2H), 1.23 – 1.30 (m, 2H), 1.13 (s, 6H), 0.85 (t, *J* = 7.1 Hz, 3H) ppm. ¹³C-NMR (101 MHz, CDCl₃): δ = 171.0, 133.2, 119.7, 46.5, 30.4, 29.2 27.8, 27.2, 22.3, 13.7 ppm. Anal. Calcd for C₁₃H₂₁NO₂: C, 69.9; H, 9.5; N, 6.3. Found: C, 69.8; H, 9.6; N, 6.3. MS (Ion trap, EI): *m/z* (%) = 224 (100, [M⁺]), 180 (32), 142 (35), 83 (42), 56 (21).

***N*-[*(Z)*-4-phenylbut-1-en-1-yl]succinimide (3d)** was prepared according to method A, using the following amounts: (cod)Ru[met]₂ (6.4 mg, 0.02 mmol), scandium triflate (19.7 mg, 0.04 mmol), succinimide (99.1 mg, 1.00 mmol), tri-*n*-butylphosphine (15 μ L, 0.06 mmol), 4-phenyl-1-butyne (281 μ L, 2.0 mmol). ¹H-NMR (400 MHz, CDCl₃): δ = 7.21 – 7.24 (m, 2H), 7.12 – 7.16 (m, 3H), 5.87 (d, ³*J* = 8.31 Hz, 1H), 5.70 (d, ³*J* = 8.41 Hz, 1H), 2.70 (t, *J* = 7.8 Hz, 2H), 2.67 (s, 4H), 2.24 (dt, *J* = 14.6, 7.3, 1.6 Hz, 2H) ppm. ¹³C-NMR (101 MHz, CDCl₃): δ = 175.5, 141.3, 131.8, 128.5, 126.0, 117.4, 34.7, 30.0, 28.4 ppm. Anal. Calcd for C₁₄H₁₅NO₂: C, 73.3; H, 6.6; N, 6.1. Found: C, 73.3; H, 6.6; N, 6.0. MS (Ion trap, EI): *m/z* (%) = 229 (5, [M⁺]), 138 (100), 110 (17), 65 (21), 56 (32).

***N*-[*(Z)*-2-phenylvinyl]succinimide (3e)** was prepared according to method A, using the following amounts: (cod)Ru[met]₂ (6.4 mg, 0.02 mmol), scandium triflate (19.7 mg, 0.04 mmol), succinimide (99.1 mg, 1.00 mmol), tri-*n*-butylphosphine (15 μ L, 0.06 mmol), phenylacetylene (220 μ L, 2.0 mmol). ¹H-NMR (400 MHz, CDCl₃): δ = 7.19 – 7.29 (m, 3H), 7.15 (d, ³*J* = 7.1 Hz, 2H), 6.65 (d, ³*J* = 9.3 Hz, 1H), 6.07 (d, ³*J* = 9.3 Hz, 1H), 2.68 (s, 4H) ppm. ¹³C-NMR (101 MHz, CDCl₃): δ = 175.2, 134.7, 131.0, 128.5, 128.2, 128.0, 117.1, 28.5 ppm. Anal. Calcd for C₁₂H₁₁NO₂: C, 71.6; H, 5.5; N, 7.0. Found: C, 71.5; H, 5.6; N, 6.7. MS (Ion trap, EI): *m/z* (%) = 201 (100, [M⁺]), 172 (5), 118 (65), 89 (25), 55 (15).

The data correspond to those reported in the literature: Ogawa, T.; Kiji, T.; Hayami, K.; Suzuki *Chem. Lett.* **1991**, 20, 1443.

***N*-[*(Z)*-oct-1-en-1-yl]succinimide (3f)** was prepared according to method A, using the following amounts: (cod)Ru[met]₂ (6.4 mg, 0.02 mmol), scandium triflate (19.7 mg, 0.04 mmol), succinimide (99.1 mg, 1.00 mmol), tri-*n*-butylphosphine (15 μ L, 0.06 mmol), 1-octyne (298 μ L, 2.0 mmol). ¹H-NMR (400 MHz, CDCl₃): δ = 5.81 (d, ³*J* = 8.51 Hz, 1H), 5.67 (dt, ³*J* = 15.1 Hz, 7.5 Hz, 1H), 2.73 (s, 4H), 1.87 (qd, *J* = 7.38, 1.61 Hz, 2H), 1.29 – 1.38 (m, 4H), 1.16 – 1.27 (m, 6H), 0.81 (t, *J* = 7.1 Hz, 3H) ppm. ¹³C-NMR (101 MHz, CDCl₃): δ = 175.6, 133.7, 116.7, 31.6, 28.8, 28.4, 28.1, 22.5, 14.0 ppm. Anal. Calcd for C₁₂H₁₉NO₂: C, 68.9; H, 9.15; N, 6.7. Found: C, 68.8; H, 9.17; N, 6.6. MS (Ion trap, EI): *m/z* (%) = 210 (10, [M⁺]), 138 (70), 110 (100), 81 (48), 55 (40).

***N*-[*(Z)*-hex-1-en-1-yl]-1H-pyrrolo[3,4-*c*]pyridine-1,3(2H)-dione (3g)** was prepared according to method A, using the following amounts: (cod)Ru[met]₂ (6.4 mg, 0.02 mmol), scandium triflate (19.7 mg, 0.04 mmol), 3,4-pyridinedicarboximide (148.1 mg, 1.00 mmol), tri-*n*-butylphosphine (15 μ L, 0.06 mmol), 1-hexyne (**2a**; 229 μ L, 2.0 mmol). ¹H-NMR (400 MHz, CDCl₃): δ = 9.18 (s, 1H), 9.08 (d, *J* = 4.8 Hz, 1H), 7.77 (t, 1H), 6.09 (d, ³*J* = 8.2 Hz, 1H), 5.85 (m, 1H), 2.04 (dt, *J* = 14.8, 7.4, 1.61 Hz, 2H), 1.36 – 1.47 (m, 2H), 1.25 – 1.33 (m, 2H), 0.86 (t, *J* = 7.2 Hz, 3H) ppm. ¹³C-NMR (101 MHz, CDCl₃): δ = 165.7, 165.3, 155.8, 145.1, 139.5, 134.4, 124.8, 116.9, 115.7, 30.8, 28.0, 22.3, 13.8 ppm. MS (Ion trap, EI): *m/z* (%) = 230 (8, [M⁺]), 187 (100), 150 (24), 82 (18), 50 (22).

3-[*(Z)*-hex-1-en-1-yl]-1,5,5-trimethylimidazolidine-2,4-dione (3h) was prepared according to method A, using the following amounts: (cod)Ru[met]₂ (6.4 mg, 0.02 mmol), scandium triflate (19.7 mg, 0.04 mmol), 1,5,5-trimethylhydantoin (156.2 mg, 1.00 mmol), tri-*n*-butylphosphine (15 μ L, 0.06 mmol), 1-hexyne (**2a**; 229 μ L, 2.0 mmol). ¹H-NMR (400 MHz, CDCl₃): δ = 5.79 (d, ³*J* = 8.41 Hz, 1H), 5.54 (dt, ³*J* = 15.1 Hz, 7.56 Hz, 1H), 2.80 (s, 3H), 1.87 (dt, *J* = 14.6, 7.3, 1.62 Hz, 2H), 1.34 (s, 6H), 1.24 – 1.33 (m, 2H), 1.13 – 1.23 (m, 6H), 0.75 (t, *J* = 7.24 Hz, 3H) ppm. ¹³C-NMR (101 MHz, CDCl₃): δ = 174.7, 154.3, 131.7, 116.8, 61.5, 30.7, 27.9, 24.5, 22.5, 22.1, 13.7 ppm. Anal. Calcd for C₁₂H₂₀N₂O₂: C, 64.2; H, 8.9; N, 12.5. Found: C, 64.0; H, 8.7; N, 12.3. MS (Ion trap, EI): *m/z* (%) = 224 (100, [M⁺]), 194 (18), 142 (36), 83 (42), 56 (22).

***N*-[*(Z)*-2-phenylvinyl]glutarimide (3i)** was prepared according to method A, using the following amounts: (cod)Ru[met]₂ (6.4 mg, 0.02 mmol), scandium triflate (19.7 mg, 0.04 mmol), glutarimide (113.1 mg, 1.00 mmol), tri-*n*-butylphosphine (15 μ L, 0.06 mmol), phenylacetylene (220 μ L, 2.0 mmol). ¹H-NMR (400 MHz, CDCl₃): δ = 7.28 (t, *J* = 7.8 Hz, 2H), 7.24 (t, *J* = 7.4 Hz, 1H), 7.18 (d, *J* = 7.4 Hz, 2H), 6.71 (d, *J* = 9.0 Hz, 1H), 6.18 (d, *J* = 8.8 Hz, 1H), 2.64 (t, *J* = 6.5 Hz, 4H), 1.92 – 1.97 (m, 2H) ppm. ¹³C-NMR (101 MHz, CDCl₃): δ = 171.8, 134.7, 131.2, 128.5, 128.1, 127.8, 121.0, 33.0, 17.0 ppm. Anal. Calcd for C₁₃H₁₃NO₂: C, 72.5; H, 6.0; N, 6.3. Found: C, 72.3; H, 5.7; N, 6.3. MS (Ion trap, EI): *m/z* (%) = 196 (100, [M⁺]), 166 (22), 138 (52), 86 (40), 56 (50).

The data correspond to those reported in the literature: Ogawa, T.; Kiji, T.; Hayami, K.; Suzuki *Chem. Lett.* **1991**, 20, 1443.

***N*-[*(Z)*-hex-1-en-1-yl]phthalimide (3j)** was prepared according to method A, using the following amounts: (cod)Ru[met]₂ (6.4 mg, 0.02 mmol), scandium triflate (19.7 mg, 0.04 mmol), phthalimide (147.2 mg, 1.00 mmol), tri-*n*-butylphosphine (15 μ L, 0.06 mmol), 1-hexyne (**2a**; 229 μ L, 2.0 mmol). ¹H-NMR (400 MHz, CDCl₃): δ = 7.79 – 7.88 (m, 2H), 7.67 – 7.74 (m, 2H), 6.05 (dt, ³*J* = 8.41, 1.66 Hz, 1H), 5.70 – 5.77 (m, 1H), 2.04 (dt, *J* = 14.55, 7.3, 1.6 Hz, 2H), 1.35 – 1.43 (m, 2H), 1.25 – 1.33 (m, 2H), 0.83 (t, *J* = 7.24 Hz, 3H) ppm. ¹³C-NMR (101 MHz, CDCl₃): δ = 166.7, 134.2, 133.5, 132.1, 123.4, 116.2, 30.8, 28.0, 22.2, 13.9 ppm. Anal. Calcd for C₁₄H₁₅NO₂: C, 73.3; H, 6.6; N, 6.1. Found: C, 73.2; H, 6.5; N, 6.1. MS (Ion trap, EI): *m/z* (%) = 229 (8, [M⁺]), 186 (100), 148 (20), 76 (14), 56 (15).

The data correspond to those reported in the literature: Lam, P.Y.S.; Vincent, G.; Bonne, D.; Clark, C.G. *Tetrahedron Lett.* **2003**, *44*, 4927.

N-[(Z)-2-phenylvinyl]phthalimide (3k) was prepared according to method A, using the following amounts: (cod)Ru[met]₂ (6.4 mg, 0.02 mmol), scandium triflate (19.7 mg, 0.04 mmol), phthalimide (147.2 mg, 1.00 mmol), tri-*n*-butylphosphine (15 μ L, 0.06 mmol), phenylacetylene (220 μ L, 2.0 mmol). ¹H-NMR (400 MHz, CDCl₃): δ = 7.86 (dd, *J* = 5.48, 3.13 Hz, 2H), 7.74 (dd, *J* = 5.48, 3.13 Hz, 2H), 7.19 – 7.28 (m, 5H), 6.70 (d, *J* = 9.19 Hz, 1H), 6.33 (d, *J* = 9.19 Hz, 1H) ppm. ¹³C-NMR (101 MHz, CDCl₃): δ = 166.3, 135.1, 134.3, 132.2, 130.2, 128.4, 128.2, 128.0, 123.7, 116.4 ppm. Anal. Calcd for C₁₆H₁₁NO₂: C, 77.1; H, 4.4; N, 5.6. Found: C, 76.5; H, 4.45; N, 5.2. MS (Ion trap, EI): *m/z* (%) = 249 (100, [M⁺]), 204 (22), 104 (25), 76 (42), 50 (35).

The data correspond to those reported in the literature: Ogawa, T.; Kiji, T.; Hayami, K.; Suzuki *Chem. Lett.* **1991**, *20*, 1443.

1,5,5-trimethyl-3-[(Z)-2-phenylvinyl]imidazolidine-2,4-dione (3l) was prepared according to method A, using the following amounts: (cod)Ru[met]₂ (6.4 mg, 0.02 mmol), scandium triflate (19.7 mg, 0.04 mmol), 1,5,5-trimethylhydantoin (156.2 mg, 1.00 mmol), tri-*n*-butylphosphine (15 μ L, 0.06 mmol), phenylacetylene (220 μ L, 2.0 mmol). ¹H-NMR (400 MHz, CDCl₃): δ = 7.30 (d, *J* = 7.6 Hz, 2H), 7.26 (t, *J* = 7.4 Hz, 1H), 7.22 (d, *J* = 7.4 Hz, 2H), 6.65 (d, *J* = 8.8 Hz, 1H), 6.19 (d, *J* = 9.0 Hz, 1H), 2.92 (s, 3H), 1.36 (s, 6H) ppm. ¹³C-NMR (101 MHz, CDCl₃): δ = 173.7, 154.0, 135.0, 130.1, 128.3, 128.1, 128.0, 116.9, 61.4, 24.7, 22.0 ppm. Anal. Calcd for C₁₃H₁₈N₂O₂: C, 74.9; H, 7.1; N, 11.2. Found: C, 74.5; H, 6.7; N, 11.2. MS (Ion trap, EI): *m/z* (%) = 244 (100, [M⁺]), 145 (62), 117 (15), 89 (14), 56 (41).

N-[(Z)-2-phenylvinyl]-4,4-dimethylglutarimide (3m) was prepared according to method A, using the following amounts: (cod)Ru[met]₂ (6.4 mg, 0.02 mmol), scandium triflate (19.7 mg, 0.04 mmol), 3,3-dimethylglutarimide (141.2 mg, 1.00 mmol), tri-*n*-butylphosphine (15 μ L, 0.06 mmol), phenylacetylene (220 μ L, 2.0 mmol). ¹H-NMR (400 MHz, CDCl₃): δ = 7.28 (d, *J* = 7.8 Hz, 2H), 7.23 (t, *J* = 7.4 Hz, 1H), 7.18 (d, *J* = 7.1 Hz, 2H), 6.76 (d, *J* = 8.8 Hz, 1H), 6.16 (d, *J* = 9.0 Hz, 1H), 2.50 (s, 4H), 1.06 (s, 6H) ppm. ¹³C-NMR (101 MHz, CDCl₃): δ = 171.3, 134.8, 131.8, 128.4, 128.0, 127.8, 120.9, 46.5, 29.4, 27.9 ppm. Anal. Calcd for C₁₂H₁₇NO₂: C, 74.0; H, 7.0; N, 5.8. Found: C, 73.9; H, 6.8; N, 5.7. MS (Ion trap, EI): *m/z* (%) = 243 (100, [M⁺]), 200 (80), 119 (35), 83 (38), 55 (22).

N-[(Z)-4-phenylbut-1-en-1-yl]glutarimide (3n) was prepared according to method A, using the following amounts: (cod)Ru[met]₂ (6.4 mg, 0.02 mmol), scandium triflate (19.7 mg, 0.04 mmol), glutarimide (113.1 mg, 1.00 mmol), tri-*n*-butylphosphine (15 μ L, 0.06 mmol), 4-phenyl-1-butyne (281 μ L, 2.0 mmol). ¹H-NMR (400 MHz, CDCl₃): δ = 7.28 (t, *J* = 7.3 Hz, 2H), 7.18 (t, *J* = 7.8 Hz, 3H), 5.97 (d, *J* = 8.3 Hz, 1H), 5.78 (d, *J* = 8.1 Hz, 1H), 2.70 (t, *J* = 7.8 Hz, 2H), 2.65 (t, *J* = 6.5 Hz, 4H), 2.20 (m, 2H), 1.92 (m, 2H) ppm. ¹³C-NMR (101 MHz, CDCl₃): δ = 171.6, 141.4, 131.5, 128.4, 126.0, 120.6, 34.4, 32.9, 29.3, 17.2 ppm. Anal. Calcd for C₁₅H₁₇NO₂: C, 61.7; H, 7.0; N, 5.8. Found: C, 61.4; H, 6.8; N, 5.7. MS (Ion trap, EI): *m/z* (%) = 244 (10, [M⁺]), 152 (100), 130 (55), 91 (24), 56 (55).

1,5,5-trimethyl-3-[(Z)-4-phenylbut-1-en-1-yl]imidazolidine-2,4-dione (3o) was prepared according to method A, using the following amounts: (cod)Ru[met]₂ (6.4 mg, 0.02 mmol), scandium triflate (19.7 mg, 0.04 mmol), 1,5,5-trimethylhydantoin (156.2 mg, 1.00 mmol), tri-*n*-butylphosphine (15 μ L, 0.06 mmol), 4-phenyl-1-butyne (281 μ L, 2.0 mmol). ¹H-NMR (400 MHz, CDCl₃): δ = 7.27 (t, *J* = 7.4 Hz, 2H), 7.19 (t, *J* = 7.8 Hz, 3H), 5.97 (d, *J* = 8.6 Hz, 1H), 5.67 (d, *J* = 8.3 Hz, 1H), 2.88 (s, 3H), 2.75 (t, *J* = 7.8 Hz, 2H), 2.33 (m, 2H), 1.39 (s, 6H) ppm. ¹³C-NMR (101 MHz, CDCl₃): δ = 174.8, 154.1, 141.4, 130.8, 128.5, 128.4, 125.9, 117.1, 61.2, 34.8, 29.9, 24.5, 22.2 ppm. Anal. Calcd for C₁₈H₂₄N₂O₂: C, 70.5; H, 9.5; N, 10.3. Found: C, 70.2; H, 9.3; N, 10.1. MS (Ion trap, EI): *m/z* (%) = 273 (25, [M⁺]), 181 (100), 153 (38), 72 (4), 56 (18).

N-[(Z)-5-chloropent-1-en-1-yl]succinimide (3p) was prepared according to method A, using the following amounts: (cod)Ru[met]₂ (6.4 mg, 0.02 mmol), scandium triflate (19.7 mg, 0.04 mmol), succinimide (99.1 mg, 1.00 mmol), tri-*n*-butylphosphine (15 μ L, 0.06 mmol), 5-chloro-1-pentyne (213 μ L, 2.0 mmol). ¹H-NMR (400 MHz, CDCl₃): δ = 5.91 (d, ³*J* = 8.61 Hz, 1H), 5.65 (q, *J* = 7.76 Hz, 1H), 3.47 (t, *J* = 7.1, 2H), 2.75 (s, 4H, H-7), 2.09 (qd, *J* = 7.2, 1.62 Hz, 2H), 1.83 (qd, *J* = 6.85, 1.65 Hz, 2H) ppm. ¹³C-NMR (151 MHz, CDCl₃): δ = 175.5, 131.2, 118.4, 44.2, 31.1, 28.4, 25.1 ppm. Anal. Calcd for C₉H₁₂NO₂: C, 53.6; H, 6.0; N, 6.9. Found: C, 53.8; H, 6.1; N, 7.1. MS (Ion trap, EI): *m/z* (%) = 202 (20, [M⁺]), 166 (48), 138 (100), 82 (45), 56 (38).

N-[(E)-hex-1-en-1-yl]glutarimide (4b) was prepared according to method A, using the following amounts: (cod)Ru[met]₂ (16 mg, 0.05 mmol), scandium triflate (19.7 mg, 0.04 mmol), glutarimide (113.1 mg, 1.00 mmol), tri-isopropylphosphine (29 μ L, 0.15 mmol), 1-hexyne (**2a**; 229 μ L, 2.0 mmol). ¹H-NMR (400 MHz, CDCl₃): δ = 6.18 (d, *J* = 14.5 Hz, 1H), 5.94 (dt, *J* =

14.5 Hz, 1H), 2.67 (t, $J = 7.5$ Hz, 4H), 2.13 (dd, $J = 7.3$ Hz, 2H), 1.91 – 1.96 (m, 2H) 1.37 – 1.43 (m, 2H), 0.87 (t, $J = 7.3$ Hz, 3H) ppm. $^{13}\text{C-NMR}$ (101 MHz, CDCl_3): $\delta = 172, 131.5, 119.7, 32.2, 31.1, 30.3, 22.1, 16.9, 13.8$ ppm. Anal. Calcd for $\text{C}_{11}\text{H}_{17}\text{NO}_2$: C, 67.7; H, 8.8; N, 7.2. Found: C, 66.7; H, 8.8; N, 7.5. MS (Ion trap, EI): m/z (%) = 195 (42, $[\text{M}^+]$), 138 (57), 114 (100), 86 (44), 56 (60).

***N*-[*(E)*-hex-1-en-1-yl]-4,4-dimethylglutarimide (4c)** was prepared according to method A, using the following amounts: (cod)Ru[met] $_2$ (16 mg, 0.05 mmol), scandium triflate (19.7 mg, 0.04 mmol), 3,3-dimethylglutarimide (141.2 mg, 1.00 mmol), tri-isopropylphosphine (29 μL , 0.15 mmol), 1-hexyne (**2a**; 229 μL , 2.0 mmol). $^1\text{H-NMR}$ (400 MHz, CDCl_3): $\delta = 6.20$ (d, $J = 14.5$ Hz, 1H), 5.96 (dt, $J = 14.5$ Hz), 2.52 (s, 4H), 2.11 – 2.15 (m, 2H) 1.37 – 1.43 (m, 2H), 1.30 – 1.36 (m, 2H), 1.07 (s, 6H), 0.85 (t, $J = 7.2$ Hz, 3H) ppm. $^{13}\text{C-NMR}$ (101 MHz, CDCl_3): $\delta = 171.4, 131.3, 119.5, 46.6, 31.1, 30.4, 29, 27.6, 22.1, 13.8$ ppm. Anal. Calcd for $\text{C}_{13}\text{H}_{21}\text{NO}_2$: C, 69.9; H, 9.5; N, 6.3. Found: C, 69.5; H, 9.4; N, 6.4. MS (Ion trap, EI): m/z (%) = 224 (83, $[\text{M}^+]$), 180 (66), 142 (100), 83 (82), 56 (46).

1-[(1*E*)-4-phenylbut-1-en-1-yl]succinimide (4d) was prepared according to method A, using the following amounts: (cod)Ru[met] $_2$ (16 mg, 0.05 mmol), scandium triflate (19.7 mg, 0.04 mmol), succinimide (99.1 mg, 1.00 mmol), tri-isopropylphosphine (29 μL , 0.15 mmol), 1-hexyne (**2a**; 229 μL , 2.0 mmol). $^1\text{H-NMR}$ (400 MHz, CDCl_3): $\delta = 7.26 - 7.35$ (m, 2H), 7.22 (m, 3H), 6.69 (dt, $J = 14.6$ Hz, 1H), 6.49 (d, $J = 14.9$ Hz, 1H), 2.73 – 2.81 (m, 2H), 2.71 (s, 4H), 2.41 – 2.51 (m, 2H) ppm. $^{13}\text{C-NMR}$ (101 MHz, CDCl_3): $\delta = 175.3, 141.2, 128.33, 128.27, 125.9, 123.5, 118.4, 77.3, 35.6, 32.8, 27.7$ ppm. Anal. Calcd for $\text{C}_{14}\text{H}_{15}\text{NO}_2$: C, 73.3; H, 6.6; N, 6.1. Found: C, 73.5; H, 6.57; N, 5.8. MS (Ion trap, EI): m/z (%) = 229 (2, $[\text{M}^+]$), 138 (100), 110 (15), 65 (18), 56 (27).

***N*-[*(E)*-2-phenylvinyl]succinimide (4e)** was prepared according to method A, using the following amounts: (cod)Ru[met] $_2$ (16 mg, 0.05 mmol), scandium triflate (19.7 mg, 0.04 mmol), succinimide (99.1 mg, 1.00 mmol), tri-isopropylphosphine (29 μL , 0.15 mmol), phenylacetylene (220 μL , 2.0 mmol). $^1\text{H-NMR}$ (400 MHz, CDCl_3): 7.69 (d, $^3J = 14.9$ Hz, 1H), 7.45 (m, 2H), 7.35 (m, 2H), 7.28 (m, 1H), 7.20 (d, $^3J = 14.9$ Hz, 1H), 2.81 (s, 4H). $^{13}\text{C-NMR}$ (101 MHz, CDCl_3): $\delta = 175.2, 135.4, 128.7, 127.9, 126.4, 121.9, 117.8, 27.7$ ppm. Anal. Calcd for $\text{C}_{12}\text{H}_{11}\text{NO}_2$: C, 71.6; H, 5.5; N, 7.0. Found: C, 71.9; H, 5.6; N, 6.9. MS (Ion trap, EI): m/z (%) = 201 (100, $[\text{M}^+]$), 172 (5), 119 (40), 89 (13), 55 (15).

The data correspond to those reported in the literature: Ogawa, T.; Kiji, T.; Hayami, K.; Suzuki *Chem. Lett.* **1991**, 20, 1443.

Synthesis of 1-deutero-1-hexyne (2q): In a flame-dried, nitrogen-flushed flask, a small amount of sodium hydride (60% in mineral oil) (28 mg, 70 mmol) was added while stirring to 99% D_2O (20 mL, 1 mol). The alkyne (82 mmol, 9.4 mL) was added, and the two-phase mixture was stirred for 7 h. The operation was repeated. The layers were separated under a nitrogen atmosphere. The organic layer was dried over molecular sieves, filtered, and distilled to afford 1-deutero-1-hexyne (95 %). $^1\text{H-NMR}$ (400 MHz, CDCl_3): $\delta = 2.15$ (t, $J = 7.0$ Hz, 2H) 1.32 - 1.55 (m, 4H) 0.88 (t, $J = 7.2$ Hz, 3H) ppm. $^{13}\text{C-NMR}$ (101 MHz, CDCl_3): $\delta = 84.0$ (t, $^3J_{\text{C-D}} = 7.40$ Hz) 67.7 (t, $^2J_{\text{C-D}} = 38$ Hz) 30.6 (s) 21.8 (s) 18.0 (s) 13.4 (s) ppm.

Syntheses of *N*-[1'-deutero-(*Z*)-hex-1-en-1-yl]succinimide (3q). An oven-dried flask was charged with (cod)Ru[met] $_2$ (6.4 mg, 0.02 mmol), scandium triflate (19.7 mg, 0.04 mmol) and succinimide (**1a**, 99.1 mg, 1.00 mmol) and flushed with argon. Subsequently, tri-*n*-butylphosphine (15 μL , 0.06 mmol), 1-deutero-1-hexyne (**2q**, 229 μL , 2.0 mmol), and dry DMF (3.0 mL) were added *via* syringe. The resulting colorless solution was stirred for 15 h at 60 °C, the volatiles were removed *in vacuo*. The residue was purified by column chromatography (silica gel, ethyl acetate/hexanes 3:1) to afford the title product (145 mg, 80 %) as yellowish oil. $^1\text{H-NMR}$ (400 MHz, CDCl_3): 5.66 (t, $J = 7.3$ Hz, 1H) 2.72 (s, 4H) 1.88 (q, $J = 7.4$ Hz, 2H) 1.16 - 1.40 (m, 4H) 0.81 (t, $J = 7.2$ Hz, 3H) ppm. $^{13}\text{C-NMR}$ (101 MHz, CDCl_3): $\delta = 175.4$ (s) 133.1 (s) 116.2 (t, $^2J_{\text{C-D}} = 27$ Hz) 30.5 (s) 28.2 (s) 27.5 (s) 22.1 (s) ppm.

Syntheses of *N*-[1'-deutero-(*E*)-hex-1-en-1-yl]pyrrolidine-2,5-dione (4q). An oven-dried flask was charged with (cod)Ru[met] $_2$ (16 mg, 0.05 mmol), scandium triflate (19.7 mg, 0.04 mmol) and succinimide (**1a**, 99.1 mg, 1.00 mmol) and flushed with argon. Subsequently, tri-isopropylphosphine (29 μL , 0.15 mmol), 1-deutero-1-hexyne (**2q**, 229 μL , 2.0 mmol), and d_7 -DMF (1.0 mL) were added with a syringe. The resulting colorless solution was stirred for 15 h at 60 °C, and the volatiles were removed *in vacuo*. The residue was purified by column chromatography (silica gel, ethyl acetate/hexanes 3:1) to afford the title product (136 mg, 75 %) as yellowish oil. $^1\text{H-NMR}$ (400 MHz, CDCl_3): 6.53 (t, $J = 7.3$ Hz, 1H) 2.67 (s, 3H) 2.07 (td, $J = 7.5$ Hz, 2H) 1.22 - 1.43 (m, 4H) 0.85 (t, $J = 7.2$ Hz, 3H) ppm. $^{13}\text{C-NMR}$ (101 MHz, CDCl_3): $\delta = 175.3$ (s) 124.3 (s) 117.6 (t, $^2J_{\text{C-D}} = 27\text{Hz}$) 31.2 (s) 30.4 (s) 27.6 (s) 21.9 (s) 13.6 (s) ppm.

3a (additional signals correspond to the minor isomer)

^1H NMR (400 MHz, *CHLOROFORM-D*) δ ppm 5.79 (d, $J=8.6$ Hz, 1 H) 5.65 (dt, $J=15.0, 7.5$ Hz, 1 H) 2.71 (s, 3 H) 1.86 (dt, $J=14.6, 7.3, 1.6$ Hz, 2 H) 1.22 - 1.33 (m, 4 H) 0.80 (t, $J=7.1$ Hz, 3 H)

^{13}C NMR (101 MHz, *CHLOROFORM-D*) δ ppm 175.4 (s) 133.4 (s) 116.6 (s) 30.5 (s) 28.2 (s) 27.6 (s) 22.1 (s) 13.6 (s)

%	C	H	N
Theorie	66.2	8.3	7.7
Analyse	66.3	8.4	7.7

MS (Ion trap, EI): m/z (%) = 182 (13, $[\text{M}^+]$), 138 (67), 100 (75), 82 (100), 56 (44)

3b

^1H NMR (400 MHz, *CHLOROFORM-D*) δ ppm 5.86 - 5.90 (m, 1 H) 5.67 - 5.73 (m, 1 H) 2.67 (t, $J=6.6$ Hz, 4 H) 1.92 - 2.00 (m, 2 H) 1.78 (qd, $J=7.3$, 1.6 Hz, 2 H) 1.23 - 1.33 (m, 4 H) 0.82 (t, $J=7.1$ Hz, 3 H)

^{13}C NMR (101 MHz, *CHLOROFORM-D*) δ ppm 171.6 (s) 132.9 (s) 120.0 (s) 77.3 (s) 76.7 (s) 32.9 (s) 30.3 (s) 26.9 (s) 22.2 (s) 17.2 (s) 13.7 (s)

%	C	H	N
Theorie	67.7	8.8	7.2
Analyse	66.9	8.7	7.0

MS (Ion trap, EI): m/z (%) = 195 (100, $[\text{M}^+]$), 138 (52), 114 (69), 86 (40), 56 (50).

3c

^1H NMR (400 MHz, *CHLOROFORM-D*) δ ppm 5.87 (d, $J=8.2$ Hz, 1 H) 5.71 (td, $J=8.2$ Hz, 1 H) 2.53 (s, 4 H) 1.80 (q, $J=7.3, 5.8$ Hz, 2 H) 1.28 - 1.35 (m, $J=7.0$ Hz, 2 H) 1.20 - 1.27 (m, $J=7.2$ Hz, 2 H) 1.10 (s, 6 H) 0.82 (t, 3 H)

^{13}C NMR (101 MHz, *CHLOROFORM-D*) δ ppm 171.0 (s) 133.2 (s) 119.7 (s) 46.5 (s) 30.4 (s) 29.2 (s) 27.8 (s) 27.2 (s) 22.3 (s) 13.7 (s)

%	C	H	N
Theorie	69.9	9.5	6.3
Analyse	69.8	9.6	6.3

MS (Ion trap, EI): m/z (%) = 224 (100, $[\text{M}^+]$), 180 (32), 142 (35), 83 (42), 56 (21).

3d

¹H NMR (400 MHz, *CHLOROFORM-D*) δ ppm 7.21 - 7.24 (m, 2 H) 7.12 - 7.16 (m, 3 H) 5.87 (d, *J*=8.6 Hz, 1 H) 5.70 (d, *J*=8.4 Hz, 1 H) 2.66 - 2.71 (m, 6 H) 2.20 - 2.27 (m, 2 H)

%	C	H	N
Theorie	73.3	6.6	6.1
Analyse	73.3	6.6	6.0

MS (Ion trap, EI): *m/z* (%) = 229 (5, [*M*⁺]), 138 (100), 110 (17), 65 (21), 56 (32)

3e

^1H NMR (400 MHz, *CHLOROFORM-D*) δ ppm 7.19 - 7.29 (m, 3 H) 7.15 (d, $J=7.1$ Hz, 2 H) 6.65 (d, $J=9.3$ Hz, 1 H) 6.07 (d, $J=9.3$ Hz, 1 H) 2.68 (s, 4 H)

^{13}C NMR (101 MHz, *CHLOROFORM-D*) δ ppm 175.0 (s) 134.5 (s) 130.9 (s) 128.3 (s) 128.0 (s) 127.8 (s) 117.0 (s) 28.4 (s)

%	C	H	N
Theorie	71.6	5.5	7.0
Analyse	71.5	5.6	6.7

MS (Ion trap, EI): m/z (%) = 201 (100, $[\text{M}^+]$), 172 (5), 118 (65), 89 (25), 55 (15)

3f (additional signals correspond to the minor isomer)

%	C	H	N
Theorie	68.9	9.15	6.7
Analyse	68.8	9.17	6.6

MS (Ion trap, EI): m/z (%) = 210 (10, $[M]^+$), 138 (70), 110 (100), 81 (48), 55 (40)

3g (additional signals correspond to the minor isomer)

^1H NMR (400 MHz, *CHLOROFORM-D*) δ ppm 9.16 - 9.22 (m, 1 H) 9.08 - 9.13 (m, 1 H) 7.76 - 7.82 (m, 1 H) 6.10 (none, 1 H) 5.85 (q, $J=7.5$ Hz, 1 H) 2.07 (q, $J=7.2$ Hz, 2 H) 1.39 - 1.50 (m, 3 H) 1.32 - 1.39 (m, 2 H) 0.87 - 0.96 (m, 4 H)

MS (Ion trap, EI): m/z (%) = 230 (8, $[\text{M}^+]$), 187 (100), 150 (24), 82 (18), 50 (22)

3h (additional signals correspond to the minor isomer)

¹H NMR (400 MHz, *CHLOROFORM-D*) δ ppm 5.79 (d, *J*=8.4 Hz, 1 H) 5.54 (q, *J*=7.6 Hz, 1 H) 2.76 - 2.82 (m, 3 H) 1.84 - 1.92 (m, 2 H) 1.24 - 1.33 (m, 8 H) 1.13 - 1.23 (m, 2 H) 0.75 (t, *J*=7.2 Hz, 3 H)

¹³C NMR (101 MHz, *CHLOROFORM-D*) δ ppm 174.8 (s) 153.7 (s) 122.3 (s) 122.2 (s) 118.0 (s) 117.9 (s) 60.3 (s) 31.5 (s) 31.4 (s) 30.5 (s) 24.4 (s) 22.2 (s) 22.1 (s) 13.8 (s)

%	C	H	N
Theorie	64.2	8.9	12.5
Analyse	64.0	8.7	12.3

MS (Ion trap, EI): *m/z* (%) = 224 (100, [*M*⁺]), 194 (18), 142 (36), 83 (42), 56 (22)

^1H NMR (600 MHz, *CHLOROFORM-D*) δ ppm 7.27 - 7.31 (m, 2 H) 7.24 (t, $J=7.4$ Hz, 1 H) 7.18 (d, $J=7.4$ Hz, 2 H) 6.71 (d, $J=9.0$ Hz, 1 H) 6.18 (d, $J=8.8$ Hz, 1 H) 2.64 (t, $J=6.5$ Hz, 4 H) 1.92 - 1.97 (m, $J=6.5$, 6.5, 6.5, 6.5 Hz, 2 H)

^{13}C NMR (151 MHz, *CHLOROFORM-D*) δ ppm 171.6 (s) 134.5 (s) 131.0 (s) 128.3 (s) 127.9 (s) 127.6 (s) 120.8 (s) 32.8 (s) 16.8 (s)

%	C	H	N
Theorie	72.5	6.0	6.5
Analyse	72.3	5.7	6.3

MS (Ion trap, EI): m/z (%) = 196 (100, $[\text{M}]^+$), 166 (22), 138 (52), 86 (40), 56 (50)

3j (additional signals correspond to the minor isomer)

¹H NMR (400 MHz, *CHLOROFORM-D*) δ ppm 7.79 - 7.88 (m, 2 H) 7.67 - 7.74 (m, 2 H) 6.05 (dt, *J*=8.4, 1.7 Hz, 1 H) 5.70 - 5.77 (m, 1 H) 2.04 (qd, *J*=7.4, 1.7 Hz, 2 H) 1.35 - 1.43 (m, 2 H) 1.29 (ddd, *J*=14.9, 7.3, 7.1 Hz, 2 H) 0.83 (t, *J*=7.2 Hz, 3 H)

¹³C NMR (101 MHz, *CHLOROFORM-D*) δ ppm 166.8 (s) 134.1 (s) 133.3 (s) 132.2 (s) 123.4 (s) 123.3 (s) 116.2 (s) 30.9 (s) 27.9 (s) 22.3 (s) 13.7 (s)

%	C	H	N
Theorie	73.3	6.6	6.1
Analyse	73.2	6.5	6.1

MS (Ion trap, EI): *m/z* (%) = 229 (8, [*M*⁺]), 186 (100), 148 (20), 76 (14), 56 (15)

3k

^1H NMR (400 MHz, *CHLOROFORM-D*) δ ppm 7.86 (dd, $J=5.5, 3.1$ Hz, 2 H) 7.74 (dd, $J=5.5, 3.1$ Hz, 2 H) 7.26 (s, 4 H) 7.19 - 7.25 (m, 2 H) 6.70 (d, $J=9.2$ Hz, 1 H) 6.33 (d, $J=9.2$ Hz, 1 H)

%	C	H	N
Theorie	77.1	4.4	5.6
Analyse	76.5	4.45	5.2

MS (Ion trap, EI): m/z (%) = 249 (100, $[\text{M}^+]$), 204 (22), 104 (25), 76 (42), 50 (35)

^1H NMR (600 MHz, *CHLOROFORM-D*) δ ppm 7.30 (d, $J=7.6$ Hz, 2 H) 7.25 (s, 1 H) 7.22 (d, $J=7.4$ Hz, 2 H) 6.65 (d, $J=8.8$ Hz, 1 H) 6.19 (d, $J=9.0$ Hz, 1 H) 2.92 (s, 3 H) 1.36 (s, 6 H)

%	C	H	N
Theorie	74.9	7.1	11.5
Analyse	74.5	6.7	11.2

MS (Ion trap, EI): m/z (%) = 244 (100, $[\text{M}^+]$), 145 (62), 117 (15), 89 (14), 56 (41)

3m

¹H NMR (600 MHz, *CHLOROFORM-D*) δ ppm 7.28 (d, *J*=7.8 Hz, 2 H) 7.23 (s, 1 H) 7.18 (d, *J*=7.1 Hz, 2 H) 6.76 (d, *J*=8.8 Hz, 1 H) 6.16 (d, *J*=9.0 Hz, 1 H) 2.50 (s, 4 H) 1.06 (s, 6 H)

¹³C NMR (151 MHz, *CHLOROFORM-D*) δ ppm 171.1 (s) 131.6 (s) 128.3 (s) 127.8 (s) 127.7 (s) 120.7 (s) 46.3 (s) 27.7 (s)

%	C	H	N
Theorie	74.0	7.0	5.8
Analyse	73.9	6.8	5.7

MS (Ion trap, EI): *m/z* (%) = 243(100, [M⁺]), 200 (80), 119 (35), 83 (38), 55 (22)

3n

¹H NMR (400 MHz, CHLOROFORM-D) δ ppm 7.28 (t, *J*=7.3 Hz, 2 H) 7.18 (t, *J*=7.8 Hz, 3 H) 5.97 (d, *J*=8.3 Hz, 1 H) 5.72 - 5.81 (m, 1 H) 2.67 - 2.73 (m, 2 H) 2.65 (t, *J*=6.5 Hz, 4 H) 2.12 - 2.20 (m, 2 H) 1.92 (dq, *J*=6.7, 6.5 Hz, 2 H)

¹³C NMR (101 MHz, CHLOROFORM-D) δ ppm 171.3 (s) 141.1 (s) 131.2 (s) 128.1 (s) 125.7 (s) 120.3 (s) 34.1 (s) 32.6 (s) 29.0 (s) 16.9 (s)

%	C	H	N
Theorie	61.7	7.0	5.8
Analyse	61.4	6.8	5.7

MS (Ion trap, EI): *m/z* (%) = 244 (10, [M⁺]), 152 (100), 130 (55), 91 (24), 56 (55)

3o

^1H NMR (400 MHz, *CHLOROFORM-D*) δ ppm 7.23 - 7.30 (m, 2 H) 7.19 (d, $J=7.8$ Hz, 3 H) 5.97 (d, $J=8.6$ Hz, 1 H) 5.67 (d, $J=8.3$ Hz, 1 H) 2.88 (s, 3 H) 2.75 (t, $J=7.8$ Hz, 2 H) 2.33 (d, $J=7.3$ Hz, 2 H) 1.39 (s, 6 H)

^{13}C NMR (101 MHz, *CHLOROFORM-D*) δ ppm 174.8 (s) 154.1 (s) 141.5 (s) 130.8 (s) 128.5 (s) 128.4 (s) 126.0 (s) 117.1 (s) 61.2 (s) 34.8 (s) 29.9 (s) 24.6 (s) 22.3 (s)

%	C	H	N
Theorie	70.5	9.5	10.3
Analyse	70.2	9.3	10.1

MS (Ion trap, EI): m/z (%) = 273 (25, $[\text{M}^+]$), 181 (100), 153 (38), 72 (4), 56 (18)

3p (additional signals correspond to the minor isomer)

¹H NMR (400 MHz, *CHLOROFORM-D*) δ ppm 5.91 (d, *J*=8.6 Hz, 1 H) 5.65 (q, *J*=7.8 Hz, 1 H) 3.44 - 3.53 (m, 2 H) 2.75 (s, 4 H) 2.05 - 2.13 (m, 2 H) 1.83 (qd, *J*=6.8, 6.7 Hz, 2 H)

¹³C NMR (101 MHz, *CHLOROFORM-D*) δ ppm 175.4 (s) 131.2 (s) 118.4 (s) 44.2 (s) 31.1 (s) 28.3 (s) 25.0 (s)

%	C	H	N
Theorie	53.6	6.0	6.9
Analyse	53.8	6.1	7.1

MS (Ion trap, EI): *m/z* (%) = 202 (20, [M⁺]), 166 (48), 138 (100), 82 (45), 56 (38)

4a

^1H NMR (600 MHz, *CHLOROFORM-D*) δ ppm 6.52 (dt, $J=14.7$ Hz, 1 H) 6.36 (d, $J=14.7$ Hz, 1 H) 2.66 (s, 4 H) 2.05 (q, $J=7.2$ Hz, 2 H) 1.32 - 1.37 (m, 2 H) 1.24 - 1.31 (m, 2 H) 0.83 (t, $J=7.2$ Hz, 3 H)

^{13}C NMR (151 MHz, *CHLOROFORM-D*) δ ppm 175.4 (s) 124.5 (s) 117.8 (s) 77.2 (s) 76.8 (s) 31.2 (s) 30.5 (s) 27.6 (s) 22.0 (s) 13.7 (s)

%	C	H	N
Theorie	66.2	8.3	7.7
Analyse	66.4	8.5	7.9

MS (Ion trap, EI): m/z (%) = 182 (30, $[\text{M}^+]$), 138 (100), 100 (75), 82 (94), 56 (53)

4b

^1H NMR (600 MHz, *CHLOROFORM-D*) δ ppm 6.18 (d, $J=14.5$ Hz, 1 H) 5.94 (dt, $J=14.7$ Hz, 1 H) 2.67 (t, $J=6.6$ Hz, 4 H) 2.13 (dd, $J=7.5$, 1.1 Hz, 2 H) 1.91 - 1.96 (m, 2 H) 1.37 - 1.43 (m, 2 H) 1.30 - 1.37 (m, 2 H) 0.87 (t, $J=7.3$ Hz, 3 H)

^{13}C NMR (151 MHz, *CHLOROFORM-D*) δ ppm 172.0 (s) 131.5 (s) 119.7 (s) 33.2 (s) 31.1 (s) 30.3 (s) 22.1 (s) 16.9 (s) 13.8 (s)

%	C	H	N
Theorie	67.7	8.8	7.2
Analyse	67.7	8.8	7.5

MS (Ion trap, EI): m/z (%) = 195 (42, $[\text{M}^+]$), 138 (57), 114 (100), 86 (44), 56 (60)

4c

^1H NMR (600 MHz, *CHLOROFORM-D*) δ ppm 6.20 (d, $J=14.5$ Hz, 1 H) 5.96 (dt, $J=14.5$ Hz, 1 H) 2.52 (s, 4 H) 2.11 - 2.15 (m, 2 H) 1.37 - 1.43 (m, 2 H) 1.30 - 1.36 (m, 2 H) 1.07 (s, 6 H) 0.86 (t, $J=7.2$ Hz, 3 H)

^{13}C NMR (151 MHz, *CHLOROFORM-D*) δ ppm 171.4 (s) 131.3 (s) 119.5 (s) 46.8 (s) 31.1 (s) 30.4 (s) 29.0 (s) 27.6 (s) 22.1 (s) 13.8 (s)

%	C	H	N
Theorie	69.9	9.5	6.3
Analyse	69.5	9.4	6.4

MS (Ion trap, EI): m/z (%) = 224 (83, $[\text{M}^+]$), 180 (66), 142 (100), 83 (82), 56 (46)

4d

¹H NMR (400 MHz, *CHLOROFORM-D*) δ ppm 7.26 - 7.35 (m, 2 H) 7.22 (d, *J*=6.4 Hz, 3 H) 6.69 (dt, *J*=14.6 Hz, 1 H) 6.49 (d, *J*=14.9 Hz, 1 H) 2.73 - 2.81 (m, 2 H) 2.71 (s, 4 H) 2.41 - 2.51 (m, 2 H)

¹³C NMR (101 MHz, *CHLOROFORM-D*) δ ppm 175.3 (s) 141.2 (s) 128.3 (s) 128.3 (s) 125.9 (s) 123.5 (s) 118.4 (s) 77.3 (s) 76.7 (s) 35.6 (s) 32.8 (s) 27.7 (s)

%	C	H	N
Theorie	73.3	6.6	6.1
Analyse	73.3	6.6	6.0

MS (Ion trap, EI): *m/z* (%) = 229 (2, [*M*⁺]), 138 (100), 110 (15), 65 (18), 56 (27)

4e

^1H NMR (400 MHz, *CHLOROFORM-D*) δ ppm 7.69 (d, $^3J=14.9$ Hz, 1 H) 7.45 (s, 2 H) 7.35 (s, 2 H) 7.28 (s, 1 H) 7.20 (d, $^3J=14.9$ Hz, 1 H) 2.81 (s, 4 H)

^{13}C NMR (151 MHz, *CHLOROFORM-D*) δ ppm 175.2 (s) 135.4 (s) 128.7 (s) 127.9 (s) 126.4 (s) 121.9 (s) 117.8 (s) 27.7 (s)

%	C	H	N
Theorie	71.6	5.5	7.0
Analyse	71.99	5.55	6.85

MS (Ion trap, EI): m/z (%) = 201 (100, $[\text{M}^+]$), 172 (5), 119 (40), 89 (13), 55 (15).

2q: 1-Deuterio-1-hexyne

^1H NMR (400 MHz, *CHLOROFORM-D*) δ ppm 2.15 (t, $J=7.0$ Hz, 2 H) 1.32 - 1.55 (m, 4 H) 0.88 (t, $J=7.2$ Hz, 3 H)

^{13}C NMR (101 MHz, *CHLOROFORM-D*) δ ppm 84.0 (t) 67.7 (t) 30.6 (s) 21.8 (s) 18.0 (s) 13.4 (s)

3q: 1-[(1Z)-hex-1-en-1-yl]pyrrolidine-2,5-dione-*d*₁

¹H NMR (400 MHz, CHLOROFORM-*D*) δ ppm 5.66 (t, *J*=7.3 Hz, 1 H) 2.72 (s, 4 H) 1.88 (q, *J*=7.4 Hz, 2 H) 1.16 - 1.40 (m, 4 H) 0.81 (t, *J*=7.2 Hz, 3 H)

¹³C NMR (101 MHz, CHLOROFORM-*D*) δ ppm 175.4 (s) 133.1 (s) 116.2 (t) 30.5 (s) 28.2 (s) 27.5 (s) 22.1 (s)

4q: 1-[(1*E*)-hex-1-en-1-yl]pyrrolidine-2,5-dione-*d*₁

¹H NMR (400 MHz, CHLOROFORM-*D*) δ ppm 6.53 (t, *J*=7.3 Hz, 1 H) 2.67 (s, 3 H) 2.07 (td, *J*=7.5 Hz, 2 H) 1.22 - 1.43 (m, 4 H) 0.85 (t, *J*=7.2 Hz, 3 H)

¹³C NMR (101 MHz, CHLOROFORM-*D*) δ ppm 175.3 (s) 124.3 (s) 117.6 (t) 31.2 (s) 30.4 (s) 27.6 (s) 21.9 (s) 13.6 (s)