

*SUPPORTING INFORMATION*

# Fast Initiating Furan-Containing Hoveyda-Type Complexes: Synthesis and Applications in Metathesis Reactions

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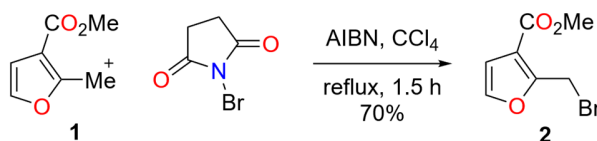
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## 1. General Remarks

All materials were purchased from commercial suppliers and used as received, unless otherwise noted. All reactions requiring the exclusion of oxygen and moisture were carried out in dry glassware using dry solvents under a dry and oxygen-free atmosphere using the Schlenk technique. The bottles with ruthenium catalysts were stored under argon atmosphere, but no special precautions were taken to avoid air or moisture exposure at the moment of extracting catalysts from the bottles. NMR ( $^1\text{H}$  and  $^{13}\text{C}$ ) spectra were recorded on an Agilent Mercury 400 MHz spectrometer at ambient temperature with  $\text{CDCl}_3$  used as a solvent. Chemical shifts ( $\delta$ ) are given in parts per million (ppm) downfield from tetramethylsilane (TMS) with a residual non-deuterated solvent peak used as a reference:  $\text{CDCl}_3$  ( $\delta \text{ H} = 7.26 \text{ ppm}$ ,  $\delta \text{ C} = 77.16 \text{ ppm}$ ). The coupling constants ( $J$ ) are reported in hertz (Hz) and refer to  $H,H$ -couplings. The following abbreviations are used in order to indicate the multiplicity of the signal: s (singlet), d (doublet), t (triplet), q (quartet), p (pentet), sex (sextet), h (heptet), dd (doublet of doublet), dt (doublet of triplet), ddd (doublet of doublet of doublet), etc., bs (broad signal), m (multiplet). The data obtained were processed with the software MestReNova. HRMS measurements were carried out using AutoSpec Premier spectrometer using electrospray (ESI) as ionisation method at the Polish Academy of Sciences, Institute of Organic Chemistry. IR spectra were recorded on a Perkin-Elmer Spectrum One FTIR spectrometer. The obtained data were processed with the software Omni32. Wavenumbers are given in  $\text{cm}^{-1}$ . The EA were carried out at the Polish Academy of Science, Institute of Organic Chemistry. Column Chromatography was performed using Merck Millipore silica gel (60, particle size 0.043 – 0.063 nm). Thin Layer Chromatography (TLC) was performed using Merck Silica Gel 60 F254 precoated aluminum sheets. Substances were visualized using UV-light (254 or 365 nm).

## 2. Preparation of ligand precursor and catalysts

### 2.1. Procedure for the synthesis of methyl (2-bromomethyl)-3-furancarboxylate (2)



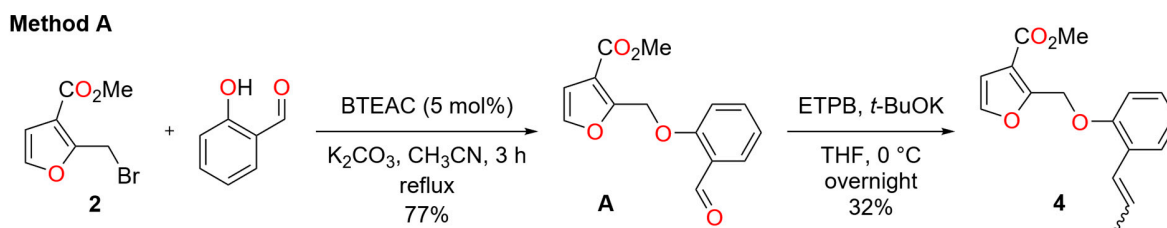
To a refluxed solution of methyl 2-methyl-3-furancarboxylate (**1**, 2.5 g, 17.8 mmol) and AIBN (33 mg, 0.2 mmol) in CCl<sub>4</sub> (25 mL) *N*-bromo succinimide (3.23 g, 17.8 mmol) was added portion wise during the period of 30 min. After the addition was completed, the resulting mixture was refluxed for 1 h and cooled down to room temperature. Floating succinimide was filtered off and washed with CCl<sub>4</sub> (3 x 5 mL). The filtrate was concentrated under vacuum. The residue was recrystallized in hexane (15 mL) leading to the desired product as colorless crystals (2.7 g, 13.3 mmol, 70%).

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):**  $\delta$  (ppm) 7.38 (d,  $J$  = 2.0 Hz, 1H), 6.69 (d,  $J$  = 2.0 Hz, 1H), 4.80 (s, 2H), 3.86 (s, 3H).

**<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>):**  $\delta$  (ppm) 163.3, 155.5, 142.9, 115.9, 111.6, 52.0, 21.3. Spectral data are in agreement with those reported in the literature [1].

### 2.2 Procedure for the synthesis of 2-((2-(prop-1-en-1-yl)phenoxy)methyl)furan-3-carboxylate (4)

#### 2.2.1. Method A



##### 2.2.1.1 Synthesis of methyl 2-((2-formylphenoxy)methyl)furan-3-carboxylate (A)

To the solution of salicylaldehyde (0.13 mL, 1.2 mmol) in CH<sub>3</sub>CN (6 mL) benzyl triethylammonium chloride (11.5 mg, 0.05 mmol) and potassium carbonate (279 mg, 2 mmol) were added. The resulting mixture was stirred at reflux for 20 min. Then, methyl (2-bromomethyl)-3-furancarboxylate (**2**, 219 mg, 1 mmol) dissolved in CH<sub>3</sub>CN (4 mL) was added dropwise. The resulting mixture was stirred at reflux for the next 2 h (TLC control,

20% EtOAc/ *n*-hexane), cool down to the room temperature, and extracted with EtOAc (3 x 10 mL). The combined organic layers were dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, and concentrated under vacuum. The product **A** was isolated by column chromatography (5% EtOAc/*n*-hexane) as a colorless powder (200 mg, 0.77 mmol, 77%).

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):**  $\delta$  (ppm) 10.47 (s, 1H, CHO), 7.84 (dd,  $J$  = 2.0, 7.4 Hz, 1H), 7.55 – 7.54 (m, 1H), 7.43 (d,  $J$  = 2.0 Hz, 1H), 7.18 (d,  $J$  = 7.4 Hz, 1H), 7.07 (t,  $J$  = 7.4 Hz, 1H), 6.74 (d,  $J$  = 2.0 Hz, 1H), 5.50 (s, 2H), 3.86 (s, 3H).

**<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>):**  $\delta$  (ppm) 189.9, 163.6, 160.8, 154.7, 143.2, 136.0, 128.5, 125.6, 121.7, 117.2, 113.6, 111.1, 61.8, 52.0.

**HRMS (ESI)** Calcd. for [M + Na]<sup>+</sup> C<sub>14</sub>H<sub>12</sub>O<sub>5</sub>Na<sup>+</sup>: 283.0582, found: 283.0575.

**ATR-IR (cm<sup>-1</sup>):** 3148, 3097, 2944, 2889, 1701, 1661, 1587, 1499, 1468, 1405, 1310, 1256, 1070, 1034, 981, 952, 846, 735, 628, 474.

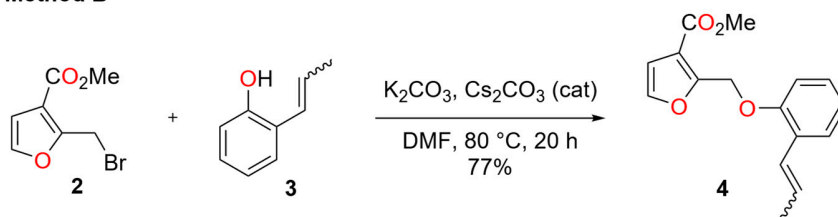
**EA** Calcd. for C<sub>14</sub>H<sub>12</sub>O<sub>5</sub>: C, 64.61; H, 4.65. Found: C, 64.66; H, 4.65.

### 2.2.1.2 Synthesis of 2-((2-(prop-1-en-1-yl)phenoxy)methyl)furan-3-carboxylate (**4**)

To the solution of ETPB (150 mg, 0.4 mmol) in dry THF (5 mL) at 0 °C potassium *tert*-butoxide (45.8 mg, 0.4 mmol) dissolved in THF (2 mL) was added dropwise. After the addition was completed, the resulting mixture was stirred for the next 40 min at room temperature. Then, compound **A** (80 mg, 0.3 mmol) dissolved in THF (3 mL) was slowly added to the mixture at 0 °C. The resulting yellow solution was further stirred at room temperature overnight (TLC control, 20% EtOAc/*n*-hexane) and extracted with EtOAc (3 x 10 mL). The combined organic layers were dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> and concentrated under vacuum. The product was isolated using column chromatography (5% EtOAc/*n*-hexane) as a colorless oil (34 mg, 0.13 mmol, 32%).

### 2.2.2. Method B

#### Method B



To a suspension of  $\text{K}_2\text{CO}_3$  (954; 6.9 mmol) and  $\text{Cs}_2\text{CO}_3$  (378 mg; 1.2 mmol) in DMF (20 mL), 2-propenylphenol (463 mg, 3.5 mmol) was added. After stirring for 30 min at room temperature, methyl (2-bromomethyl)-3-furancarboxylate (**1**, 504 mg, 2.3 mmol) was added and the reaction mixture was stirred for 20 h at 80 °C. The resulting mixture was poured into 40 mL of water and extracted three times with diethyl ether (3 x 20 mL). The combined extracts were washed with brine, water, and dried. The solvent was evaporated, the crude product was isolated using column chromatography (3% EtOAc/hexane) as a colorless oil (480 mg, 1.8 mmol, 77%, *E/Z* = 1:3).

**$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) of the major isomer:**  $\delta$  (ppm) 7.40 (d, *J* = 2.0 Hz, 1H), 7.28 (dd, *J* = 2.0, 7.4 Hz, 1H), 7.23 – 7.18 (m, 1H), 7.04 (d, *J* = 7.4 Hz, 1H), 6.97 (m, 1H), 6.72 (d, *J* = 2.0 Hz, 1H), 6.55 (dd, *J* = 2.0, 11.7 Hz, 1H), 5.85 – 5.77 (m, 1H), 5.38 (s, 2H), 3.84 (s, 3H), 1.87 (dd, *J* = 2.0, 7.4 Hz, 3H).

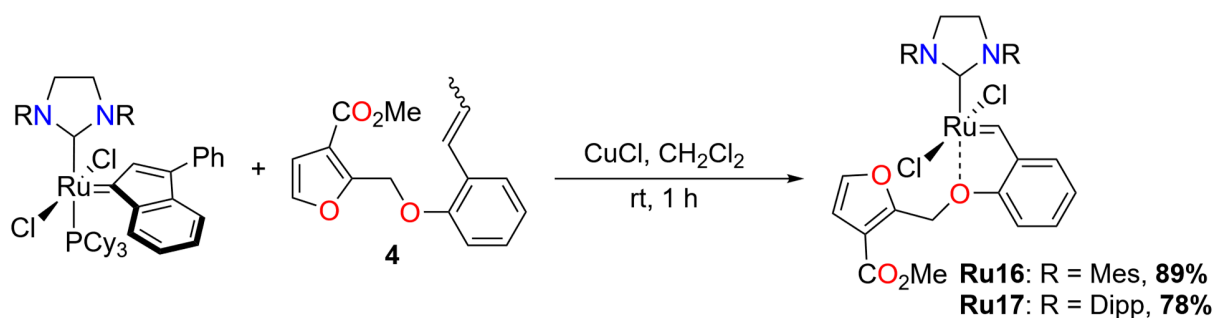
**$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ) of the major isomer:**  $\delta$  (ppm) 163.8, 156.0, 155.8, 142.8, 130.4, 128.0, 127.3, 127.2, 125.1, 121.1, 116.6, 113.1, 111.0, 61.9, 51.9, 14.8.

**HRMS (ESI)** Calcd. For  $[\text{M} + \text{Na}]^+ \text{C}_{16}\text{H}_{16}\text{O}_4\text{Na}^+$ : 295.0946, found: 295.0941.

**ATR-IR ( $\text{cm}^{-1}$ ):** 3155, 3130, 3032, 2952, 2853, 1719, 1597, 1486, 1440, 1311, 1235, 1197, 1088, 1011, 967, 745.

**EA** Calcd. for  $\text{C}_{16}\text{H}_{16}\text{O}_4$ : C, 70.58; H, 5.92; Found: C, 70.69; H, 5.92.

## 2.3. Procedure for the synthesis of ruthenium complexes



### 2.3.1. Preparation of Ru16

Indenylidene ruthenium complex (R = Mes) (106 mg, 0.12 mmol) dissolved in dry  $\text{CH}_2\text{Cl}_2$  (2 mL), 2-((2-(prop-1-en-1-yl)phenoxy)methyl)furan-3-carboxylate (**4**, 40 mg, 0.15 mmol), and CuCl (13.4 mg, 0.13 mmol) were added to a Schlenk flask equipped with a magnetic stirring bar under argon atmosphere. The reaction mixture was stirred at room temperature

for 1 h and monitored by TLC (30% EtOAc/*n*-hexane). After full conversion of starting material solvent was removed under reduced pressure and crude product was purified by flash chromatography (from 3% EtOAc/*n*-hexane to 30% EtOAc/*n*-hexane) to give product **Ru16** as a green solid (72 mg, 0.1 mmol, 89%).

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):**  $\delta$  (ppm) 16.54 (s, 1H), 7.52 (ddd,  $J$  = 8.6, 7.1, 1.9 Hz, 1H), 7.17 (d,  $J$  = 8.6 Hz, 1H), 7.09 (br.s, 4H), 7.05 (d,  $J$  = 1.9 Hz, 1H), 6.94 – 6.86 (m, 2H), 6.59 (d,  $J$  = 1.9 Hz, 1H), 5.65 (s, 2H), 4.09 (s, 4H), 3.78 (s, 3H), 2.46 – 2.44 (m, 18H).

**<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>):**  $\delta$  (ppm) 292.1, 210.0, 163.5, 152.9, 152.4, 144.4, 143.4, 138.8, 138.7, 136.9, 129.9, 129.8, 123.8, 122.5, 117.8, 113.2, 110.7, 62.2, 52.0, 51.8, 21.3, 19.4.

**MS (ES):** For [M-Cl]<sup>+</sup> C<sub>35</sub>H<sub>38</sub>ClN<sub>2</sub>O<sub>4</sub>Ru: 687.15.

**ATR-IR (cm<sup>-1</sup>):** 2971, 2935, 2875, 1752, 1654, 1553, 1464, 1336, 1220, 1178, 1120, 1058, 1004, 928, 881, 816, 773, 677, 632, 554, 449.

**EA Calcd.** for C<sub>35</sub>H<sub>38</sub>Cl<sub>2</sub>N<sub>2</sub>O<sub>4</sub>Ru: C, 58.17; H, 5.30; N, 3.88; Found: C, 55.31; H, 5.30; N, 3.60.

### 2.3.2. Preparation of Ru17

Indenylidene ruthenium complex (R = Dipp) (116 mg, 0.12 mmol) dissolved in dry CH<sub>2</sub>Cl<sub>2</sub> (2 mL), 2-((2-(prop-1-en-1-yl)phenoxy)methyl)furan-3-carboxylate (**4**, 40 mg, 0.15 mmol), and CuCl (13.4 mg, 0.13 mmol) were added to a Schlenk flask equipped with a magnetic stirring bar under argon atmosphere. The reaction mixture was stirred at room temperature for 1 h and monitored by TLC (30% EtOAc/*n*-hexane). After full conversion of starting material solvent was removed in vacuo and crude product was purified by flash chromatography (3% EtOAc/*n*-hexane → 30% EtOAc/*n*-hexane) to give product **Ru17** as a green solid (75 mg, 0.1 mmol, 78%).

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):**  $\delta$  (ppm) 16.34 (s, 1H), 7.55 (t,  $J$  = 7.7 Hz, 2H), 7.46 (ddd,  $J$  = 8.6, 7.7, 1.8 Hz, 1H), 7.39 (d,  $J$  = 7.7 Hz, 4H), 7.20 (d,  $J$  = 8.6 Hz, 1H), 6.98 (d,  $J$  = 2.0 Hz, 1H), 6.89 – 6.79 (m, 2H), 6.55 (d,  $J$  = 2.0 Hz, 1H), 5.70 (s, 2H), 4.14 (s, 4H), 3.76 (s, 3H), 3.60 (p,  $J$  = 6.7 Hz, 4H), 1.27 – 1.22 (m, 24H).

**<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>):**  $\delta$  (ppm) 287.2, 213.1, 163.6, 153.5, 153.0, 149.2, 143.7, 143.6, 137.4, 129.7, 129.7, 124.7, 123.7, 122.1, 117.1, 113.5, 110.6, 62.7, 54.8, 51.9, 34.3, 28.8, 26.5, 23.9, 22.5, 14.2.

**HRMS (ESI)** Calcd. For [M-Cl]<sup>+</sup> C<sub>41</sub>H<sub>50</sub>ClN<sub>2</sub>O<sub>4</sub>Ru: 771.2503, found: 771.2499.

**ATR-IR (cm<sup>-1</sup>):** 2963, 2926, 2866, 1716, 1661, 1593, 1510, 1474, 1454, 1442, 1407, 1317, 1261, 1233, 1193, 1159, 1089, 1035, 973, 804, 746, 645, 554, 458.

**EA Calcd.** for C<sub>41</sub>H<sub>50</sub>Cl<sub>2</sub>N<sub>2</sub>O<sub>4</sub>Ru: C, 61.04; H, 6.25; N, 3.47; **Found:** C, 61.18; H, 6.52; N, 3.49.

### 3. Catalytic activity assessment

#### 3.1 Procedure for the RCM of diethyl diallylmalonate (**5**)

Schlenk flask was charged under inert atmosphere with diethyl diallylmalonate (**5**) (152 mg, 0.62 mmol, 1 equiv.) and *n*-dodecane (internal standard, 28 µL, 21.1 mg, 0.12 mmol, 0.2 equiv.) followed by 30 mL of DCM. To the resulting mixture, a stock solution of the catalyst in DCM (1 mol%, 0.0062 mmol in 1 mL of DCM) was added. The solution was stirred at 0 °C and samples of the solution (200 µL) were withdrawn from the reaction after given time. The samples were placed in a GC-vial, quenched with a solution of SnatchCat [2] (4.4 equiv. vs the catalyst). Obtained samples were submitted to the GC analysis. Conversion of substrate was determined by substrate/product ratio using calibrated method.

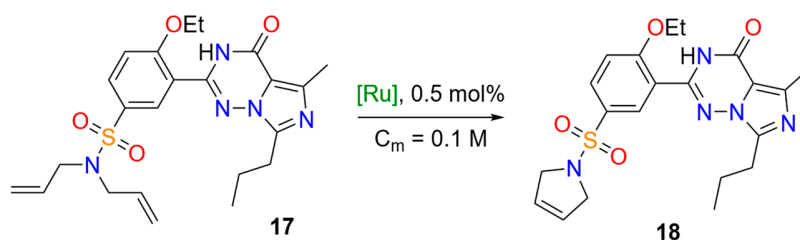
#### 3.2 General procedure for RCM and ene-yne reactions

Comparative experiments with model substrates were performed in a dry DCM under argon at room temperature with an initial concentration of substrates *c* = 0.1 M and catalyst loading 0.2, 0.5, or 1 mol%. To a stirred solution of the substrate (1 equiv.) in a dry DCM, a solution of catalyst (**Ru10**, **Ru16** or **Ru17**) in dry DCM was added in a single portion. The reaction mixture was stirred for an appropriate time at the same temperature. In case of substrates **7** and **11** aliquots (0.1 mL), taken in regular intervals, were quenched immediately with an ice-cold solution of SnatchCat (0.1 mL) and analyzed by GC. The reaction mixture after RCM of **9** was treated with SnatchCat, the solvent was evaporated, and the residue was purified on column chromatography.

#### 3.3 General procedure for CM reactions

A comparative experiments with the model substrates (were performed in dry DCM under argon at a given temperature with an initial concentration of **13** or **15** *c* = 0.1 M and a catalyst loading 1 mol%. To a stirred solution of **13** or **15** (1 equiv.) and *cis*-1,4-diacetoxy-2-butene or methyl acrylate (2 equiv.) in dry DCM catalyst (**Ru2**, **trans-Ru6**, **cis-Ru6**) in dry DCM was added in a single portion at RT or 30 °C. The reaction mixture was stirred for an appropriate time at a given temperature. The reaction mixtures were treated with SnatchCat, the solvent was evaporated, and the residue was purified on column chromatography.

### 3.4 RCM of Vardenafil derivative 17



Vardenafil derivative **17** (117 mg, 0.25 mmol, 1 equiv.) was dissolved in anhydrous CH<sub>2</sub>Cl<sub>2</sub> (2.5 mL) under argon atmosphere. The corresponding Ruthenium complex (for **Ru16**: 0.75 mg, 0.0012 mmol, 0.5 mol%; for **Ru17**: 1 mg, 0.0012 mmol, 0.5 mol%) was added to the solution of **17**. The resulting mixture was stirred at 40 °C for 20 h and the reaction progress was monitored by TLC (80% EtOAc/*n*-hexane). After the reaction completion, the mixture was quenched using SnatchCat [2] (4.4 equiv. vs the catalyst). All volatiles were removed under reduced pressure and the crude product was purified using column chromatography (60% EtOAc/*n*-hexane). The product **18** was obtained as a white solid.

The procedure was repeated using anhydrous DMC and toluene as a solvent for the RCM reaction of Vardenafil derivative **17** with **Ru17** (see Table 1).

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)**  $\delta$  (ppm) 9.70 (s, 1H), 8.54 (t, *J* = 2.3 Hz, 1H), 7.95 (dd, *J* = 2.3, 8.8 Hz, 1H), 7.16 (d, *J* = 8.8 Hz, 1H), 5.70 (s, 2H), 4.35 – 4.30 (m, 2H), 4.18 – 4.16 (m, 4H), 2.99 (t, *J* = 7.7 Hz, 2H), 2.63 (s, 3H), 1.87 (h, *J* = 7.4 Hz, 2H), 1.58 (t, *J* = 7.7 Hz, 3H), 1.02 (t, *J* = 7.4 Hz, 3H).

**<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)**:  $\delta$  (ppm) 159.9, 154.8, 146.5, 144.7, 140.5, 132.2, 131.1, 129.8, 125.6, 118.8, 113.8, 113.4, 66.2, 55.1, 28.1, 21.1, 14.7, 14.6, 14.1. Spectral data are in agreement with those reported in the literature [3].

**Table S1.** Conditions of the RCM reaction of Vardenafil derivative **17**

Entry	[Ru]	Solvent	T [°C]	t [h]	Yield of <b>18</b> [%]
1	<b>Ru16</b>	DCM	40	20	45
2	<b>Ru17</b>	DCM	40	20	37
3	<b>Ru17</b>	DMC	65	20	64
4	<b>Ru17</b>	Toluene	100	4	77
5	<b>Ru17<sup>a</sup></b>	Toluene	100	4	91

<sup>a</sup> 2 mol% of catalyst was used.

#### 4. Crystallographic data

The data were collected using the BRUKER KAPPA APEXII ULTRA controlled by APEXII software [4], equipped with MoK $\alpha$  rotating anode X-ray source ( $\lambda = 0.71073 \text{ \AA}$ , 50.0 kV, 22.0 mA) monochromatized by multi-layer optics and APEX-II CCD detector. The experiments were carried out at 100K using the Oxford Cryostream cooling device. The crystal was mounted on thin cactus needle with a droplet of Pantone-N oil and immediately cooled. Indexing, integration and initial scaling were performed with *SAINT* [5] and *SADABS* [6] software (Bruker, 2007). The data collection and processing statistics are reported in Table 1.

The crystal was positioned at 50 mm from the CCD camera. 1295 frames were measured at  $0.5^\circ$  intervals with a counting time of 5-15 sec.

The structures were solved by direct methods approach using the SHELXS-97 [7] program and refined with the SHELXL-97 [8]. Multi-scan absorption correction have been applied in the scaling procedure. The refinement was based on  $F^2$  for all reflections except those with negative intensities. Weighted R factors wR and all goodness-of-fit S values were based on  $F^2$ , whereas conventional R factors were based on the amplitudes, with  $F$  set to zero for negative  $F^2$ . The  $F_0^2 > 2\sigma (F_0^2)$  criterion was applied only for R factors calculation was not relevant to the choice of reflections for the refinement. The R factors based on  $F^2$  are for all structures about twice as large as those based on  $F$ . The hydrogen atoms were located in idealized geometrical positions, except hydrogen in solvent molecule. Scattering factors were taken from Tables 4.2.6.8 and 6.1.1.4 from the International Crystallographic Tables Vol.C [9].

**Table S2.** Experimental details

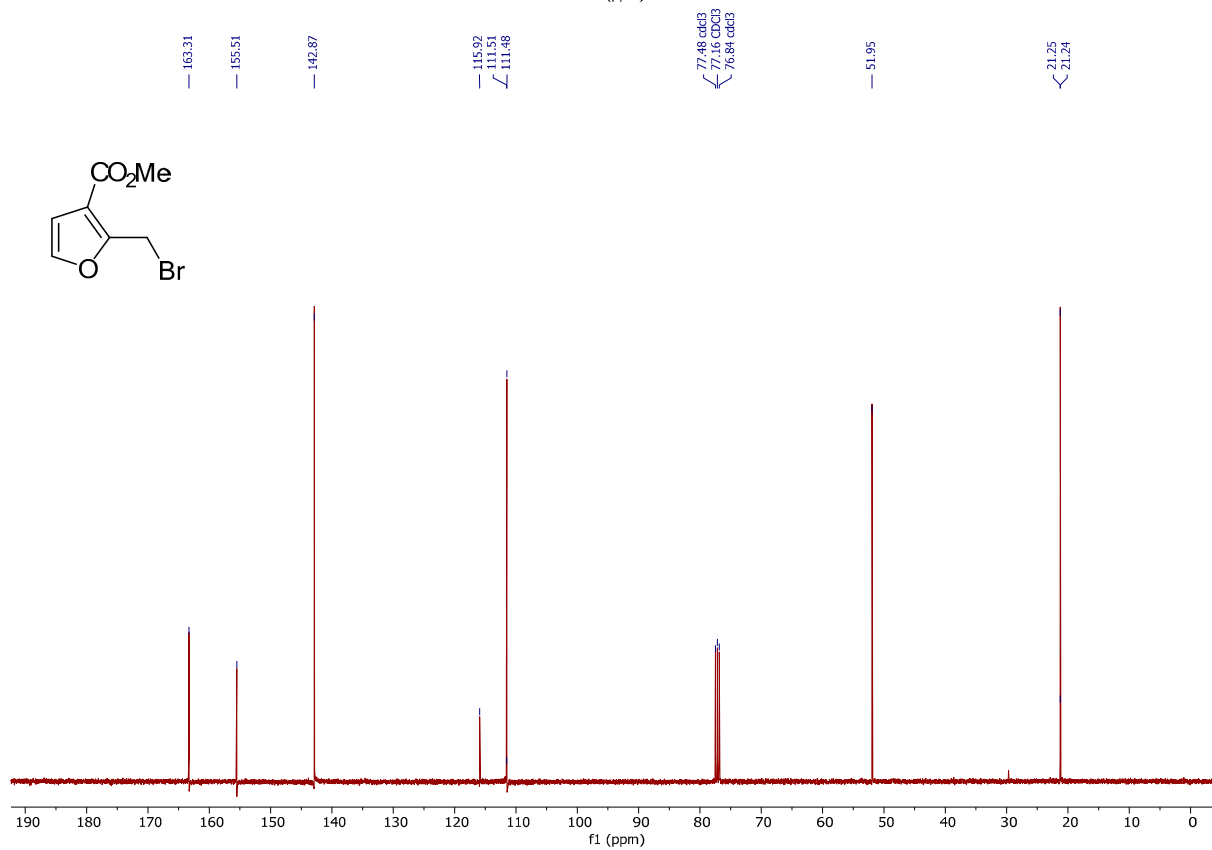
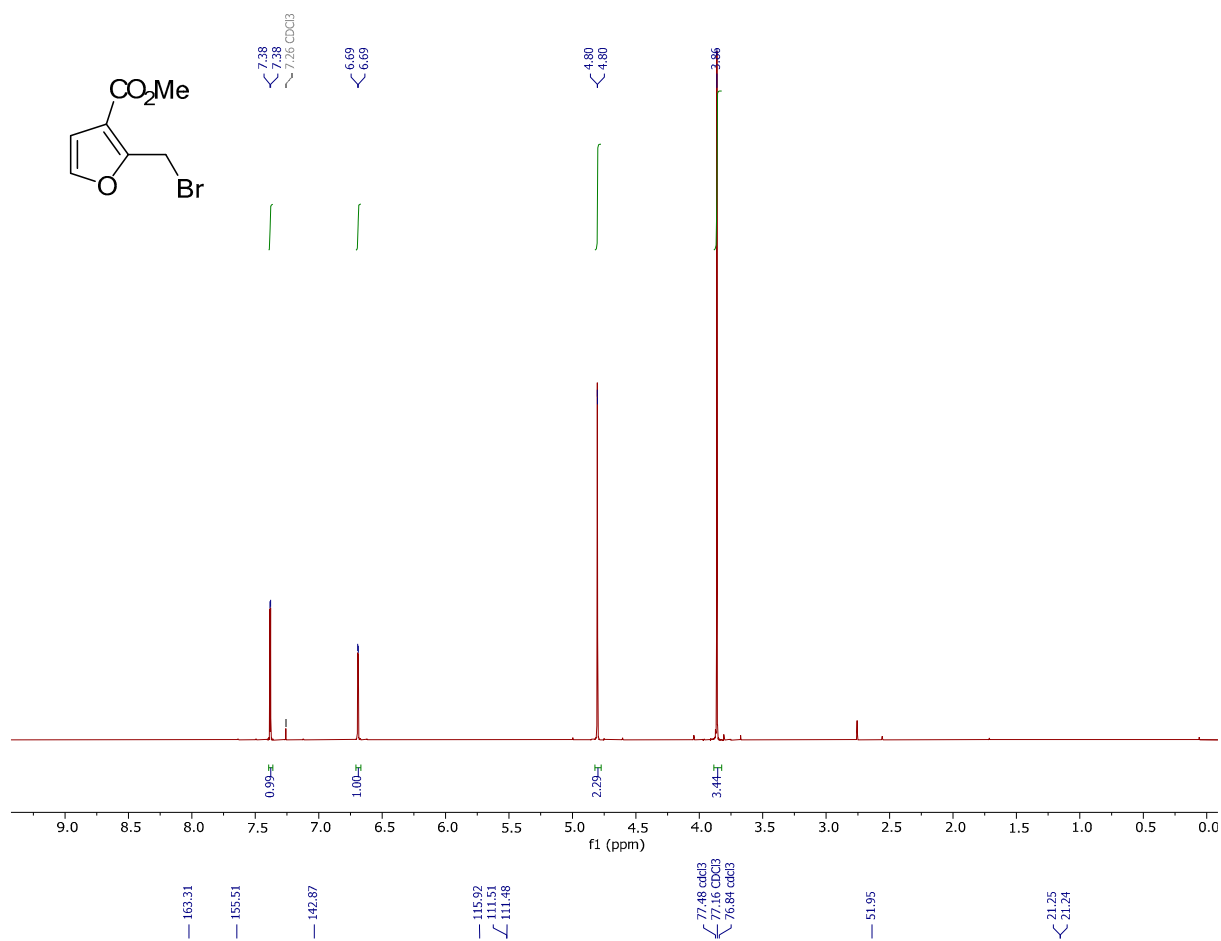
	<i>SHELXL</i>
Crystal data	
Chemical formula	C <sub>35</sub> H <sub>38</sub> Cl <sub>2</sub> N <sub>2</sub> O <sub>4</sub> Ru
$M_r$	722.64
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	100
$a, b, c$ (Å)	20.1261 (10), 8.6362 (5), 20.4281 (11)
$\beta$ (°)	114.248 (3)

$V (\text{\AA}^3)$	3237.4 (3)
$Z$	4
Radiation type	Mo $K\alpha$
$\mu (\text{mm}^{-1})$	0.69
Crystal size (mm)	0.30 × 0.10 × 0.05
Data collection	
Diffractometer	Kappa ApexII Ultra CCD
Absorption correction	Multi-scan <i>SADABS2004/1</i> - Bruker Nonius area detector scaling and absorption correction
$T_{\min}, T_{\max}$	0.820, 0.966
No. of measured, independent and observed [ $I > 2\sigma(I)$ ] reflections	32905, 7429, 6387
$R_{\text{int}}$	0.030
$(\sin \theta/\lambda)_{\max} (\text{\AA}^{-1})$	0.649
Refinement	
$R[F^2 > 2\lambda(F^2)], wR(F^2), S$	0.026, 0.063, 1.02
No. of reflections	7429
No. of parameters	407
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\max}, \Delta\rho_{\min} (\text{e}\text{\AA}^{-3})$	0.68, -0.39

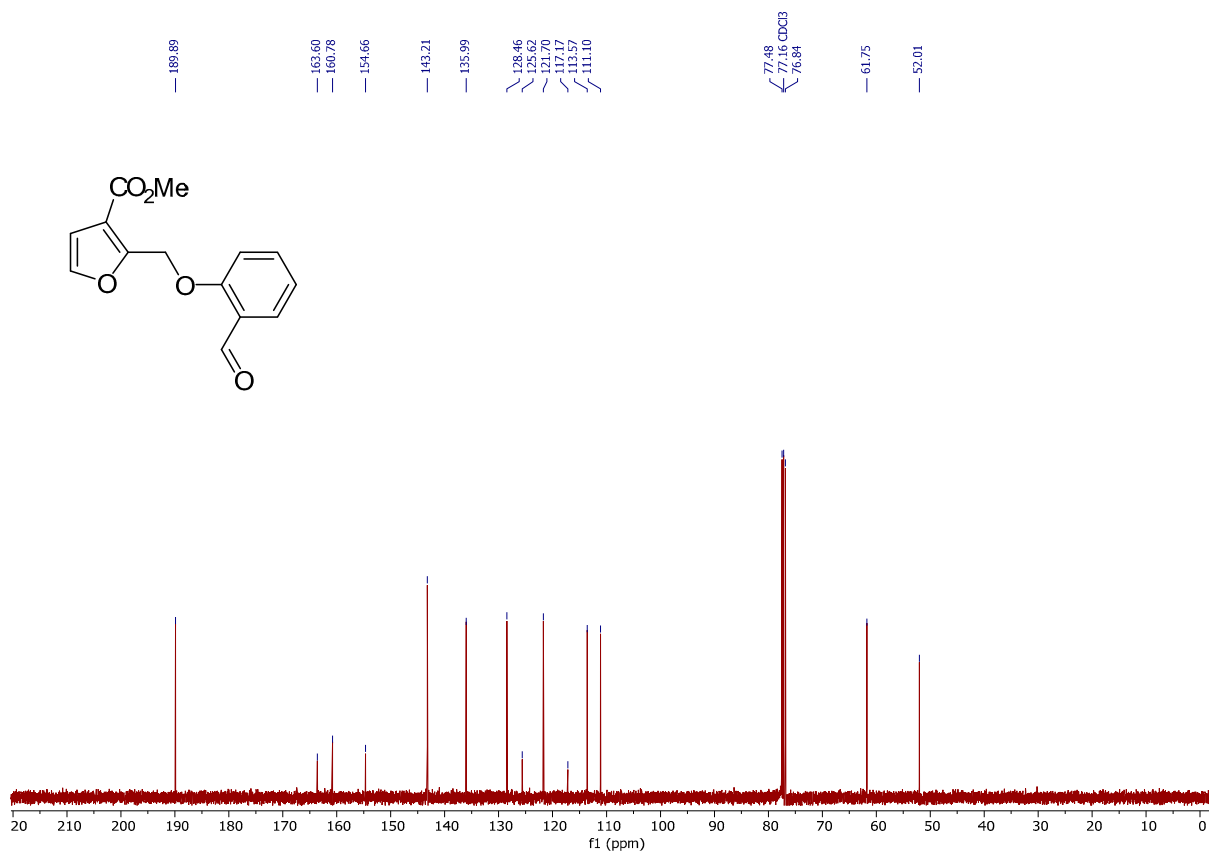
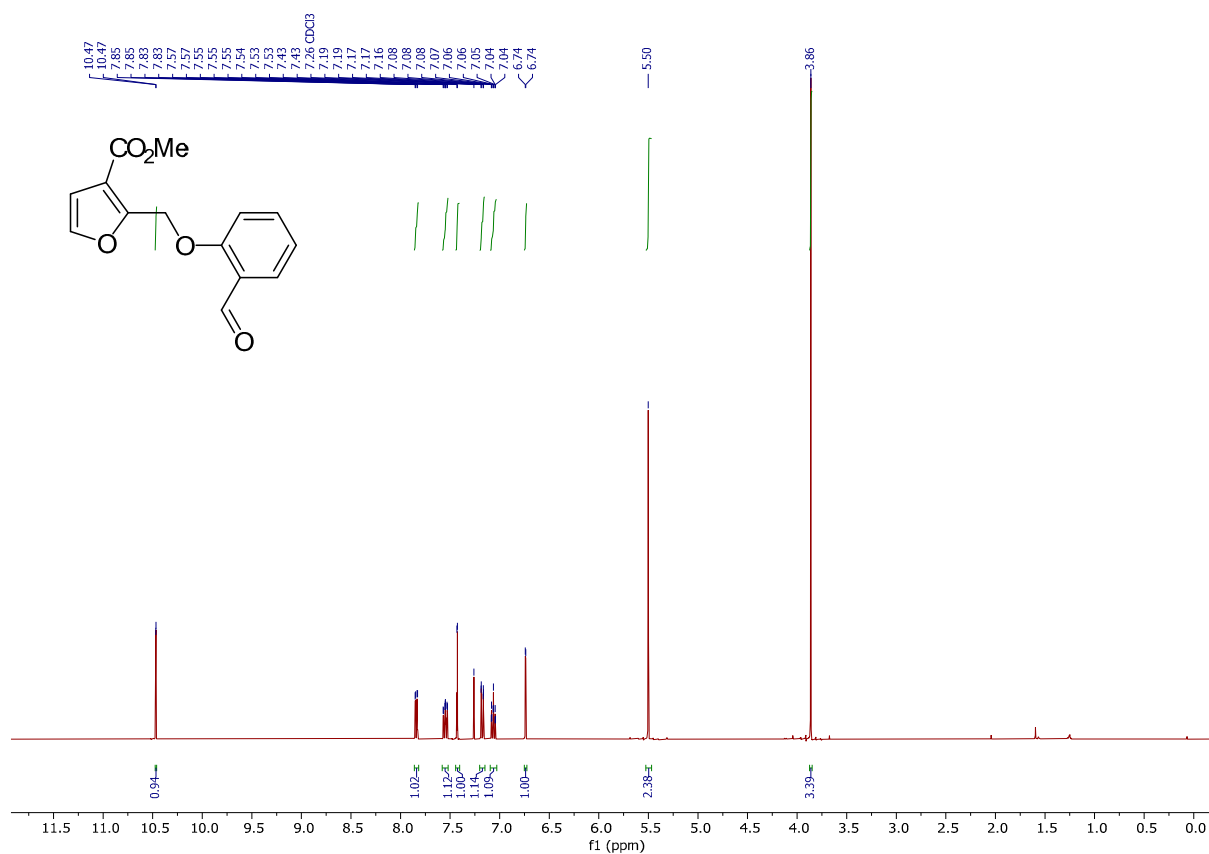
Computer programs: Bruker *SMART*, Bruker *SAINT*, *SHELXS97* (Sheldrick, 1990), *SHELXL97* (Sheldrick, 1997), *WinGX*, *Mercury*, Bruker *SHELXTL*.

## 5. NMR Spectra

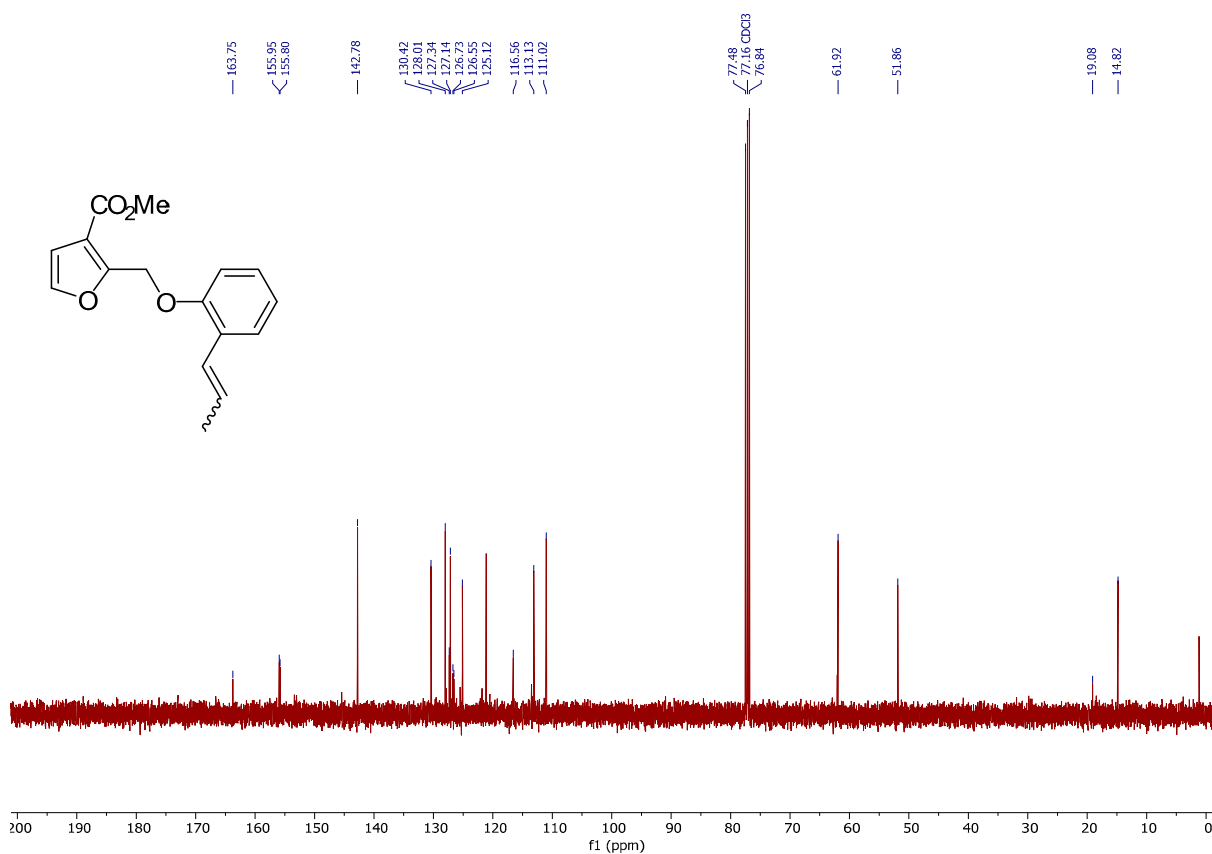
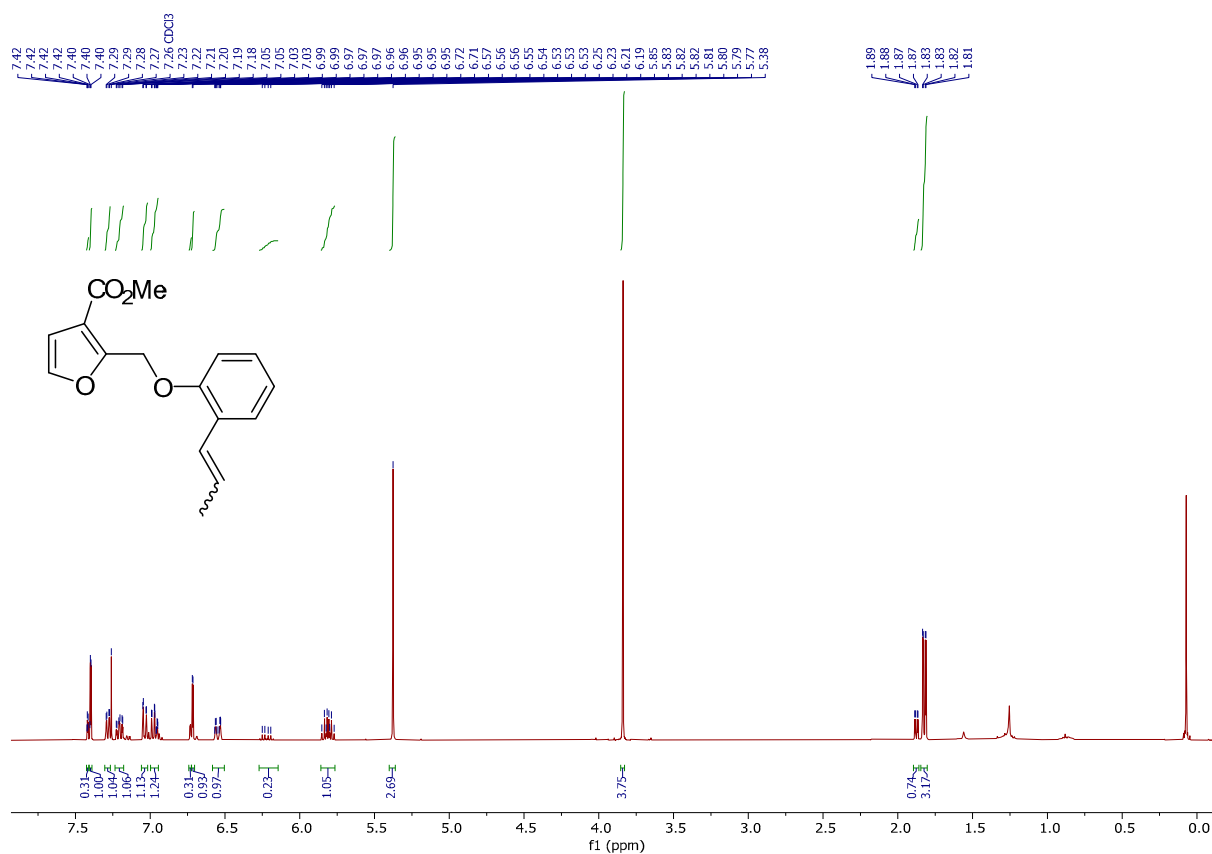
### Compound 2



# Compound A



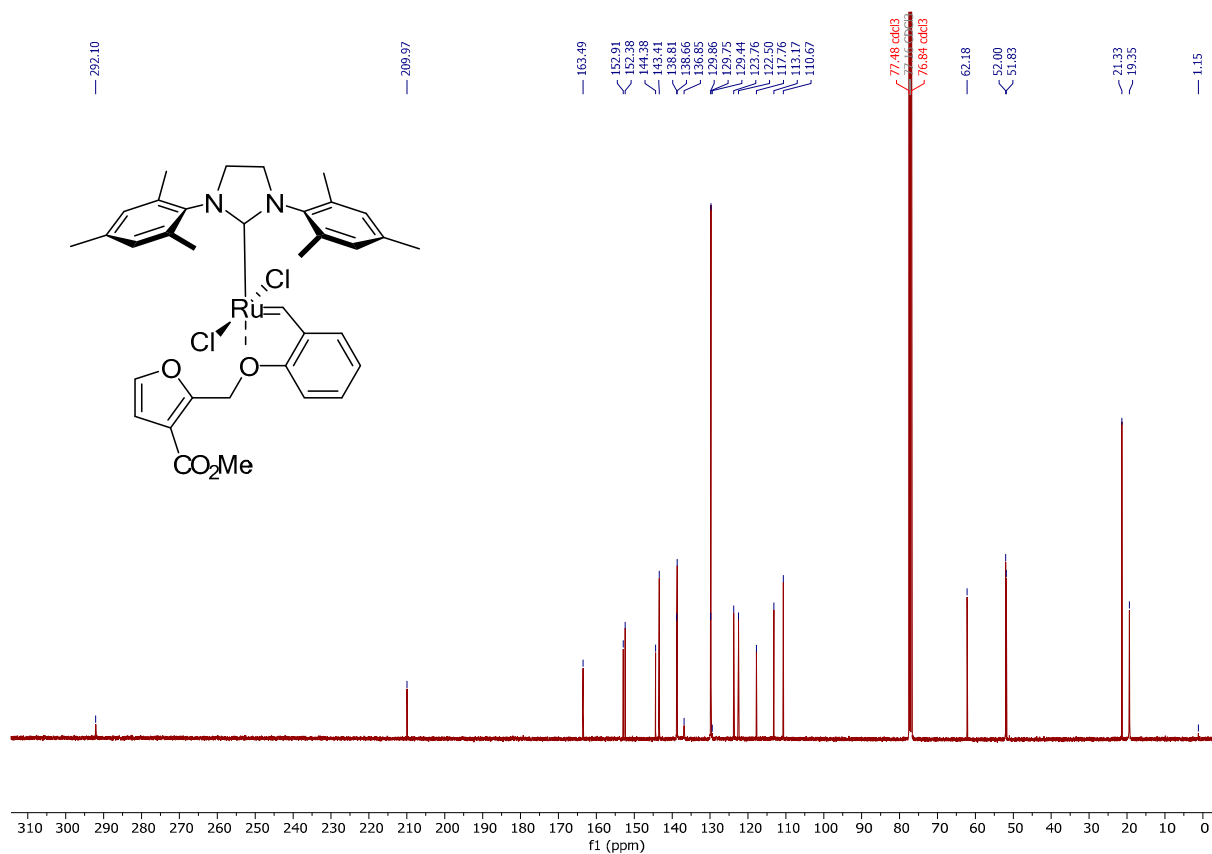
# Compound 4



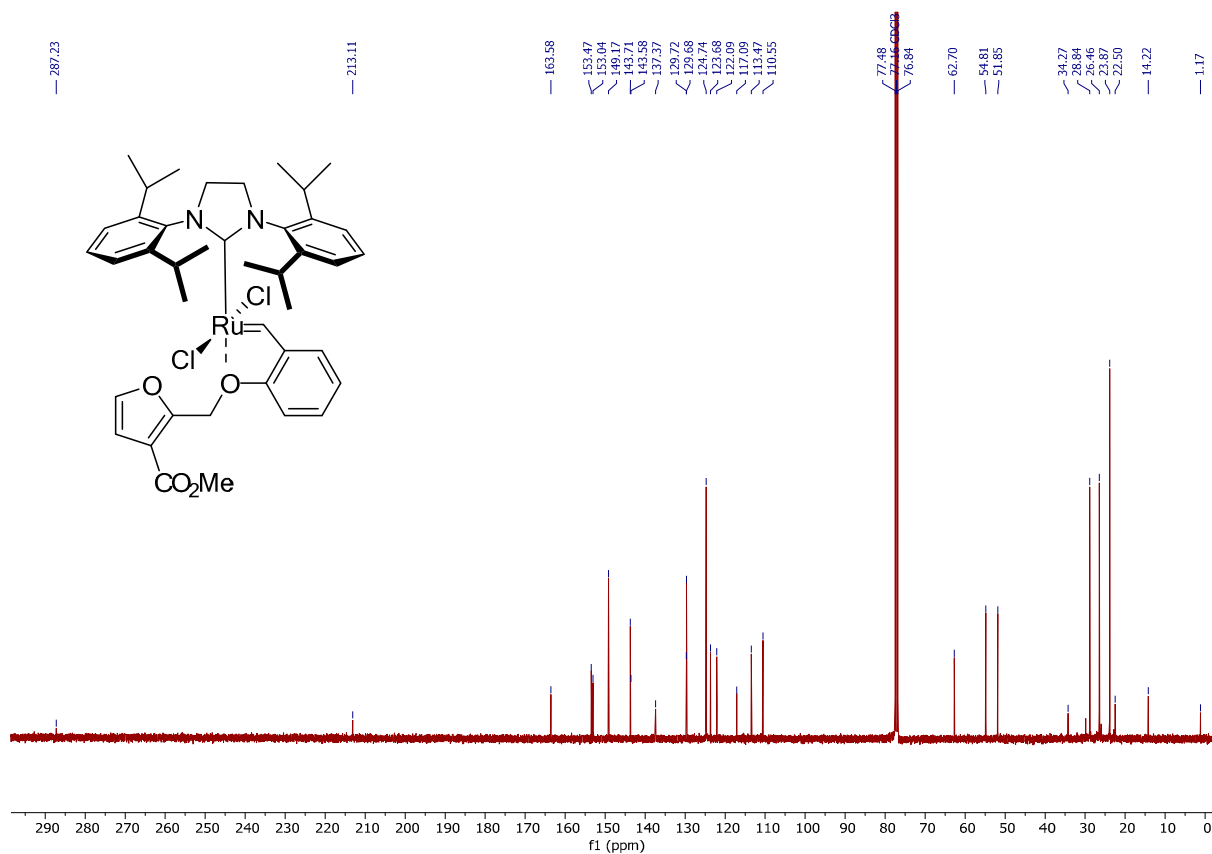
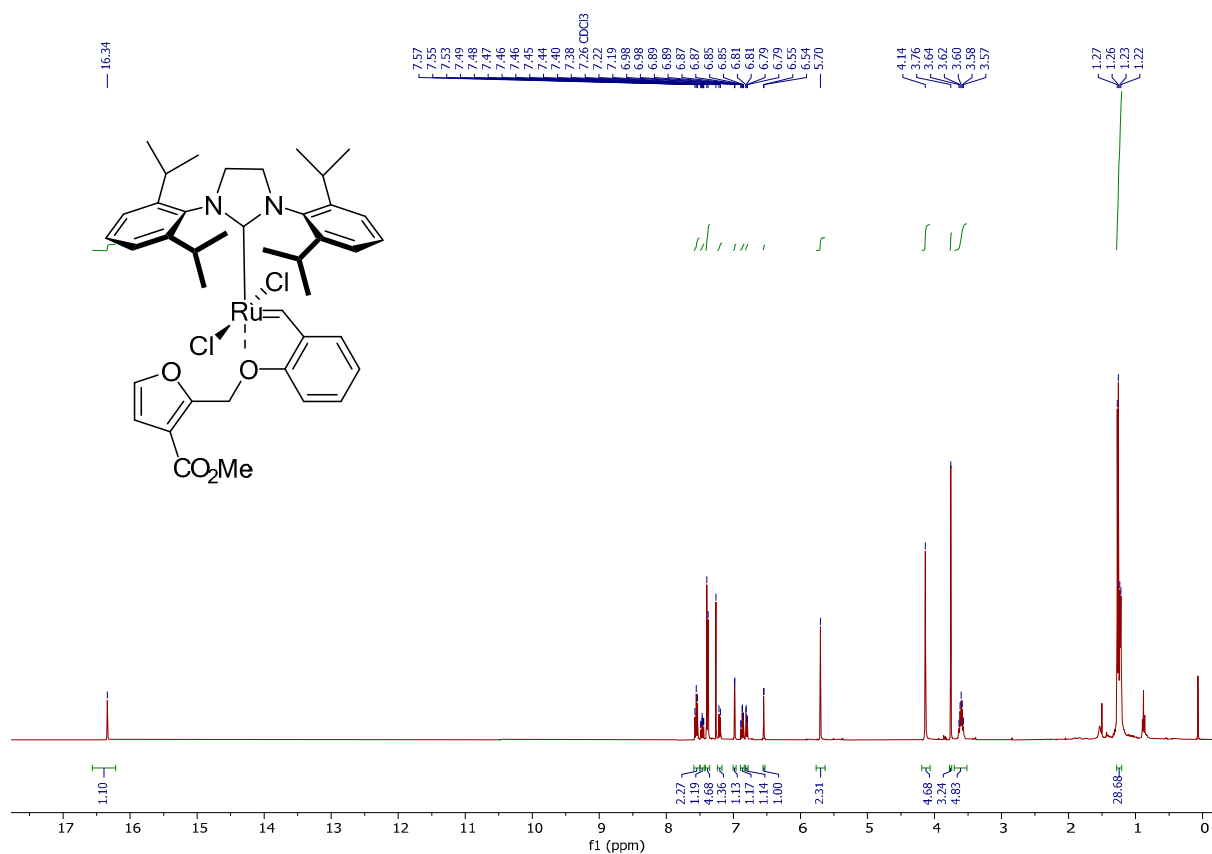
Chemical structure of compound 16 is shown above the  $^1\text{H}$  NMR spectrum. The structure is a ruthenium complex with a chiral ligand (1,2-bis(4-methylphenyl)ethane-1,2-diamine) and a chiral auxiliary (1,2-dichloro-1-phenyl-2-(4-methoxycarbonylphenoxy)ethane).

The  $^1\text{H}$  NMR spectrum (CDCl<sub>3</sub>) shows peaks from 0 to 8 ppm. The x-axis is labeled f1 (ppm). Integration values are provided below the peaks: 1.09, 1.14, 1.14, 1.14, 1.14, 1.00, 2.25, 4.50, 3.33, and 19.40.

Peak list (ppm): 7.54, 7.54, 7.52, 7.52, 7.51, 7.50, 7.50, 7.48, 7.48, 7.16, 7.09, 7.06, 6.94, 6.92, 6.91, 6.89, 6.88, 6.86, 6.86, 6.59, 6.53, 4.09, 3.78, 2.61, 2.60, 2.57, 2.56, 2.52, 2.46, 2.44.



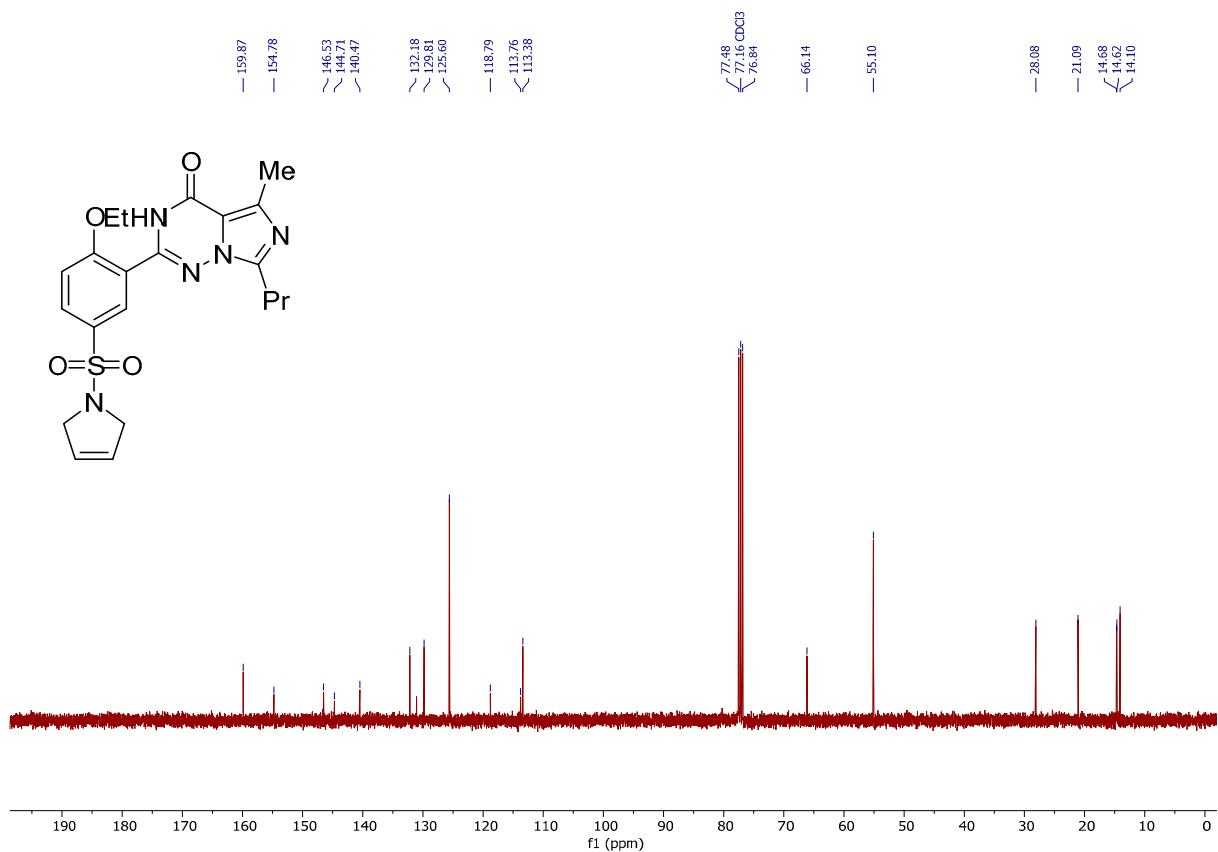
# Compound Ru17



**Chemical Structure of 10:** CC1=CN(C(=O)NC2=CC=C(S(=O)(=O)N3C=CC=C3)C=C2)N1CCC

**<sup>1</sup>H NMR Spectrum (CDCl<sub>3</sub>):**

Chemical Shift (ppm)	Integration
9.70	0.89
7.97, 7.96, 7.95, 7.94, 7.26, 7.17, 7.14	0.94, 1.00, 1.05
5.71, 5.70	1.94
4.35, 4.34, 4.33, 4.32, 4.31, 4.30, 4.18, 4.17, 4.16	2.18, 4.13
3.01	2.13
2.97, 2.97, 2.63, 1.91, 1.89, 1.88, 1.86, 1.84, 1.82, 1.60, 1.58, 1.57, 1.56, 1.04, 1.02, 1.00	3.10, 2.16, 3.24, 3.23
1.00	-



## 6. References

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