Supplementary Material for “Tandem Cyclopropanation/Ring-Closing Metathesis of Dienynes” by B. P. Peppers and S. T. Diver*

General Experimental

Reactions were conducted under argon atmosphere in oven dried schlenk tubes equipped with cold finger condensers and magnetic stir bar. Anhydrous solvents were obtained from a solvent purifier (alumina; alumina and Q5 for hydrocarbons) immediately before use. Products were purified by flash column chromatography on Merck silica gel 60 (230-400 mesh). EM science F254 silica plates were used for thin layer chromatography. All R_f values are reported for 1:2 ether-hexane unless otherwise noted. Compounds were visualized with ultraviolet light and/or iodine staining. Analytical capillary GC was performed on a HP-5 Crosslinked 5% PH ME Siloxane capillary column (40 °C to 200 °C over 20 min, J&W Scientific, 0.25 mm x 30 m, 0.25 mm film thickness). High resolution EI mass spectra were recorded on a VG 70-SE mass spectrometer. IR spectra were recorded on a Nicolet Impact series 420 IR. ^1H NMR and ^{13}C NMR were recorded in CDCl_3 on a Unity-500 MHz spectrometer. NMR spectra were referenced with CDCl_3 (7.24 ppm for ^1H and 77.0 ppm ^{13}C).

Experimental

Preparation of 2a and 3a: Catalyst **4** (4.2 mg, 0.005 mmol) was added to a 0.1 M solution of **1a** (30.6 mg, 0.1 mmol) in dichloromethane at 25 °C. After 2.5 hours the

reaction was purified by flashed chromatography (hexane to 1:9 ether-hexane) to give **2a** (7%, R_f 0.40), and **3a** (75%, R_f 0.29).

2a: ^1H NMR (500 MHz, CDCl_3): δ 7.66 (d, $J=10.5$ Hz, 2H), 7.30 (d, $J=10$ Hz, 2H), 5.87 (dd, $J=12.0, 3.5$ Hz), 5.47 (ddd, $J=12.0, 8.5, 3.0$ Hz, 1H), 3.68 (dd, $J=12.0, 12.0$ Hz, 2H), 2.86 (dd, $J=11.0, 11.0$ Hz, 2H), 2.41 (s, 3H), 1.98 (m, 2H), 1.74 (m, 1H), 1.27 (m, 1H), 0.89 (s, 2H). ^{13}C NMR (125 MHz, CDCl_3): δ 143.4, 133.7, 129.6, 127.5, 126.7, 124.1, 55.2, 52.8, 29.2, 25.1, 21.5, 21.1, 20.7, 17.7. FT-IR (thin film, cm^{-1}) 3029, 2926, 2850, 1598, 1445, 1347, 1167, 1101, 1026, 816, 741, 713, 665, 605, 551. High resolution ESI molecular ion calcd for $\text{C}_{16}\text{H}_{19}\text{NO}_2\text{S}$ 289.1138, found 290.1209 ($M + H$), -1.2 ppm. $M_p = 97\text{-}98^\circ\text{C}$.

3a: ^1H NMR (500 MHz, CDCl_3): δ 7.70 (d, $J=8.5$ Hz, 2H), 7.30 (d, $J=8.0$ Hz, 2H), 5.74 (m, 2H), 4.06 (s, 4H), 2.40 (s, 3H), 2.23 (m, 2H) 2.09 (m, 2H). ^{13}C NMR (125 MHz, CDCl_3): δ 143.3, 134.3, 129.7, 129.3, 128.1, 127.4, 127.0, 119.7, 56.4, 54.9, 22.7, 21.5, 20.5. FT-IR (thin film, cm^{-1}) 2923, 1343, 1165, 1098, 664. High resolution ESI molecular ion calcd for $\text{C}_{15}\text{H}_{17}\text{NO}_2\text{S}$ 275.0981, found 276.1053 ($M + H$), error -1.2 ppm. $M_p = 114\text{-}116^\circ\text{C}$ (dec).

Preparation of 2b and 3b: Catalyst **4** (8.3 mg, 0.01 mmol) was added to a 0.1 M solution of **1b** (26.4 mg, 0.1 mmol) in dichloromethane at 25°C . The reaction was filtered (2.0 x 0.5 inch plug of silica gel with 75mL of 1:1 ether-hexane) after 24 hours and concentrated. Mesitylene (4.65 μL , 0.33 mmol) was then added as an internal standard for NMR integration, which indicated **2b** (31%, R_f 0.20) and **3b** (63%, R_f 0.18).

Preparation of 2c: Catalyst **4** (4.2 mg, 0.005 mmol) was added to a 0.1 M solution of **1c** (42.8 mg, 0.1 mmol) in refluxing benzene. The reaction was purified by flash chromatography (1:2 ether-hexane) after 24 hours and concentrated to yield 34.4 mg of **2c** (83%, R_f 0.32, 1:1 ether-hexane).

^1H NMR (500 MHz, CDCl_3): δ 7.95 (dd, $J=8.5$, 0.5 Hz, 4H), 7.90 (dd, $J=8.0$, 1.0 Hz, 4H), 7.56 (m, 2H), 7.92 (m, 4H), 5.80 (dd, $J=10.0$, 2.75 Hz, 1H), 5.19 (ddd, $J=8.5$, 2.75 Hz, 1H), 2.90 (d, $J=16.0$ Hz, 1H), 2.76 (dd, $J=16.5$, 5.5 Hz 2H), 2.62 (d, $J=16.5$ Hz, 1H), 1.74 (m 2H), 1.46 (m 1H), 1.00 (m 1H), 0.92 (d, $J=4.5$ Hz, 1H), 0.88 (d, $J=5.0$ Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3): δ 137.1, 135.9, 134.5(CH), 134.4(CH), 131.7(CH), 131.2(CH), 129.2(CH), 128.8(CH), 128.6(CH), 122.1(CH), 97.3, 41.6(CH_2), 40.8(CH_2), 34.2, 29.7, 24.5(CH_2), 22.0(CH_2), 21.0(CH_2); FT-IR (thin film, cm^{-1}) 3401, 2921, 1447, 1329, 1310, 1143, 1077, 688. High resolution ESI molecular ion calcd for $\text{C}_{22}\text{H}_{22}\text{O}_4\text{S}_2$ 414.0961 found 437.0851 ($\text{M} + \text{Na}$), error -0.1 ppm. Mp 153°C (dec).

Preparation of 3c: Gallium (III) chloride (20 μL , 0.5 M) was diluted with 3 mL of benzene. This solution was added to **1c** (85.6 mg, 0.2 mmol). After 3 hours catalyst **4** (8.4 mg, 0.01 mmol) was added. After 1 hr, reaction was filtered (2.0 x 0.5 inch plug of silica gel eluted with 100 mL of 1:1 ether-hexane) and concentrated. Flash chromatography (1:4 to 1:1 ether-hexane) yielded 61.4 mg of **3c** (76%, R_f 0.21 in 1:1 ether-hexane) obtained as a white powder.

Preparation of 7c and 8c: Platinum (II) chloride (2.7 mg, 0.01 mmol) was added to a 0.1 M solution of **1c** (42.8 mg, 0.1 mmol) in benzene and refluxed. The reaction was filtered

(2.0 x 0.5 inch plug of silica gel eluted with 100 mL of 1:1 ether-hexane) after 2 hours and concentrated. Flash chromatography (1:4 to 1:1 ether-hexane) yielded **8c** (27%, R_f 0.17) and **7c** (66%, R_f 0.1) as a white powder.

7c ^1H NMR (500 MHz, CDCl_3): δ 7.97 (d, $J=7.0$ Hz, 2H), 7.67 (t, $J=7.0$ Hz, 2H), 7.53 (dd, $J=7.5$, 7.5 Hz, 4H), 6.21 (m, 1H), 5.62 (m, 1H), 4.97 (m, 4H), 3.46 (s, 2H), 3.34 (s, 2H), 1.89 (m, 4H); ^{13}C NMR (125 MHz, CDCl_3): δ 136.9, 136.1, 134.5, 134.4, 132.0, 131.4, 128.6, 128.6, 96.1, 42.3, 41.1, 31.3, 30.3, 22.4, 20.6, 18.0, 14.6, 11.2, 6.2. FT-IR (thin film cm^{-1}) 3432, 3068, 2925, 1447, 1329, 1312, 1078, 912, 729, 688. High resolution ESI molecular ion calcd for $\text{C}_{23}\text{H}_{24}\text{O}_4\text{S}_2$ 428.1117, found 451.1008 ($\text{M} + \text{Na}$), error -0.7 ppm. Mp 103-104 $^\circ\text{C}$.

8c ^1H NMR (500 MHz, CDCl_3): δ 8.11 (d, $J=7.0$ Hz, 2H), 8.03 (d, $J=7.0$ Hz, 2H), 7.70 (m, 2H), 7.59 (m, 4H), 2.91 (d, $J=16.0$ Hz, 1H), 2.85 (d, 16.5 Hz, 1H), 2.68 (d, 16.0 Hz, 1H), 2.62 (d, 16.0 Hz, 1H), 1.71 (m, 2H), 1.59 (m, 1H), 1.48 (m, 1H), 1.17 (m, 1H), 0.80 (m, 1H), 0.71 (m, 2H), 0.33 (m, 2H). ^{13}C NMR (125 MHz, CDCl_3): δ 137.2, 136.3, 134.7(CH), 132.3(CH), 131.7(CH), 128.9(CH), 128.8(CH), 96.4, 42.6(CH_2), 41.3(CH_2), 31.5, 30.6, 22.7(CH_2), 20.8(CH_2), 18.3(CH_2), 14.9(CH), 11.4(CH), 6.4(CH_2). FT-IR (thin film, cm^{-1}) 3073, 3001, 2919, 2856, 1447, 1325, 1310, 1144, 1077, 728, 688. High resolution ESI molecular ion calcd for $\text{C}_{23}\text{H}_{24}\text{O}_4\text{S}_2$ 428.1117, found 451.1020 ($\text{M} + \text{Na}$), error 2.6 ppm. Mp 188-189 $^\circ\text{C}$.

7c was also prepared using similar procedures as above with RuCl_3 (24 hours reaction time; this reaction also produced **8c**) and GaCl_3 (0.25M solution, 6 hours reaction time, catalyst was added from a pre-made stock solution of 0.5M GaCl_3 in

methylcyclohexane). The benzene and methylcyclohexane in the GaCl₃ reactions were dried additionally by refluxing over sodium and subsequent distillation.

Preparation of 9 and 10: Platinum (II) chloride (2.7 mg, 0.01 mmol) was added to a 0.1 M solution of **1a** (30.3 mg, 0.1 mmol) in benzene and refluxed. The reaction was filtered (2.0 x 0.5 inch plug of silica gel eluted with 75 mL of 1:1 ether-hexane) after 3 hours and concentrated. Flash chromatography (1:4 to 1:1 ether-hexane) yielded **9** (75%, R_f 0.56) and **10** (19%, R_f 0.41) as clear and colorless oils.

9: ¹H NMR (500 MHz CDCl₃): δ 7.62 (d, J=8.5 Hz, 2H), 7.29 (d, J=8.0 Hz, 2H), 6.27 (d, J=8.0 Hz, 1H), 5.73 (m, 1H), 5.38 (dd, J=8.0, 5.5 Hz, 1H), 4.96 (dd, J=17.0, 2.0 Hz, 1H), 4.91 (dd, J=10.0, 1.0 Hz, 1H), 3.85 (d, J=12.0 Hz, 1H), 2.78 (d, J=11.0 Hz, 1H), 2.40 (s, 3H), 2.07 (m, 2H), 1.58 (m, 1H), 1.24 (m, 1H), 0.92 (m, 1H), 0.632 (ddd, J=8.0, 4.0, 1.0 Hz, 1H), 0.48 (dd, J=4.5, 4.5 Hz, 1H). ¹³C NMR (125 MHz, CDCl₃): δ 143.6, 138.0(CH), 134.9, 129.7(CH), 127.0(CH), 120.6(CH), 114.8(CH₂), 112.7(CH), 44.4(CH₂), 35.9(CH₂), 30.6(CH₂), 29.4, 21.5(CH₃), 19.4(CH₂), 15.2(CH). FT-IR (thin film, cm⁻¹) 2922, 1641, 1352, 1168, 684. High resolution ESI molecular ion calcd for C₁₇H₂₁NO₂S 303.1294 found 326.1183 (M + Na), error -0.5 ppm.

10: ¹H NMR (500 MHz, CDCl₃): δ 7.64 (d, J=8.5 Hz, 2H), 7.30 (d, J=8.0 Hz, 2H), 3.59 (d, J=9.0 Hz, 1H), 3.43 (d, J=9.5 Hz, 1H), 2.97 (d, J=9.0 Hz, 1H), 2.66 (d, J=8.0 Hz, 1H), 2.42 (s, 3H), 1.69 (m, 2H), 1.58 (m, 1H), 1.27 (m, 1H), 1.07 (m, 1H), 0.86 (m, 1H), 0.66 (d, J=5.0 Hz, 1H), 0.47 (d, J=5.0 Hz, 1H), 0.36 (m, 1H), -0.09 (m, 1H). ¹³C NMR (125 MHz, CDCl₃): δ 143.2, 134.0, 129.5, 127.4, 55.7, 54.0, 26.8, 25.3, 21.5, 20.4, 17.6, 15.5, 11.5, 11.6, 11.0, 6.8. FT-IR (thin film, cm⁻¹) 3070, 3003, 2930,

2863, 1598, 1454, 1343, 1170, 1104, 1034, 820, 661, 603, 547. Low resolution ESI molecular ion calcd for $C_{17}H_{21}NO_2S$ 303.1294, found 304.1 (M + H), error 122.9 ppm.

Preparation of 11a: NaH (206 mg, 8.58 mmol, 60% dispersion in mineral oil) was added to a solution of 2-[*p*-toluenesulfonamido)methyl]-1,5-hexadiene (1.752 g 6.60 mmol) in 22 mL of THF in small portions over 5 minutes. The solution was stirred at room temperature for 1 h, there upon 1-bromo-2-butyne (2.02 mL, 23.10 mmol) was added drop wise by syringe. The reaction was placed in a 65 °C oil bath and heated for 5 hours. The reaction was quenched with saturated aqueous ammonium chloride solution, extracted with dichloromethane, washed with brine, dried ($MgSO_4$), filtered, concentrated and purified by flash chromatography (hexane to 1:10 ether-hexane) to give 1.370 g of **11a** (66%, R_f 0.49) as a clear and pale yellow oil.

1H NMR (500 MHz, $CDCl_3$): δ 7.73 (d, $J=8.0$ Hz, 2H), 7.28 (d, $J=8.0$ Hz, 2H), 5.78 (m, 1H), 5.00 (m, 4H) 3.96 (d, $J=2.5$ Hz, 2H), 3.70 (s, 1H), 2.40 (s, 3H), 2.23 (m, 2H), 2.14 (m, 2H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 143.2, 142.6, 138.0, 136.1, 129.1, 128.0, 114.9, 114.7, 81.6, 71.4, 51.1, 36.0, 32.2, 31.5, 21.5, 3.2. FT-IR (thin film, cm^{-1}) 3076, 2920, 1642, 1599, 1440, 1348, 1165, 1094, 909, 762, 710, 659, 570. High resolution ESI molecular ion calcd for $C_{18}H_{23}NO_2S$ 317.1451, found 318.1522 (M + H), error -1.5 ppm.

Preparation of 11b: NaH (260 mg, 6.5 mmol, 60% dispersion mineral oil) was added to a solution of bis-(phenylsulfonyl)methane (1.752 g, 6.5 mmol) in 10 mL of DMF in small portions over 5 minutes. The solution was stirred at room temperature for five minutes

where upon 1-bromo-2-butyne (0.55 mL, 6.25 mmol) was added dropwise with a syringe. The reaction was placed in a 70 °C oil bath and heated for 15 hours. The reaction was quenched with saturated aqueous ammonium chloride solution, extracted with dichloromethane, washed with brine, dried (MgSO₄), filtered, concentrated, and purified by flash chromatography (hexane to 1:2.5 ethyl acetate-hexane) to give 1.28 g 1,1-bis(phenylsulfonyl)-pent-3-yne (63%) as a white powder. Tetrakis(triphenylphosphine)palladium (52 mg, 0.045 mmol) was added to a room temperature solution of 1,1-(bisphenyl sulfonyl)-pent-3-yne (786 mg, 2.26 mmol), stirred at room temperature and methyl 2-methylene-5-hexenyl ester carbonic acid (424 mg, 2.48 mmol) in THF (4.0 mL). The resulting solution was placed in a 55 °C oil bath, stirred for 12 hours, cooled, concentrated and flash purified by chromatography (hexane to 1:1 ether-hexane) to give 952 mg **11b** (95%, R_f 0.26) as a white powder.

¹H NMR (500 MHz, CDCl₃): δ 8.00 (d, J=8.0 Hz, 4H), 7.64 (t, J=7.5 Hz, 2H), 7.51 (dd, J= 8.0, 8.0 Hz, 4H), 5.77 (m, 1H), 5.26 (s, 1H), 5.07 (s, 1H), 4.98 (dd, J=17.5, 2.0 Hz, 1H), 4.92 (d, J=10.5 Hz, 1H), 3.33 (d, J=2.5 Hz, 2H), 3.06 (s, 2H), 2.33 (t, J=7.5 Hz, 2H), 2.18 (m 2H). ¹³C NMR (125 MHz, CDCl₃): δ 141.2, 138.0, 137.7, 134.4, 131.5, 128.3, 117.8, 114.6, 90.6, 82.6, 72.0, 36.1, 34.8, 32.4, 22.4, 3.5. FT-IR (thin film, cm⁻¹) 2915, 1447, 1335, 1312, 1146, 1077, 689. High resolution ESI molecular ion calcd for C₂₄H₂₆O₄S₂ 442.1274, found 465.1167 (M + Na), error 0.4 ppm. Mp 96 °C.

Preparation of 12a: Catalyst **4** (4.2 mg, 0.005 mmol) was added to a 0.1 M solution of **11a** (31.6 mg, 0.1 mmol) in dichloromethane at room temperature and refluxed for 50

minutes. Flash chromatography (hexane to 1:9 ether-hexane) yielded 22.2 mg (70%) of **12a** (70%, R_f 0.43) as clear pale yellow oil.

^1H NMR (500 MHz, CDCl_3): δ 7.69 (d, $J=8.5\text{Hz}$, 2H), 7.29 (d, $J=8\text{Hz}$, 2H), 5.96 (t, $J=8.0\text{Hz}$, 1H), 5.00 (s, 1H), 4.87 (s, 2H), 4.78 (s, 1H), 4.00 (s, 2H), 3.72 (s, 2H), 2.53 (m, 2H), 2.41 (s, 3H), 2.26 (t, $J=6.5\text{Hz}$, 2H), 1.84 (s, 3H). ^{13}C NMR (125 MHz, CDCl_3): δ 144.1, 143.1, 141.9, 136.6, 135.5, 129.6(CH), 129.4(CH), 127.3(CH), 119.3(CH_2), 112.3(CH_2), 52.0(CH_2), 45.7(CH_2), 36.8(CH_2), 25.9(CH_2), 21.5(CH_3), 21.4(CH_3). FT-IR (thin film, cm^{-1}) 2925, 1599, 1450, 1333, 1094, 1040, 905, 815, 661. High resolution ESI molecular ion calcd for $\text{C}_{18}\text{H}_{23}\text{NO}_2\text{S}$ 317.1451, found 318.1522 ($\text{M} + \text{H}$), error 0.4 ppm. Further 2D ^1H NMR, HSQC, and ω -refocused 1,1-ADEQUATE provided proof of structure. The full details will be represented in the full paper.

Preparation of 12b: Catalyst **4** (4.2 mg, 0.005 mmol) was added to a 0.1 M solution of **11b** (44.2 mg, 0.1 mmol) in benzene at room temperature and refluxed for 24 hours. Purification by flash chromatography (hexane to 1:2 ether-hexane) yielded 36.2 mg of **12b** (82%, R_f 0.20) as a white powder.

^1H NMR (500 MHz, CDCl_3): δ 7.99 (d, $J=1.5\text{ Hz}$, 4H), 7.67 (t, $J=7.5\text{ Hz}$, 2H), 7.54 (dd, $J=8.0, 8.0\text{ Hz}$, 4H), 5.92 (t, $J=6.0\text{ Hz}$, 1H), 5.12 (s, 1H), 5.10 (s, 1H), 4.99 (s, 2H), 3.66 (s, 2H), 3.23 (s, 2H), 2.28 (m, 4H), 1.89 (s, 3H). ^{13}C NMR (125 MHz, CDCl_3): δ 144.5, 142.3, 137.8, 134.4, 133.5, 131.4, 130.6, 128.5, 119.5, 112.6, 93.8, 35.3, 34.8, 29.3, 27.0, 22.4. FT-IR (thin film, cm^{-1}) 2930, 1447, 1334, 1311, 1144, 1077, 689. High resolution ESI molecular ion calcd for $\text{C}_{24}\text{H}_{26}\text{O}_4\text{S}_2$ 442.1274, found 465.1165 ($\text{M} + \text{Na}$), error 2.9 ppm. Mp 191-192 $^\circ\text{C}$ (dec).

Preparation of 13: Tetrakis(triphenylphosphine)palladium (27 mg, 0.023 mmol) was added to a room temperature solution of 1,1-bis(phenylsulfonyl)-3-butyne (390 mg, 1.17 mmol), and carbonic acid methyl ester 2-methylene-hexyl ester (215 mg, 1.26 mmol) in THF (2.0 mL). The resulting solution was placed in a 55 °C oil bath, stirred for 12 hours, cooled, concentrated, and purified by flash chromatography (hexane to 1:4 ethyl acetate-hexane) to give **13** (93%, R_f 0.2) as a white powder.

^1H NMR (500 MHz, CDCl_3): δ 8.04 (d, $J=8.5$ Hz, 4H), 7.68 (t, $J=7.5$ Hz, 2H), 7.53 (dd, $J=8.0, 8.0$ Hz, 4H), 5.20 (s, 1H), 5.06 (s, 1H), 3.36 (d, $J=3.0$ Hz, 2H), 3.11 (s, 2H), 2.19 (t, $J=7.0$ Hz, 2H), 1.98 (t, $J=2.5$ Hz, 1H). ^{13}C NMR (125 MHz, CDCl_3): δ 142.0, 137.2, 134.7, 131.7, 128.5, 117.2, 90.4, 75.1, 36.9, 34.8, 30.4, 22.3, 22.1, 14.0. FT-IR (thin film, cm^{-1}) 3272, 2956, 1448, 1335, 1312, 1146, 1077, 735, 687. High resolution ESI molecular ion calcd for $\text{C}_{23}\text{H}_{26}\text{O}_4\text{S}_2$ 430.1268, found 430.1267, error 0.3 ppm. Mp 135-136°C (dec).

Preparation of 14: Catalyst **4** (4.2 mg, 0.005 mmol) was added to a 0.1 M solution of **13** (43.0 mg, 0.1 mmol) in benzene at room temperature and refluxed for 24 h. Flash chromatography (hexane to 1:1 ethyl acetate-hexane) yielded a 1:1.5 (*syn:anti*) mixture of diastereomers 85% of **14** (85% combined yield, R_f 0.29) as a white powder. The diastereomeric mixture was dissolved in a 0.25 mL dichloromethane and crystallized in an atmosphere saturated with hexane vapor. This process was repeated five times to give only the *anti*-diastereomer.

Anti-14: ^1H NMR (500 MHz, CDCl_3): δ 7.93 (d, $J=7.5$ Hz, 8H), 7.61 (t, $J=7.5$ Hz, 2H), 7.54 (t, $J=7.0$ Hz, 2H), 7.48 (m, 8H), 5.05 (s, 2H), 2.80 (s, 1H), 2.77 (s, 1H), 2.64 (m, 4H), 1.21 (m, 10H), 0.85 (d, $J=4.5$ Hz, 2H). ^{13}C NMR (125 MHz, CDCl_3): δ 137.2, 136.1, 135.0(CH), 134.9(CH), 132.0(CH), 131.5(CH), 129.8, 129.1(CH), 128.6(CH), 95.9, 40.4(CH_2), 40.3(CH_2), 39.2, 37.4, 32.4(CH_2), 30.3(CH_2), 25.4(CH_2), 23.0(CH_2), 14.3(CH_3). FT-IR (thin film, cm^{-1}) 2954, 2931, 2857, 1448, 1327, 1311, 1145, 1078, 912, 729, 689, 609. High resolution ESI molecular ion calcd for $\text{C}_{46}\text{H}_{52}\text{O}_8\text{S}_4$ 860.2548, found 883.2437, error -1.2 ppm ($\text{M} + \text{Na}$). Mp 180°C (dec).