Supplementary Material (ESI) for Chemical Communications

Supporting Information

belonging to

Rhodium-mediated functionalization of an unactivated C_{alkyl}–H Bond

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Experimental Procedures

General comments. The N-alkylated imidazoles[Si] and compound **4** [Sii] were prepared according to literature procedures. All other reagents are commercially available and were used as received. Unless specified otherwise, NMR spectra were recorded at 25 °C on Bruker spectrometers operating at 400 or 500 MHz (^{1}H NMR) and 100 or 125 MHz (^{13}C{^{1}H} NMR), respectively. Chemical shifts (δ in ppm, coupling constants J in Hz) were referenced to residual solvent resonances. Assignments are based on homo- and heteronuclear shift correlation spectroscopy. Elemental analyses were performed by the Microanalytical Laboratory at the Federal Institute of Technology in Zurich, Switzerland and at University College Dublin, Ireland.

General procedure for the synthesis of the diimidazolium salts 1a-b. The respective 1,2-diimidazoles were stirred together with 1,3-dibromopropane (0.5 mol equiv.) in refluxing toluene (20 mL) for 16 hours. The formed precipitate was isolated by centrifugation, washed with toluene and recrystallized from CH_2CH_2/Et_2O.

***N*,*N*-Propylenedi(*N*'-isopropyl-2-methyl)imidazolium dibromide (1a).** According to the general procedure, starting from 2-methyl *N*-isopropyl imidazole (2.82 g, 22.7 mmol) and 1,3-dibromopropane (2.28 g, 11.3 mmol). Compound **1a** was obtained as an off-white solid (2.89 g, 57%).
^{1}H NMR (CD_3CN, 500 MHz): δ 7.88 (d, 2H, $^3J_{HH}$ = 2.3 Hz, H_{imi}), 7.43 (d, 2H, $^3J_{HH}$ = 2.3 Hz, H_{imi}), 4.59 (septet, 2H, $^3J_{HH}$ = 6.5 Hz, NC*H*$(CH_3)_2$), 4.40 (t, 4H, $^3J_{HH}$ = 8.0 Hz, NC*H*$_2$CH$_2$C*H*$_2$N), 2.70 (s, 6H, $C_{imi}CH_3$), 2.34 (quintet, 2H, $^3J_{HH}$ = 8.0 Hz, NCH$_2$C*H*$_2$CH$_2$N), 1.46 (d, 12H, $^3J_{HH}$ = 6.5 Hz, NCH(C*H*$_3$)$_2$). ^{13}C{^{1}H} NMR (CD_3CN, 125 MHz): δ 144.6 (NCN), 122.9 (C_{imi}), 118.4 (C_{imi}), 51.7 (N*C*H$(CH_3)_2$), 45.8 (N*C*H$_2$CH$_2$*C*H$_2$N), 30.6 (NCH$_2$*C*H$_2$CH$_2$N), 22.4 (NCH(*C*H$_3$)$_2$), 10.9 (C_{imi}*C*H$_3$). Anal. calc. for $C_{17}H_{30}Br_2N_4$ (450.26) × H_2O: C, 43.60; H, 6.89; N, 11.96. Found: C, 43.37; H, 6.56; N, 11.62.

***N*,*N*-Propylenedi(*N*'-*n*-butyl-2-methyl)imidazolium dibromide (1b).** According to the general procedure, starting from 2-methyl *N*-*n*-butyl imidazole (3.55 g, 25.7 mmol) and 1,3-dibromopropane (2.59 g, 12.6 mmol). Compound **1b** was obtained as a white solid (5.56 g, 90%).
^{1}H NMR (CD_3CN, 500 MHz): δ 7.74 (d, 2H, $^3J_{HH}$ = 2.3 Hz, H_{imi}), 7.34 (d, 2H, $^3J_{HH}$ = 2.3 Hz, H_{imi}), 4.34 (t, 4H, $^3J_{HH}$ = 8.0 Hz, NC*H*$_2$CH$_2$C*H*$_2$N), 4.04 (t, 4H $^3J_{HH}$ = 7.5 Hz, NC*H*$_2$CH$_2$CH$_2$CH$_3$), 2.64 (s, 6H, $C_{imi}CH_3$), 2.31 (quintet, 2H, $^3J_{HH}$ = 8.0 Hz, NCH$_2$C*H*$_2$CH$_2$N), 1.76 (quintet, 4H, $^3J_{HH}$ = 7.5 Hz, NCH$_2$C*H*$_2$CH$_2$CH$_3$), 1.36 (sextet, 4H, $^3J_{HH}$ = 7.5 Hz, NCH$_2$CH$_2$C*H*$_2$CH$_3$), 0.95 (t, 6H, $^3J_{HH}$ = 7.5 Hz, NCH$_2$CH$_2$CH$_2$C*H*$_3$). ^{13}C{^{1}H} NMR (CD_3CN, 125 MHz): δ 145.2 (NCN), 122.3 (C_{imi}), 122.1 (C_{imi}), 48.9 (N*C*H$_2$CH$_2$CH$_2$CH$_3$), 45.8 (N*C*H$_2$CH$_2$*C*H$_2$N), 32.1 (NCH$_2$*C*H$_2$CH$_2$CH$_3$), 30.5 (NCH$_2$*C*H$_2$CH$_2$N), 20.1 (NCH$_2$CH$_2$*C*H$_2$CH$_3$), 13.7 (NCH$_2$CH$_2$CH$_2$*C*H$_3$), 10.6 (C_{imi}*C*H$_3$). Anal. calc. for $C_{19}H_{34}N_4Br_2$ (478.31): C, 47.71; H, 7.16; N, 11.71. Found: C, 47.58; H, 7.19; N, 11.42.

General procedure for the synthesis of [RhI(*C*,*C*,*C*-dicarbene)(NCMe)$_2$]I (2a-b). and [(μ-I)$_3$-{Rh(*C*,*C*,*C*-dicarbene)}$_2$]I (3a-b). The corresponding diimidazolium salt **1**, $RhCl_3.H_2O$ (1 mol equiv.) and NaOAc (8 mol equiv.) were stirred together in refluxing acetonitrile (30 mL) for 16 hours, after which KI (4 mol equiv.) was added and stirring at reflux continued for a further hour. The reaction mixture was cooled to room temperature

and all volatiles were removed under reduced pressure. The residue was purified by column chromatography (SiO_2; CH_2Cl_2/acetone, 5:2).

Synthesis of 2a and 3a. According to the general procedure, starting from **1a** (450 mg, 1.0 mmol), $RhCl_3.H_2O$ (263 mg, 1.0 mmol), NaOAc (656 mg, 8.0 mmol) and KI (664 mg, 4.0 mmol). After column chromatography **3a** was obtained as a light brown solid (380 mg, 59%). Dissolving **3a** in CH_3CN and subsequent precipitation with Et_2O yielded complex **2a**. Analytically pure material was obtained by the slow diffusion of Et_2O into a solution of **2a** in CH_3CN.

Anal. calc. for $C_{21}H_{33}I_2N_6Rh$ (726.25) × H_2O: C, 33.89; H, 4.74; N, 11.29. Found: C, 34.11; H, 4.49; N, 11.02.

Spectroscopic data for 2a.

Two species are distinguishable in the NMR spectra in a 2.5:1 ratio:

Major species: ^{1}H NMR (CD_3CN, 500 MHz): δ 6.78 (br, 2H, H_{imi}), 4.28 (septet, 2H, $^3J_{HH}$ = 6.5 Hz, NC*H*$(CH_3)_2$), 4.17 (m, 2H, NC*H*$_2$CHC*H*$_2$N), 3.74 (m, 3H, RhCH, NC*H*$_2$CHC*H*$_2$N) 2.39 (s, 6H, $C_{imi}CH_3$), 1.37, 1.28 (2 × d, 6H, $^3J_{HH}$ = 6.5 Hz, NCH(C*H*$_3$)$_2$). ^{13}C{^{1}H} NMR (CD_3CN, 125 MHz): δ 138.3 (s, NCN), 119.5 (br, C_{imi}), 58.5 (s, NC*H*$_2$CH*C*H$_2$N), 49.7 (s, N*C*H$(CH_3)_2$), 33.7 (d, $^1J_{RhC}$ = 28.5 Hz, RhCH), 22.8, 22.6 (2 × s, NCH(*C*H$_3$)$_2$), 11.4 (s, C_{imi}*C*H$_3$).

Minor species: ^{1}H NMR (CD_3CN, 500 MHz): δ 6.67 (s, 2H, H_{imi}), 4.34 (septet, 2H, $^3J_{HH}$ = 6.5 Hz, NC*H*$(CH_3)_2$), 4.17 (m, 2H, NC*H*$_2$CHC*H*$_2$N), 3.92 (br, 3H, RhCH, NC*H*$_2$CHC*H*$_2$N) 2.42 (s, 6H, $C_{imi}CH_3$), 1.40, 1.31 (2 × d, 6H, $^3J_{HH}$ = 6.5 Hz, NCH(C*H*$_3$)$_2$). ^{13}C{^{1}H} NMR (CD_3CN, 125 MHz): δ 144.6 (d, $^1J_{RhC}$ = 45.8 Hz, C_{imi}), 139.2 (s, NCN), 116.3 (d, $^2J_{RhC}$ = 3.2 Hz, C_{imi}), 58.2 (br, NC*H*$_2$CH*C*H$_2$N), 50.0 (s, N*C*H$(CH_3)_2$), 38.8 (br, RhCH), 22.9, 22.6 (2 × s, NCH(*C*H$_3$)$_2$), 11.5 (s, C_{imi}*C*H$_3$).

Spectroscopic data for 3a.

Three sets of signals are distinguishable in the NMR spectra in approximate 1:1:1 ratio. Two of these sets have been assigned to the *anti* eclipsed conformation (C_2 symmetry), one set to the *syn* eclipsed conformer (C_{2v} symmetry).

^{1}H NMR (CD_2Cl_2, 500 MHz): δ 6.72, 6.65, 6.64 (3 × s, 4H, H_{imi}), 4.48-4.14 (m, 12H, RhCH, NC*H*$_2$CHC*H*$_2$N, NC*H*CH$_3$), 3.79 (m, 4H, NC*H*$_2$CHC*H*$_2$N), 2.47, 2.44, 2.43 (3 × s, 12H, $C_{imi}CH_3$), 1.46, 1.45, 1.43, 1.35, 1.33, 1.32 (6 × d, 24H, $^3J_{HH}$ = 6.5 Hz, NCH(C*H*$_3$)$_2$). ^{13}C{^{1}H} NMR (CD_2Cl_2, 125 MHz): δ 152.4, 152.3, 152.0 (3 × d, $^1J_{RhC}$ = 51.0 Hz, C_{imi}), 136.4, 136.3, 136.2 (3 × s, NCN), 116.2, 116.0, 115.9 (3 × d, $^2J_{RhC}$ = 3.8 Hz, C_{imi}), 60.5, 60.4, 60.2 (3 × s, NC*H*$_2$CH*C*H$_2$N), 49.6 (s, N*C*H$(CH_3)_2$), 38.1, 38.0 (2 × d, $^1J_{RhC}$ = 30.4 Hz, RhCH), 22.9, 22.9, 22.8, 22.7, 22.6 (5 × s, NCH(*C*H$_3$)$_2$), 12.0, 12.0, 11.9 (3 × s, C_{imi}*C*H$_3$).

Synthesis of 2b and 3b. According to the general procedure, starting from **1b** (478 mg, 1.0 mmol), $RhCl_3.H_2O$ (263 mg, 1.0 mmol), NaOAc (656 mg, 8.0 mmol) and KI (664 mg, 4.0 mmol). After column chromatography **3b** was obtained as a brown solid (480 mg, 71%). Analytically pure material was obtained by slow diffusion of Et_2O into a solution of **3b** in CH_2Cl_2 and acetone (1:1).

Anal. calc. for $C_{38}H_{62}I_4N_8Rh_2$ (1344.38): C, 33.95; H, 4.65; N, 8.33. Found: C, 34.02; H, 4.85; N, 8.22.

Dissolving **3b** in CH_3CN and subsequent precipitation with Et_2O yielded complex **2b**.

Spectroscopic data for 2b.

Two species can be distinguished in the ^1H NMR spectrum in a 3:1 ratio.

Major species: ^1H NMR (CD_3CN, 500 MHz): δ 6.72 (br, 2H, H_{imi}), 4.18 (m, 2H, NCH_2CHCH_2N), 3.79 (m, 7H, RhCH, NCH_2CHCH_2N, $NCH_2CH_2CH_2CH_3$), 2.37 (s, 6H, $C_{imi}CH_3$), 1.61 (quintet, 4H, $^3J_{HH}$ = 7.5 Hz, $NCH_2CH_2CH_2CH_3$), 1.24 (sextet, 4H, $^3J_{HH}$ = 7.5 Hz, $NCH_2CH_2CH_2CH_3$), 0.88 (t, 6H, $^3J_{HH}$ = 7.5 Hz, $NCH_2CH_2CH_2CH_3$). $^{13}C\{^1H\}$ NMR (CD_3CN, 125 MHz): δ 139.0 (s, NCN), 121.2 (s, C_{imi}), 58.6 (s, NCH_2CHCH_2N), 47.5 (s, $NCH_2CH_2CH_2CH_3$), 33.7 (d, $^1J_{RhC}$ = 28.0 Hz, RhCH), 32.8 (s, $NCH_2CH_2CHCH_3$), 20.2 (s, $NCH_2CH_2CH_2CH_3$), 13.8 (s, $NCH_2CH_2CH_2CH_3$), 11.3 ($C_{imi}CH_3$).

Minor species: ^1H NMR (CD_3CN, 500 MHz): δ 6.63 (s, 2H, H_{imi}), 4.18 (br, 2H, NCH_2CHCH_2N), 3.93 (br, 1H, RhCH), 3.79 (m, 6H, NCH_2CHCH_2N, $NCH_2CH_2CH_2CH_3$), 2.40 (s, 6H, $C_{imi}CH_3$), 1.63 (quintet, 4H, $^3J_{HH}$ = 7.5 Hz, $NCH_2CH_2CH_2CH_3$), 1.25 (sextet, 4H, $^3J_{HH}$ = 7.5 Hz, $NCH_2CH_2CH_2CH_3$), 0.89 (t, 6H, $^3J_{HH}$ = 7.5 Hz, $NCH_2CH_2CH_2CH_3$). $^{13}C\{^1H\}$ NMR (CD_3CN, 125 MHz): δ 144.4 (d, $^1J_{RhC}$ = 45.5 Hz, C_{imi}), 140.0 (s, NCN), 120.6 (s, C_{imi}), 58.3 (br, NCH_2CHCH_2N), 47.7 (s, $NCH_2CH_2CH_2CH_3$), 39.0 (br, RhCH), 32.7 (s, $NCH_2CH_2CH_2CH_3$), 20.2 (s, $NCH_2CH_2CH_2CH_3$), 13.8 (s, $NCH_2CH_2CH_2CH_3$), 11.3 ($C_{imi}CH_3$).

Spectroscopic data for 3b.

Three sets of signals are distinguishable in the NMR spectra in approximate 1:1:1 ratio. Two of these sets have been assigned to the *anti* eclipsed conformation (C_2 symmetry), one set to the *syn* eclipsed conformer (C_{2v} symmetry).

^1H NMR (CD_2Cl_2, 500 MHz): δ 6.64, 6.57 (2 × s, 4H, H_{imi}), 4.49-4.25 (m, 18H, RhCH, NCH_2CHCH_2N, $NCH_2CH_2CH_2CH_3$), 2.45, 2.42 (2 × s, 12H, $C_{imi}CH_3$), 1.67 (m, 8H, $NCH_2CH_2CH_2CH_3$), 1.28 (m, 8H, $NCH_2CH_2CH_2CH_3$), 0.91 (m, 12H, $NCH_2CH_2CH_2CH_3$). $^{13}C\{^1H\}$ NMR (CD_2Cl_2, 125 MHz): δ 152.6, 152.5, 152.4 (3 × d, $^1J_{RhC}$ = 50.3 Hz, C_{imi}), 137.4, 137.3 (2 × s, NCN), 120.9, 120.8 (2 × s, C_{imi}), 60.7, 60.6 (2 × s, NCH_2CHCH_2N), 47.7 (s, $NCH_2CH_2CH_2CH_3$), 38.2 (d, $^1J_{RhC}$ = 30.2 Hz, RhCH), 32.6 (s, $NCH_2CH_2CHCH_3$), 20.3 (s, $NCH_2CH_2CH_2CH_3$), 13.9 (s, $NCH_2CH_2CH_2CH_3$), 12.0 ($C_{imi}CH_3$).

Synthesis of 5. According to the general procedure described for **2a-b**, starting from **4** (48 mg, 0.1 mmol), $RhCl_3.H_2O$ (26 mg, 0.1 mmol), NaOAc (66 mg, 0.8 mmol) and KBr (48 mg, 0.4 mmol). After column chromatography the product was dissolved in CH_3CN and precipitated with Et_2O. **5** was obtained as a yellow solid (13 mg, 21%). Analytically pure material was obtained by slow diffusion of Et_2O into a solution of **5** in CH_3CN and acetone (1:1).

^1H NMR (DMSO–d_6, 500 MHz): δ 7.61 (d, 2H, $^3J_{HH}$ = 2.5 Hz, H_{imi}), 7.43 (d, 2H, $^3J_{HH}$ = 2.5 Hz, H_{imi}) 4.61–4.55 (m, 2H, $NCH_2CH_2CH_2CH_3$), 4.35–4.29 (m, 2H, $NCH_2CH_2CH_2CH_3$), 3.94–3.91 (m, 2H, $NCH_2CH_2CH_2N$), 3.55–3.49 (m, 2H, $NCH_2CH_2CH_2N$), 1.83-1.71 (m, 6H, $NCH_2CH_2CH_2N$, $NCH_2CH_2CH_2CH_3$), 1.40–1.34 (m, 4H, $NCH_2CH_2CH_2CH_3$), 0.93 (m, 6H, $NCH_2CH_2CH_2CH_3$). $^{13}C\{^1H\}$ NMR (DMSO–d_6, 125 MHz): δ 186.5 (s, $OOCCH_3$), 147.7 (d, $^1J_{RhC}$ = 46.5 Hz, C_{imi}), 124.2 (s, NCN), 123.2 (s, C_{imi}), 50.0 (s, $NCH_2CH_2CH_2CH_3$), 44.8 (s, $NCH_2CH_2CH_2N$), 33.6 (s, $NCH_2CH_2CHCH_3$), 31.8 (s, $NCH_2CH_2CH_2N$), 24.5 (s, $OOCCH_3$), 19.4 (s,

$NCH_2CH_2CH_2CH_3$), 13.8 (s, $NCH_2CH_2CH_2CH_3$). Anal. calc. for $C_{19}H_{31}Br_2N_4O_2Rh$ (610.19): C, 37.40; H, 5.12; N, 9.18. Found: C, 37.32; H, 5.02; N, 8.98.

Synthesis of 6. A suspension of complex **3a** (97 mg, 75 μmol) and $AgBF_4$ (58 mg, 0.30 mmol) was stirred at room temperature for 16 hours. The reaction mixture was filtered over Celite and the solvent removed *in vacuo*, yielding the title product as a dark red solid (101 mg, 97%).

^{1}H NMR (CD_3CN, 360 MHz): δ 6.64 (s, 2H, H_{imi}), 4.31 (septet, 2H, $^3J_{HH}$ = 6.8 Hz, $NCH(CH_3)_2$), 4.15 (m, 2H, NCH_2CHCH_2N), 3.67 (m, 3H, RhCH, NCH_2CHCH_2N) 2.38 (s, 6H, $C_{imi}CH_3$), 1.39, 1.29 (2 × d, 6H, $^3J_{HH}$ = 6.8 Hz, $NCH(CH_3)_2$). ^{13}C{^{1}H} NMR (CD_3CN, 125 MHz): δ 144.6 (d, $^1J_{RhC}$ = 45.5 Hz, C_{imi}), 139.2 (s, NCN), 116.2 (d, $^2J_{RhC}$ = 3.3 Hz, C_{imi}), 58.5 (s, NCH_2CHCH_2N), 50.0 (s, $NCH(CH_3)_2$), 33.6 (d, $^1J_{RhC}$ = 28.6 Hz, RhCH), 22.7, 22.5 (2 × s, $NCH(CH_3)_2$), 11.1 (s, $C_{imi}CH_3$). Anal. calc. for $C_{21}H_{35}B_2F_8N_6ORh$ (664.06): C, 37.98; H, 5.31; N, 12.66. Found: C, 38.19; H, 5.31; N, 12.94.

General procedure for hydrogenation reactions. A mixture of cyclooctene (1 mmol), catalyst (10 μmol Rh), and solvent (5 mL) was placed in a glass tube inside an autoclave. A pressure of 60 bar H_2 was applied and the autoclave placed in a preheated oil bath. At the relevant time, the autoclave was removed from the oil bath and cooled under a stream of running water after which the pressure was slowly released. A sample was taken and filtered over a short path of silica. The filtrate was subjected to GC analysis.

For reactions carried out at atmospheric pressure, a mixture of cyclooctene (1 mmol), catalyst (10 μmol Rh) and solvent (5 mL) was placed in a round bottomed flask. The reaction mixture was stirred at the appropriate temperature while H_2 was bubbled through the solution for 5 minutes. The reaction mixture was kept under H_2 pressure using a balloon. At the relevant time a sample was taken, filtered through a short path of silica and subjected to GC analysis.

Structure determination and refinement of 2a and 3a

Suitable single crystals were mounted on a Stoe Imaging Plate Diffractometer System[Siii] equipped with a one-circle φ gionometer and a graphite monochromator. Data collection was performed at -100(2) °C using Mo-Kα radiation (λ = 0.71073 Å). The resolution was D_{min} - D_{max} 12.45 - 0.81 Å. **3a** crystallized in an orthorhombic cell (Pnma) and **2a** in a non-centrosymmetric orthorhombic cell ($Pca2_1$). The absolute structure was determined for **2a** (Flack parameter x = -0.08(3)).

Both structures were solved by direct methods using the program SHELXS-97[Siv] and refined by full matrix least squares on F^2 with SHELXL-97.[Sv] The hydrogen atoms were included in calculated positions and treated as riding atoms using SHELXL-97 default parameters. All non-hydrogen atoms were refined anisotropically. A semi-empirical absorption correction was applied using MULscanABS in PLATON03.

3a produced pronounced disorder in co-crystallised hexane molecules. The SQUEEZE option in PLATON was used to calculate the potential solvent accessible volume. 1320 Å^3 was calculated corresponding to about 247 electrons. Four hexane molecules (4 x 50 electrons) per unit cell were thus included in all further calculations.

Further details on data collection and refinement parameters are summarized in Table S1. Crystallographic data (excluding structure factors) have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication nos. CCDC 743284 and 743285. Copies of the data can be obtained free of charge on

application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK [fax: +44-1223-336-033; e-mail: deposit@ccdc.cam.ac.uk].

Table S1. Crystallographic data for complexes 2a and 3a

	2a	**3a**
color, shape	orange, plate	orange, rod
cryst size /mm	0.5, 0.3, 0.15	0.5, 0.3, 0.2
refined formula	$C_{21}H_{33}I_2N_6Rh$	$C_{40}H_{68}I_4N_8Rh_2$
Fw	726.24	1374.44
crystal system	orthorhombic	orthorhombic
space group	*P*ca2$_1$ (No. 29)	*P*nma (No. 62)
unit cell		
a /Å	13.2142(12)	15.2889(13)
b /Å	13.7680(13)	15.9079(13)
c /Å	15.0238(10)	20.8008(19)
V /Å^3	2733.2(4)	5059.1(8)
Z	4	4
D_{calc}/g cm^{-3}	1.765	1.805
μ /mm^{-1}	2.902	3.128
no. of total, unique reflections	18938, 4629	36742, 5201
R_{int}	0.0593	0.1149
transmission range	0.492–0.554	0.358–0.514
no. parameters, restraints	255, 1	140, 0
R,[a] R_w[b]	0.0374, 0.0846	0.0751, 0.1913
GOF	0.985	0.844
min., max. residual density /e Å^{-3}	–1.467, 1.099	–1.722, 1.850

[*a*] $R_1 = \Sigma||F_O|-|F_C||/\Sigma|F_O|$ for all $I > 2\sigma(I)$; [*b*] $wR_2 = [\Sigma w(F_O^2-F_C^2)^2/\Sigma(w(F_O^4)]^{1/2}$.

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