Synthesis and Structure of Fused *N*–Heterocyclic Carbenes and Their Rhodium Complexes

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Supplemental Information

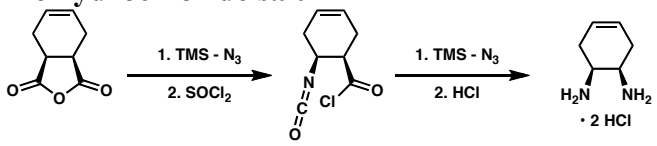
Contents

Materials and Methods

All reactions involving metal complexes were conducted under nitrogen or argon atmospheres using standard glovebox or standard Schlenk techniques. Solvents were purified by passage through alumina.[1] Resonances for NMR spectra are reported relative to Me_4Si (δ 0.0) for ^1H and ^{13}C. Spectra are reported as follows: chemical shift (δ ppm), integration, multiplicity and coupling constant (Hz). Ruthenium starting materials were provided by Materia, Inc. All other reagents were purchased from Aldrich and used without prior purification.

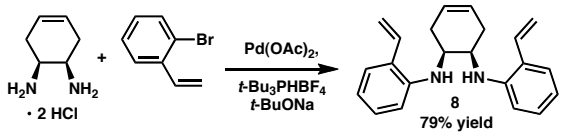
I. Experimental Procedures

Cyclohex-4-ene-1,2-diamine hydrochloride salt



For detailed procedures, see: Witiak, et al. *J. Med. Chem.* **1987**, *30*, 1327-1336.

Synthesis of 8



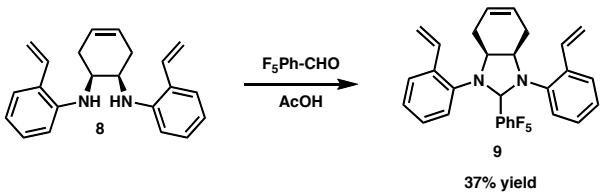
A flame-dried 50 mL 2-neck r.b. flask was charged with $Pd(OAc)_2$ (25 mg, 0.11 mmol), $tBu_3PH^+BF_4^-$ (96 mg, 0.33 mmol), NaOtBu (0.62 g, 6.0 mmol) and a stirbar. Toluene (10 mL) was added via canulla and the suspension was stirred for 15 minutes at r.t. 2-Bromostyrene (0.34 mL, 2.7 mmol) was added via syringe, followed by the diamine (0.2 g, 1.08 mmol) via spatula. The suspension was heated to 100 ˚C and stirred for 18 h. After cooling to r.t., the suspension was filtered through a pad of Celite, which was then washed with additional toluene (100 mL). Concentration of the combined toluene solutions afforded a dark brown oil. The desired product was obtained via silica gel chromatography (40:1 → 10:1 pentane:Et_2O) as a pale beige oil.

^1H NMR (300 MHz, $CDCl_3$) δ 7.23 (dd, J = 7.5, 1.7 Hz, 2H), 7.16 – 7.04 (m, 2H), 6.84 – 6.54 (m, 6H), 5.69 (t, J = 1.5 Hz, 2H), 5.52 (dd, J = 17.3, 1.6 Hz, 2H), 5.21 (dd, J = 11.0, 1.6 Hz, 2H), 4.13 (br s, 2H), 3.93 (t, J = 5.0 Hz, 2H), 2.53 (dd, J = 16.5, 3.9 Hz, 2H), 2.17 (dd, J = 16.6, 5.2 Hz, 2H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 144.7, 133.0, 130.4, 129.1, 127.9, 125.6, 125.2, 118.1, 116.6, 113.0, 50.9, 30.1.

HR-MS (FAB+) Calculated for $C_{22}H_{25}N_2$: 317.2018; found: 317.2032.

Synthesis of 9[3]

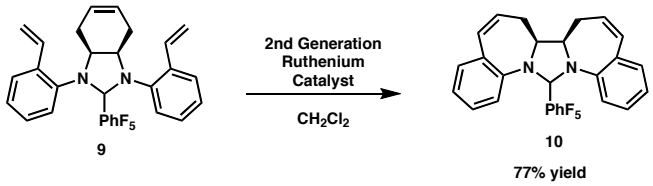
[1] Pangborn, A. B.; Giardello, M. A.; Grubbs, R. H.; Rosen, R. K.; Timmers, F. J. *Organometallics* **1996**, *15*, 1518–1520.



^{1}H NMR (300 MHz, $CDCl_3$) δ 18.79 (s, 1H), 7.97 (d, J = 4.2 Hz, 2H), 7.64 – 7.59 (m, 2H), 7.39 – 7.12 (m, 8H), 6.97 (d, J = 7.8 Hz, 2H), 4.38 – 3.93 (m, 4H), 2.49 (s, 2H), 2.28 (s, 2H).
^{19}F NMR (470 MHz, $CDCl_3$) δ -137.35 (1F), -144.88 (1F), -154.52 (1F), -162.96 (1F), -163.60 (1F).

HRMS (EI+) calculated 494.1781 found 494.1764

Synthesis of 10

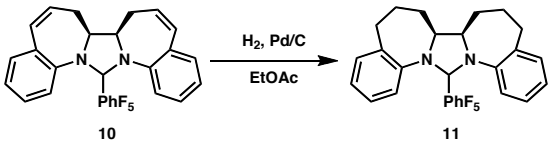


A procedure analogous to the formation of (**16**) was followed.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.13 (dd, J = 1.5, 7.8 Hz, 2H), 7.08 – 7.02 (m, 3H), 6.88 (dt, J = 1.5, 8.1 Hz, 2H), 6.67 (d, J = 3.3 Hz, 2H), 6.63 (s, 1H), 6.52 (dd, J = 2.1, 11.4 Hz, 2H), 6.29 – 5.99 (m, 2H), 4.26 – 4.00 (m, 2H), 2.94 - 2.36 (m, 2H), 1.55 (s, 1H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 147.8, 143.9, 132.3, 132.1, 128.8, 128.2, 127.5, 126.1, 120.5, 116.1, 72.5, 67.4, 31.1.

HRMS (EI+) calculated 494.1781 found 494.1764

Synthesis of 11

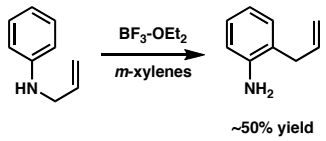


A procedure analogous to the formation of **(14)** was followed.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.05 – 6.95 (m, 4H), 6.85 (dt, J = 1.2, 7.2 Hz, 2H), 6.64 (d, J = 8.4 Hz, 2H), 6.47 (s, 1H), 4.03 – 4.00 (m, 2H), 3.08 – 2.99 (m, 2H), 2.69 – 2.63 (m, 2H), 1.95 – 1.87 (m, 2H), 1.69 – 1.59 (m, 6H).
^{19}F NMR (470 MHz, $CDCl_3$) δ -146.51 (bs, 1F), -154.58 (t, 1F), -163.12 (bs, 2F).

HRMS (FAB+) calculated for $C_{27}H_{22}N_2F_5$ 469.1703 found 469.1705

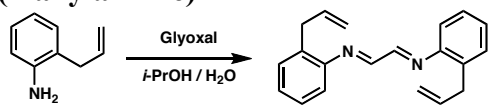
2-allylaniline



To a r.t. solution of *N*-allylaniline (2.7 mL, 20 mmol) in m-xylene (40 mL) in a 200 mL round bottom flask with a teflon stockcock adapter was added BF_3•Et_2O (3 mL, 24 mmol) via syringe. The flask was

sealed and the solution was heated at 150 ˚C for 18 h. After cooling to r.t., excess BF_3 was quenched by careful addition of 15% NaOH. The resulting suspension was diluted with Et_2O (~ 100 mL) and the organic layer was separated, and washed with brine. The aqueous layer was extracted with additional (~ 100 mL) of Et_2O. The combined organic layers were dried with $MgSO_4$, filtered and concentrated to afford a mixture of the starting material and product, which could be purified by fractional distillation.[2]

_N_,_N_'-(ethane-1,2-diylidene)bis(2-allylaniline)



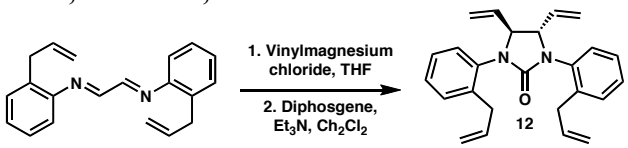
100% yield

A flask was charged with 2-allylaniline (0.79 g, 5.9 mmol), 2-propanol (6 mL), water (6 mL) and a stirbar. Glyoxal (0.31 mL, 2.7 mmol) was then added dropwise via syringe. After stirring at r.t. for 1 h, the bright yellow precipitate was collected via filtration using a C frit and washed sparingly with 2-propanol to afford the desired product.

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.30 (s, 2H), 7.34 – 7.17 (m, 4H), 7.08 – 6.94 (m, 2H), 6.07 – 5.87 (m, 2H), 5.09 – 4.92 (m, 4H), 3.57 (dt, J = 6.5, 1.4 Hz, 4H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 160.1, 149.2, 137.3, 134.9, 130.1, 127.8, 127.5, 117.6, 115.9, 35.9.

HRMS (FAB+): Calculated for $C_{20}H_{21}N_2$: 289.1705; found: 289.1719.

_N_^{3},_N_^{4}-bis(2-allylphenyl)hexa-1,5-diene-3,4-diamine



64% yield over 2 steps
single diastereomer

A flame-dried 50 mL 2-neck r.b. flask was charged with the diimine (1.0 g, 3.5 mmol) and a stirbar. Dry THF (15 mL) was added via canulla and the orange solution was cooled to -78 ˚C. Vinyl Grignard (13.9 mL, 1.0 M in Et_2O, Aldrich) was added dropwise via syringe, causing the orange solution to turn green. After stirring for 30 min at -78 ˚C, the solution was warmed to r.t. and stirred for an additional 2 h. After this time the solution was again clear and orange. Excess Grignard was quenched by careful addition of brine. The resulting suspension was diluted with Et_2O and washed with brine. The organic layer was dried with Na_2SO_4 and concentrated to afford an orange solid (sometimes a reddish oil).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.25 – 6.95 (m, 4H), 6.78 – 6.60 (m, 4H), 6.01 – 5.70 (m, 4H), 5.32 (dd, J = 16.9, 8.6 Hz, 4H), 5.09 (m, 4H), 4.12 (d, J = 8.8 Hz, 2H), 3.30 (d, J = 6.1 Hz, 4H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 145.1, 136.4, 136.0, 130.2, 127.7, 124.3, 118.5, 117.8, 116.7, 112.0, 59.1, 36.8.

HR-MS (FAB+) Calculated for $C_{24}H_{29}N_2$: 345.2331; found: 345.2339.

1,3-bis(2-allylphenyl)-4,5-divinylimidazolidin-2-one (12)

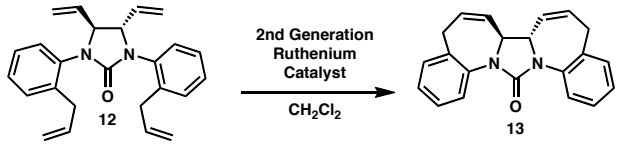
[2] Gagne, M. R.; Marks, T. J. *J. Am. Chem. Soc.*, **1989**, *111*, 4108-4109.

Crude N^3,N^4-bis(2-allylphenyl)hexa-1,5-diene-3,4-diamine was taken up in dry CH_2Cl_2 (30 mL). Et_3N (0.73 mL, 5.2 mmol) and DMAP (~ 10 mg) were added and the solution was cooled to 0 ˚C. Diphosgene (0.46 mL, 3.82 mmol) was added and the solution was stirred for 2 h. Excess diphosgene was then quenched by careful addition of H_2O. The suspension was then washed with brine, dried with $MgSO_4$, filtered and concentrated. The resulting pale yellow solid was washed with hexanes and dried. Silica gel chromatography afforded the desired product (4:1 hexanes:EtOAc).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.40 – 7.10 (m, 8H), 6.09 – 5.89 (m, 2H), 5.89 – 5.68 (m, 2H), 5.23 – 5.02 (m, 8H), 4.35 (dd, J = 6.0, 1.9 Hz, 2H), 3.66 – 3.25 (m, 4H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 157.0, 138.9, 137.3, 136.4, 134.5, 130.5, 127.6, 127.0, 121.3, 116.4, 66.6, 36.0.

HR-MS (FAB+) Calculated for $C_{25}H_{27}N_2O$: 371.2123; found: 371.2119.

Synthesis of 13

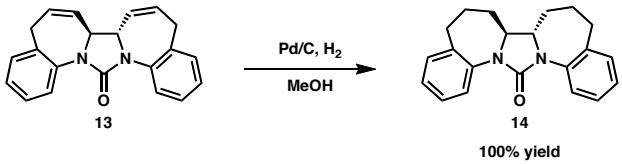


A 100 mL r.b. flask was charged with **12** (0.69 g, 1.86 mmol) and a stirbar. The flask was flushed with argon, and dry CH_2Cl_2 was added via canulla. An oven-dried 10 mL r.b. flask was cooled under argon and then charged with the catalyst (63 mg, 0.07 mmol), to which CH_2Cl_2 (1 mL) was added via syringe. The catalyst solution was then added dropwise to the substrate solution. After 4 h at r.t., the amber solution was filtered through a pad of SiO_2, which was washed with a 3:1 hexanes:ethyl acetate solution (200 mL) to afford the desired product.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.51 (d, J = 7.9 Hz, 2H), 7.34 – 6.98 (m, 6H), 6.09 – 5.79 (m, 2H), 5.58 (dd, J = 11.4, 2.4 Hz, 2H), 4.15 – 3.75 (m, 4H), 3.06 (dd, J = 16.1, 8.7 Hz, 2H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 167.6, 139.2, 138.8, 138.3, 129.0, 127.8, 126.6, 126.2, 125.7, 77.7, 77.4, 77.2, 76.8, 63.1, 32.4.

HR-MS (FAB+) Calculated for $C_{21}H_{18}N_2O$: 314.1419; found: 314.1416.

Synthesis of 14

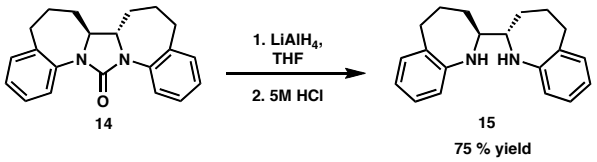


A 100 mL r.b. flask was charged with the tetracylic urea (0.58 g, 1.86 mmol), MeOH (20 mL) and a stirbar. 10% Pd/C (0.1g, 0.09 mmol) was added and the flask was flushed with argon. A balloon of H_2 was attached and the suspension was stirred until TLC indicated complete consumption of starting material. The suspension was then filtered through a plug of Celite and concentrated to afford the desired product as a beige solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.47 (dd, J = 8.0, 1.2 Hz, 2H), 7.31 – 7.05 (m, 4H), 3.17 (d, J = 10.8 Hz, 2H), 2.99 – 2.72 (m, 2H), 2.03 (d, J = 12.3 Hz, 4H), 1.80 (d, J = 12.5 Hz, 2H), 1.47 – 1.26 (m, 2H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 156.6, 139.8, 138.7, 130.4, 127.2, 126.0, 125.9, 64.1, 38.8, 35.6, 24.9.

HR-MS (FAB+) Calculated for $C_{21}H_{22}N_2O$: 318.1732; found: 318.1742.

Synthesis of 15

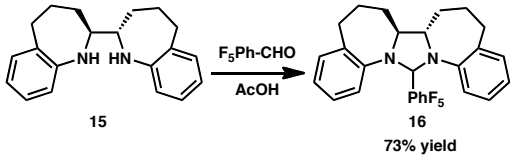


A 10 mL 2-neck r.b. flask was charged with **14** (0.35 g, 1.10 mmol) and a stirbar. A reflux condenser was attached and the system was flushed with argon. Dry THF (15 mL) was added by syringe and $LiAlH_4$ (0.22 g, 5.5 mmol) was added portionwise via spatula. The suspension was then heated at reflux for 2 h. After cooling to r.t., excess $LiAlH_4$ was quenched by careful addition of H_2O and 1 M NaOH. The slurry was filtered through a pad of Celite and extracted with Et_2O. The combined organic layers were concentrated, and the resulting residue was treated with 5 M HCl and heated to 60 ˚C for 2 h. The solution was cooled to r.t. and the pH was adjusted to 12 using 1N NaOH. The desired product was then extracted with CH_2Cl_2, dried with Na_2SO_4, and concentrated to afford a pale beige solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.17 – 7.03 (m, 4H), 6.93 – 6.80 (m, 4H), 2.97 – 2.61 (m, 6H), 2.04 – 1.77 (m, 6H), 1.63 – 1.44 (m, 2H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 148.6, 133.6, 130.8, 127.0, 121.3, 119.9, 61.9, 35.4, 34.6, 25.4.

HR-MS (FAB+) Calculated for $C_{20}H_{25}N_2$: 293.2018; found: 293.2018.

Synthesis of 16 [3]



A 10 mL r.b. flask was charged with a solution of the diamine (0.35 g, 1.2 mmol) in acetic acid (0.8 mL) and a stirbar. Pentafluorobenzaldehyde (0.4 g, 2 mmol) was added and the solution was stirred at r.t. overnight. The resulting precipitate was collected by filtration using an M frit and washed with cold methanol.

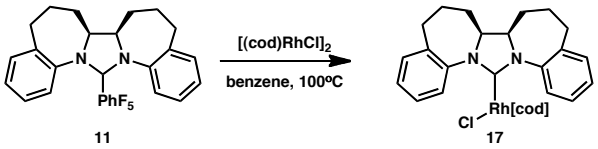
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.34 (d, J = 8.4 Hz, 1H), 7.21 (t, J = 7.8 Hz, 1H), 7.14 (t, J = 6 Hz, 1H), 7.13 (d, J = 6 Hz, 1H), 7.03 (s, 1H), 6.98 (d, J = 7.8 Hz, 1H), 6.93 (t, J = 7.2 Hz, 1H), 6.86 (d, J = 7.8Hz, 1H), 6.72 (t, J = 7.2 Hz, 1H), 4.36 (t, J = 12.6 Hz, 1H), 3.25 (m, 1H), 2.78 (m, 2H), 2.71 (t, J = 15.6 Hz, 1H), 2.48 (dd, J = 4.2 Hz, 15.6 Hz, 1H), 2.04 (d, J = 12.6 Hz, 2H), 1.93 (m, 3H), 1.79 (m, 2H), 1.34 (m, 1H).
^{13}C NMR (125 MHz, $CDCl_3$) δ 146.0 (d, 2C), 145.4, 143.7, 141.0 (d, 1C), 137.7 (d, 2C), 137.1, 131.3, 130.8, 129.1, 127.7, 127.1, 122.7, 119.2, 116.6, 116.4, 112.5, 69.4, 68.9, 67.5, 36.3, 34.4, 31.8, 28.6, 26.1, 24.9.
^{19}F NMR (470 MHz, $CDCl_3$) δ -143.75 (dd, 2F), -155.30 (t, 1F), -162.67 (dt, 2F).

HRMS (FAB+) calculated for $C_{27}H_{22}N_2F_5$ 469.1703; found: 469.1709

[3] Blum, A. P.; Ritter, T.; Grubbs, R. H. *Organometallics* **2007**, *26*, 2122.

FORMATION OF RHODIUM COMPLEXES:

Synthesis of 17



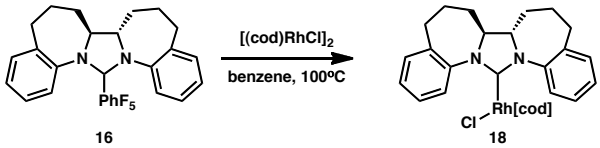
yield = 50%

See below for the analogous procedure to the formation of (**18**).

For proton and carbon shifts, please see attached spectra. Proton NMR and 2D experiments indicated two rotational isomers at room temperature. Variable temperature experiments were conducted up to 60°C and showed no exchange between the two species at that temperature.

HRMS (FAB+) calculated for $C_{29}H_{34}ClN_2Rh$ 548.1466 found 548.1460

Synthesis of 18

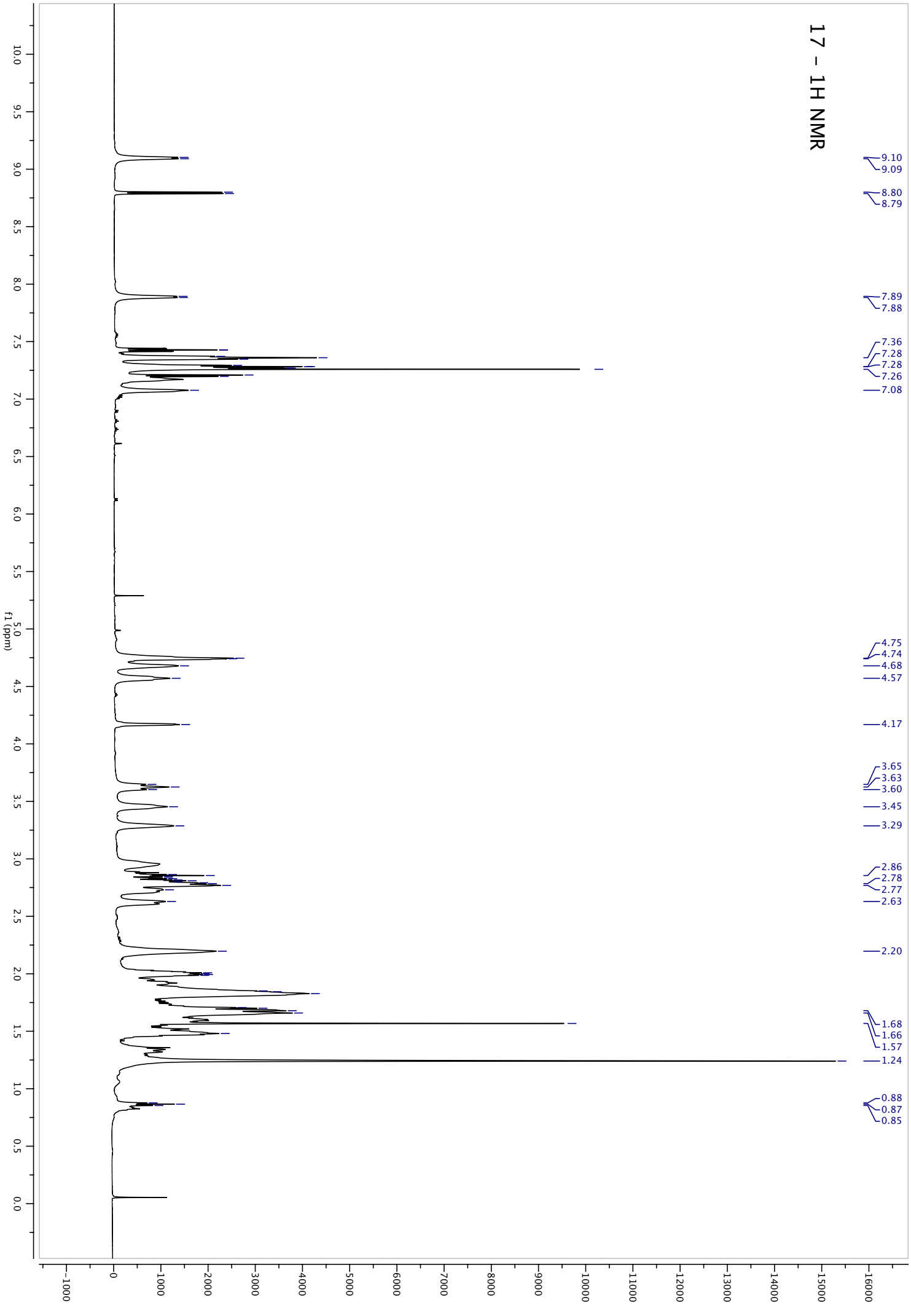


yield = 37%

In the glovebox, a 25 mL Schlenck flask equipped with a stirbar was charged with $[Rh(COD)Cl]_2$ (18 mg, 0.073 mmols) and the pentafluorophenyl adduct (34 mg, 0.072 mmols). To this was added 2 mL of benzene. The flask was sealed, removed from the glovebox and heated at 100°C for 48h. The reaction mixture was purified on silica gel (50% ether/hexanes) to afford a crystalline yellow solid. A single rotational isomer was identified in solution.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.56 (d, J = 8.4 Hz, 1H), 7.74 (d, J = 7.8 Hz, 1H), 7.38 (t, J = 6.6 Hz, 1 H), 7.31 – 7.28 (m, 1H), 7.25 – 7.21 (m, 3H), 7.17 (d, J = 8.4 Hz, 1H), 4.68 – 4.63 (m, 1H), 4.61 – 4.57 (m, 1H), 3.66 (t, J = 15 Hz, 1H), 3.34 – 3.31 (m, 1H), 3.27 (t, J = 12 Hz, 2H), 2.86 (dd, J = 5.4, 18.6 Hz1H), 2.74 – 2.68 (m, 2H), 2.62 (t, J = 18 Hz, 1H). 2.00 – 1.95 (m, 3H), 1.86 (d, J = 21.6 Hz, 1H), 1.77 – 1.71 (m, 4H), 1.61 (s, 2H), 1.50 – 1.34 (m, 6H).
^{13}C NMR (125 MHz, $CDCl_3$) δ 209.2 (d, J= 47.1 Hz, *C*–Rh), 142.5, 141.7, 140.9, 137.4, 130.8, 129.7, 129.2, 127.9, 127.8, 127.4, 127.2, 126.0, 99.6 (d, J= 6.3 Hz, 1C), 98.1 (d, J=5.1 Hz, 1C), 71.2, 71.1, 70.7 (d, J= 15.6 Hz, 1C), 65.8 (d, J= 14.4 Hz, 1C), 38.2, 37.8, 36.0, 35.5, 33.3, 31.2, 29.5, 27.2, 24.7, 24.6.

HRMS (FAB+) calculated for $C_{29}H_{34}ClN_2Rh$ 548.1544; found: 548.1530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9.09
8.80
8.79
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7.88
7.36
7.28
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4.57
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3.65
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3.60
3.45
3.29
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2.20
1.68
1.66
1.57
1.24
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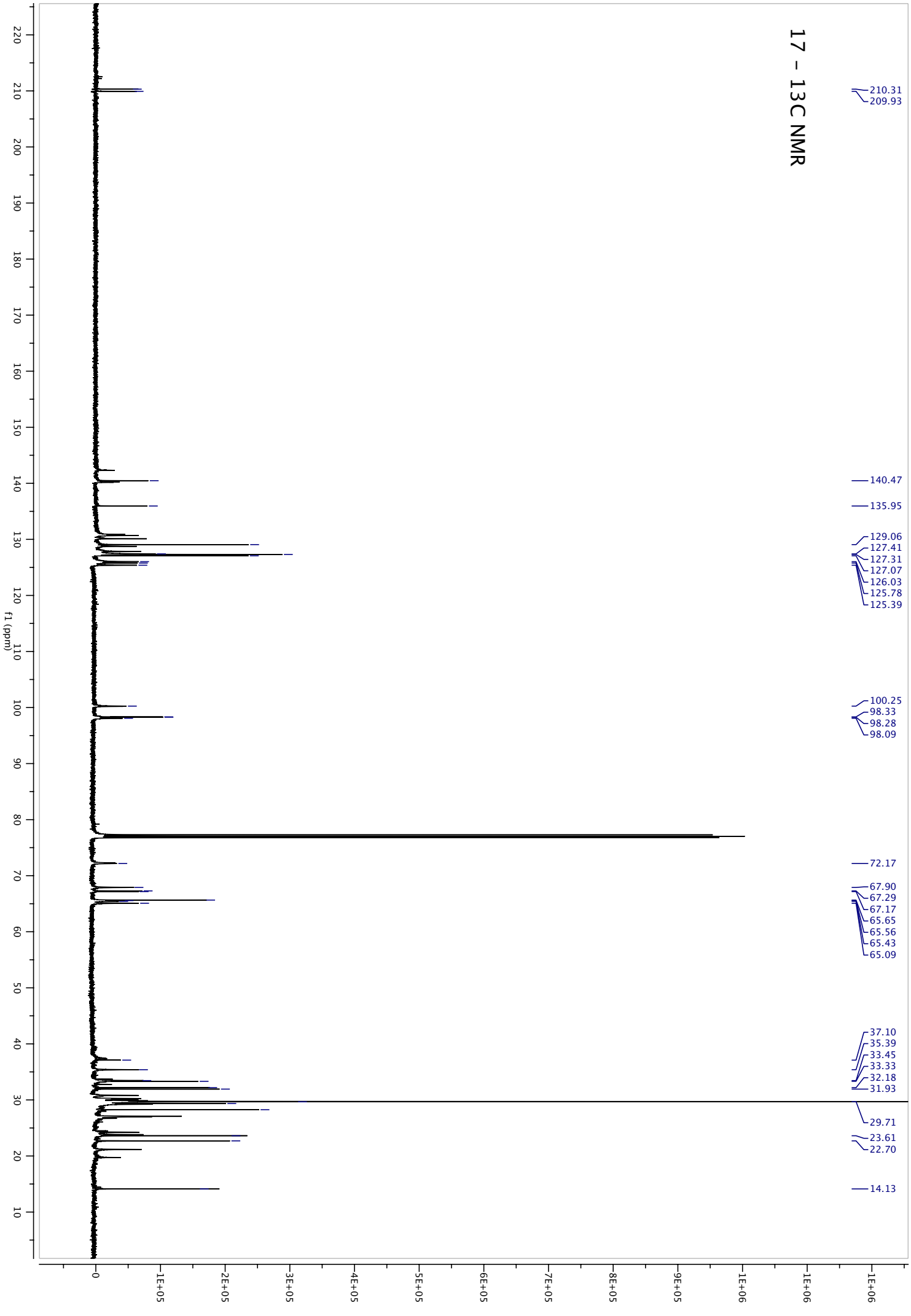
0.87
0.85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140.47
135.95
129.06
127.41
127.31
127.07
126.03
125.78
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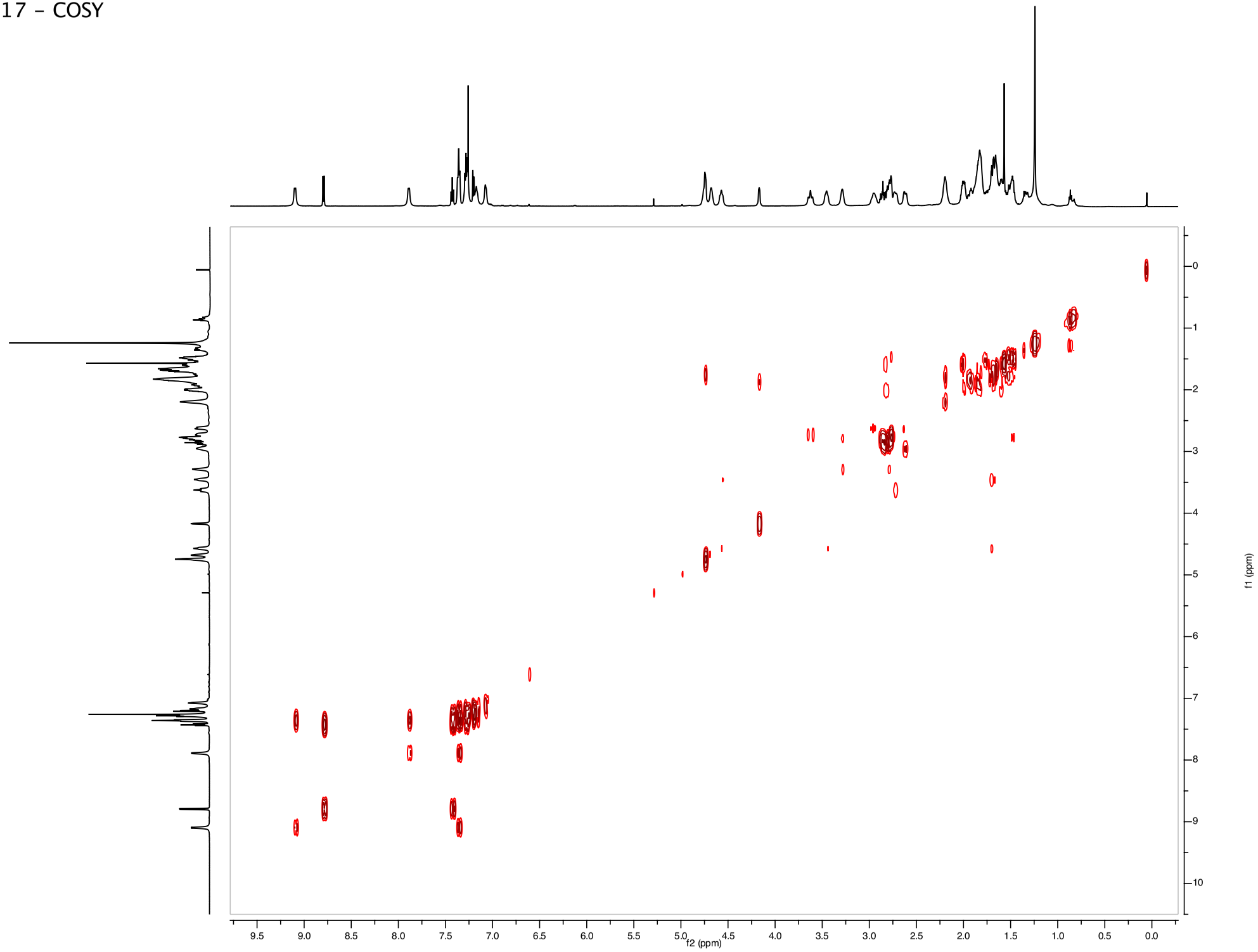
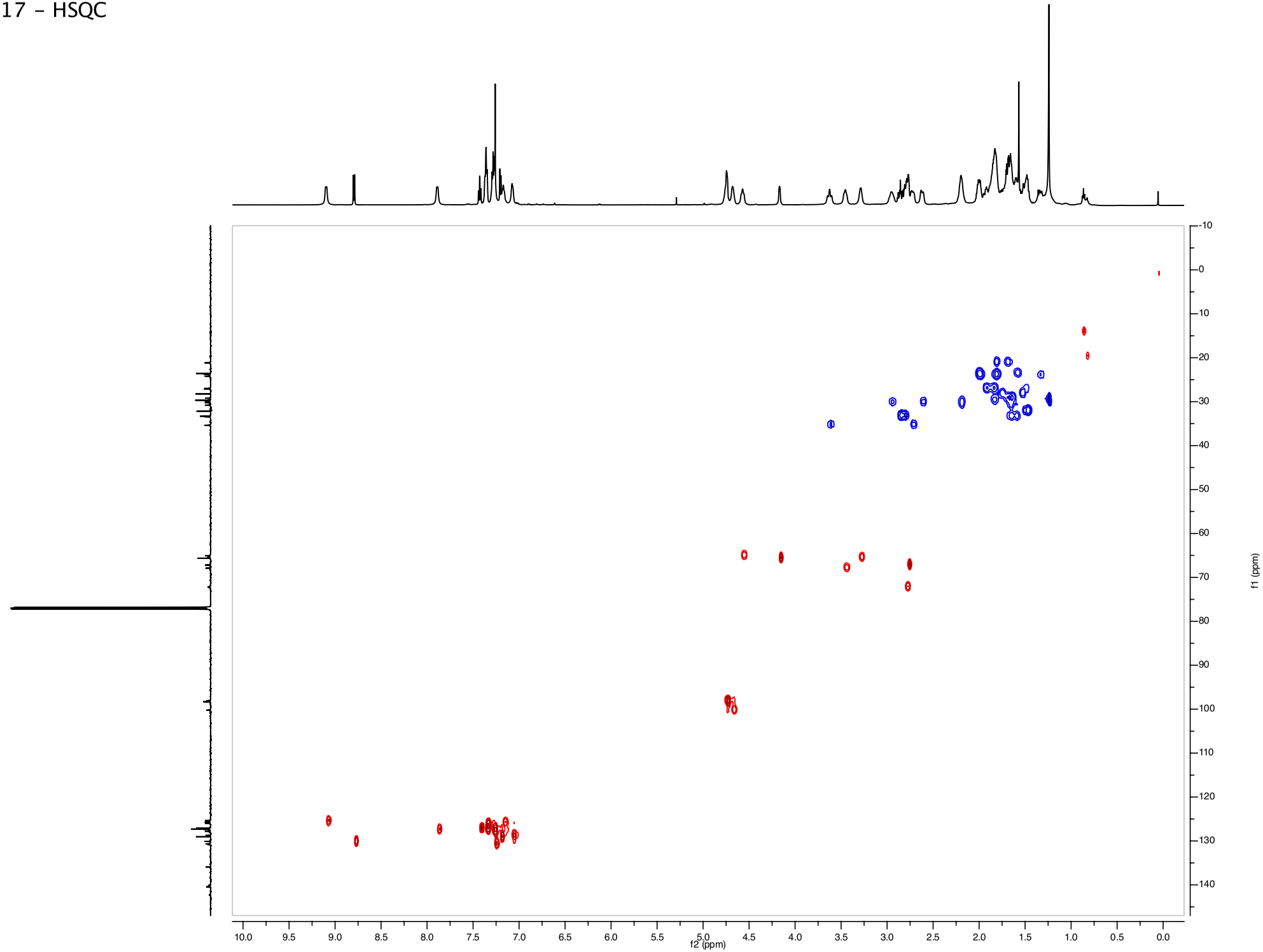
98.33
98.28
98.09
72.17
67.90
67.29
67.17
65.65
65.56
65.43
65.09
37.10
35.39
33.45
33.33
32.18
31.93
29.71
23.61
22.70
14.13
f1 (ppm)

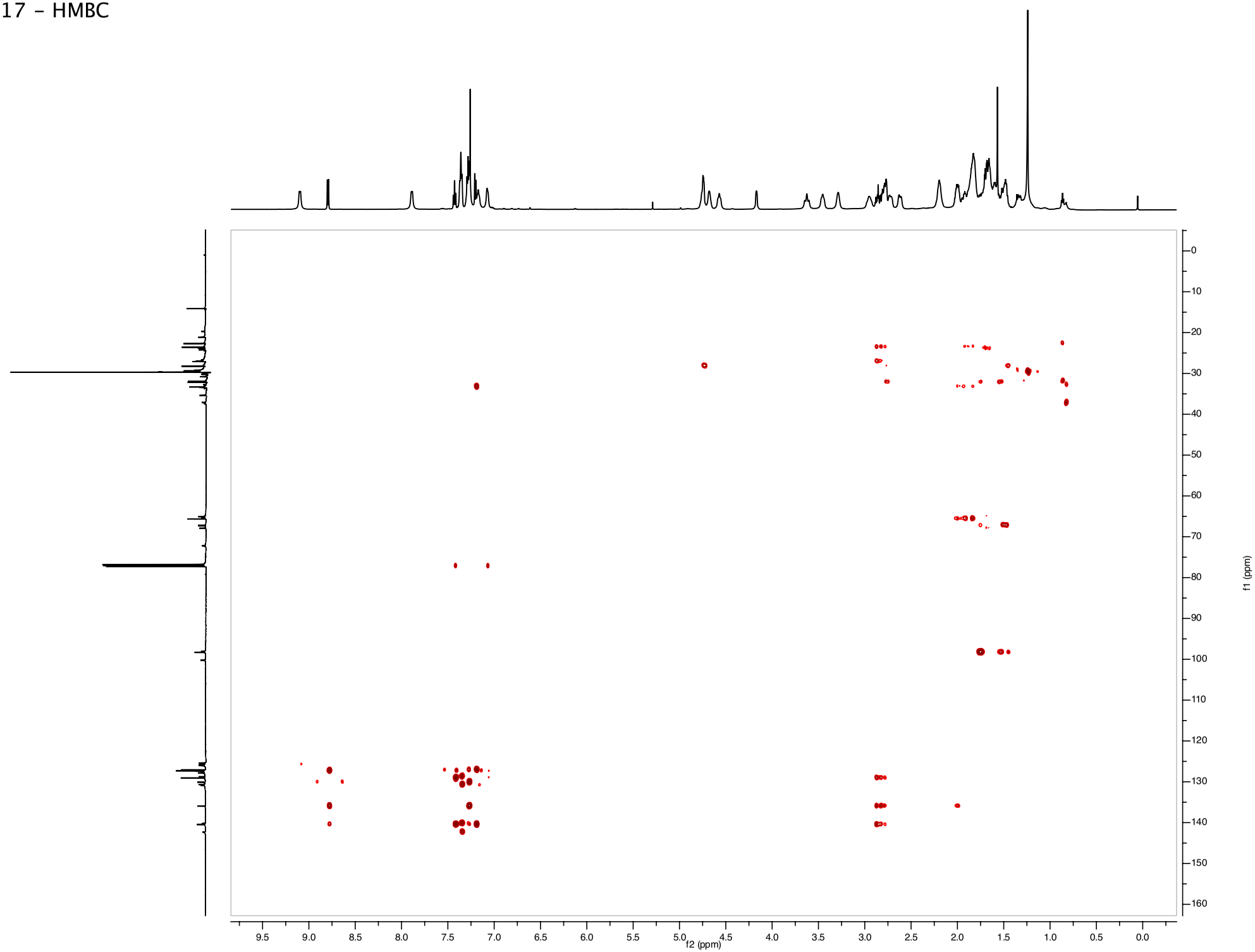
17 - COSY



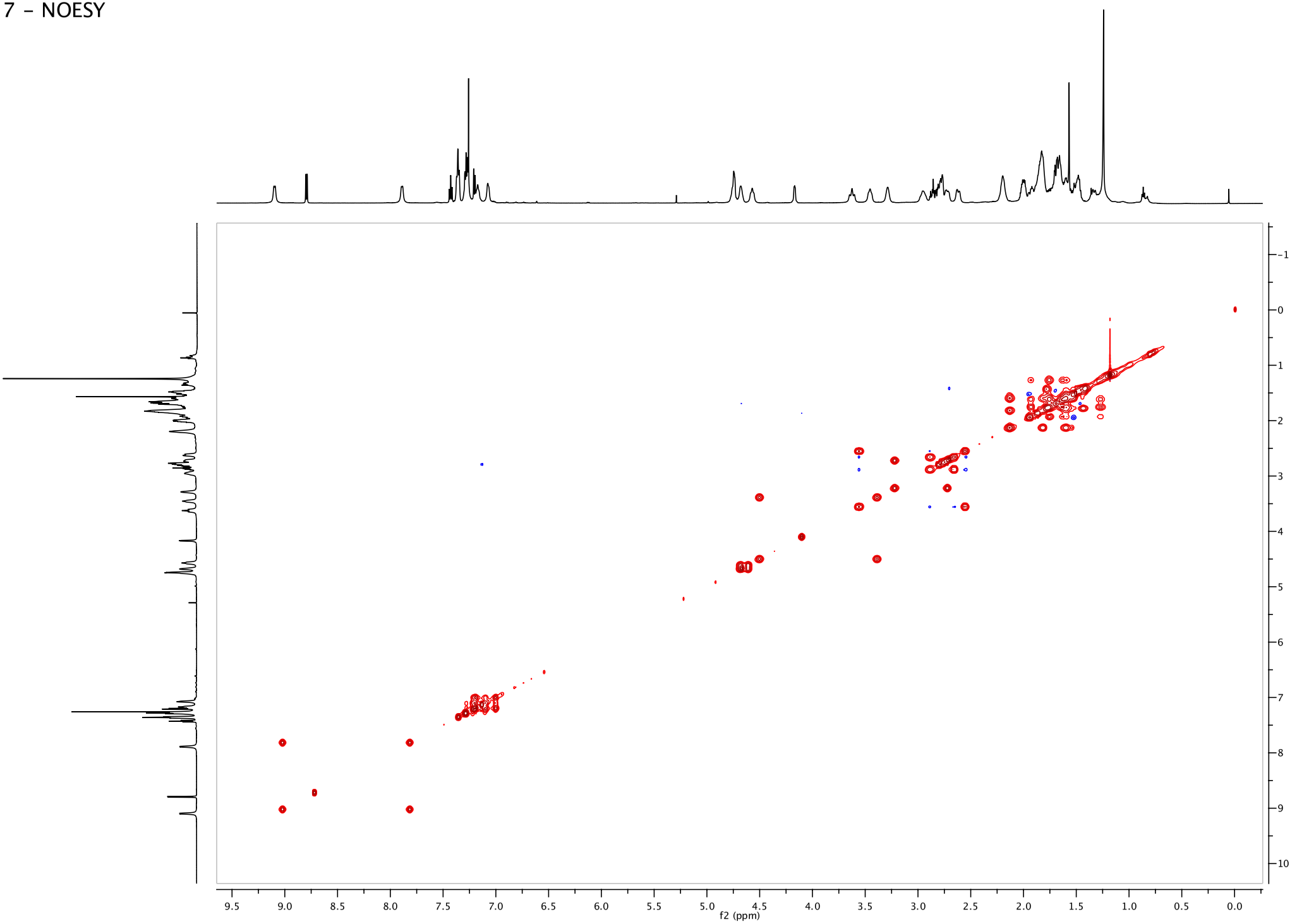
17 – HSQC

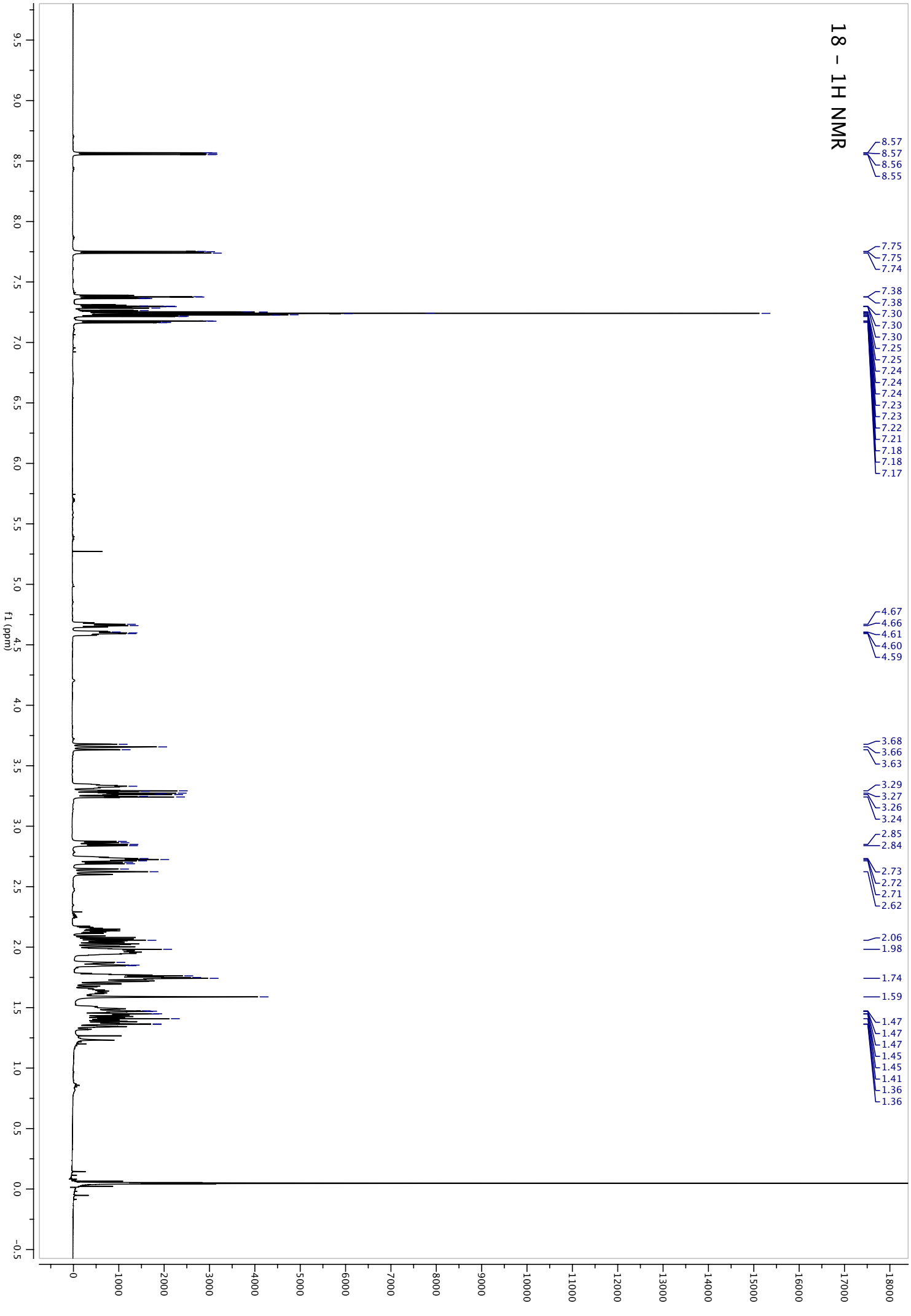

10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f2 (ppm)
-10
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
f1 (ppm)

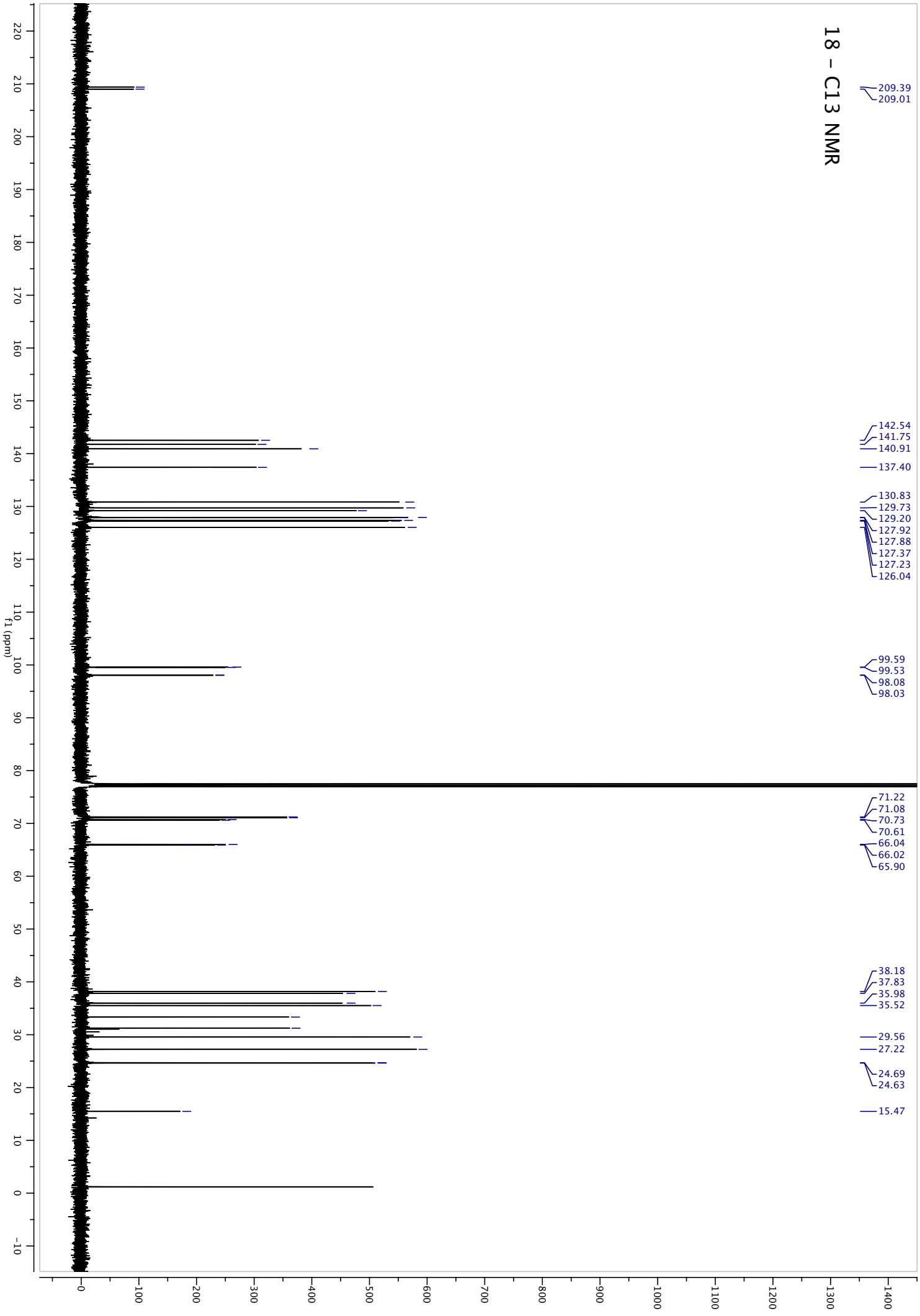
17 - HMBC



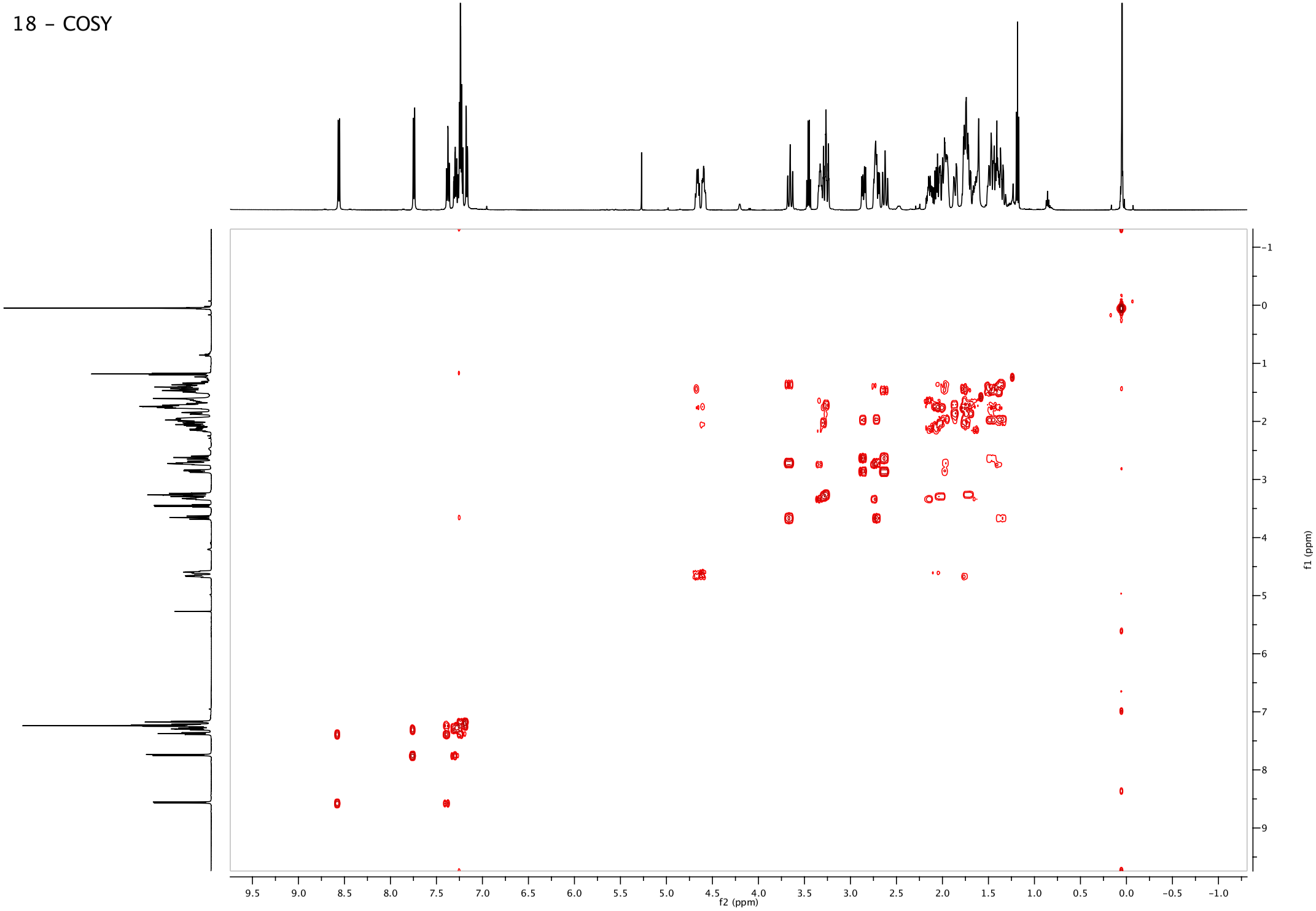
17 – NOESY



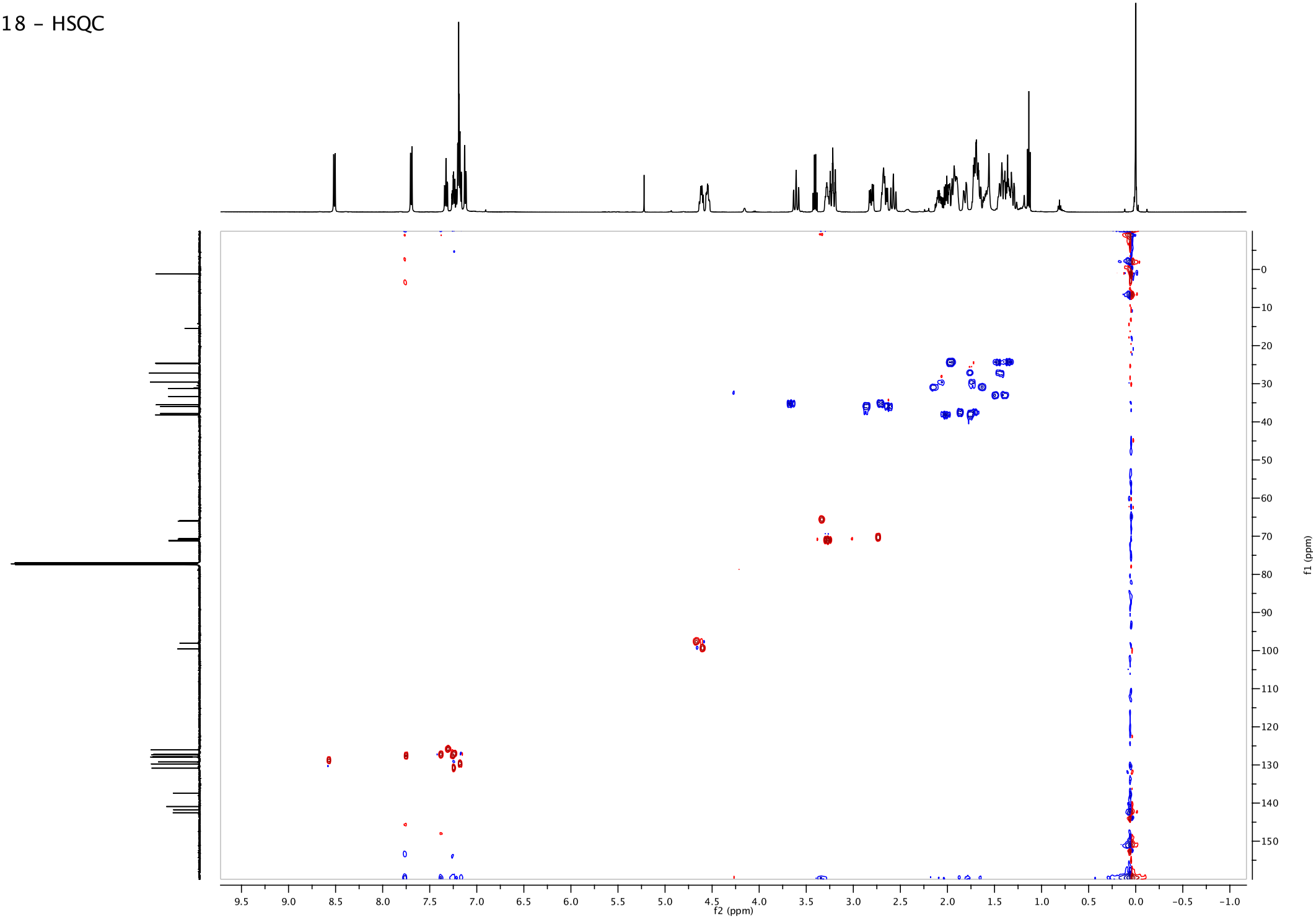

18 – 1H NMR
8.57
8.57
8.56
8.55
7.75
7.75
7.74
7.38
7.38
7.30
7.30
7.30
7.25
7.25
7.24
7.24
7.24
7.23
7.23
7.22
7.21
7.18
7.18
7.17
4.67
4.66
4.61
4.60
4.59
3.68
3.66
3.63
3.29
3.27
3.26
3.24
2.85
2.84
2.73
2.72
2.71
2.62
2.06
1.98
1.74
1.59
1.47
1.47
1.47
1.45
1.45
1.41
1.36
1.36
f1 (ppm)


18 – C13 NMR
209.39
209.01
142.54
141.75
140.91
137.40
130.83
129.73
129.20
127.92
127.88
127.37
127.23
126.04
99.59
99.53
98.08
98.03
71.22
71.08
70.73
70.61
66.04
66.02
65.90
38.18
37.83
35.98
35.52
29.56
27.22
24.69
24.63
15.47
f1 (ppm)

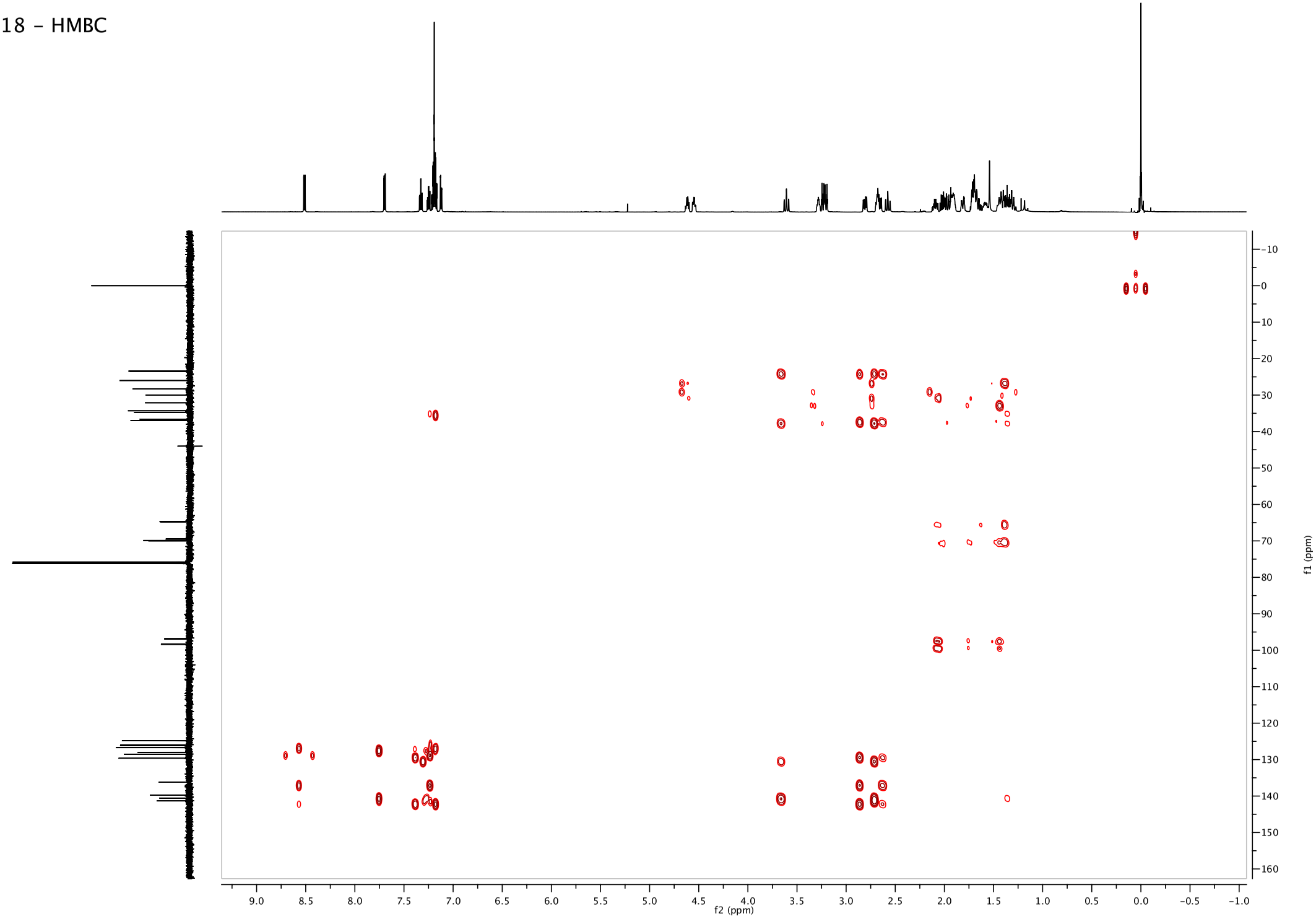
18 - COSY



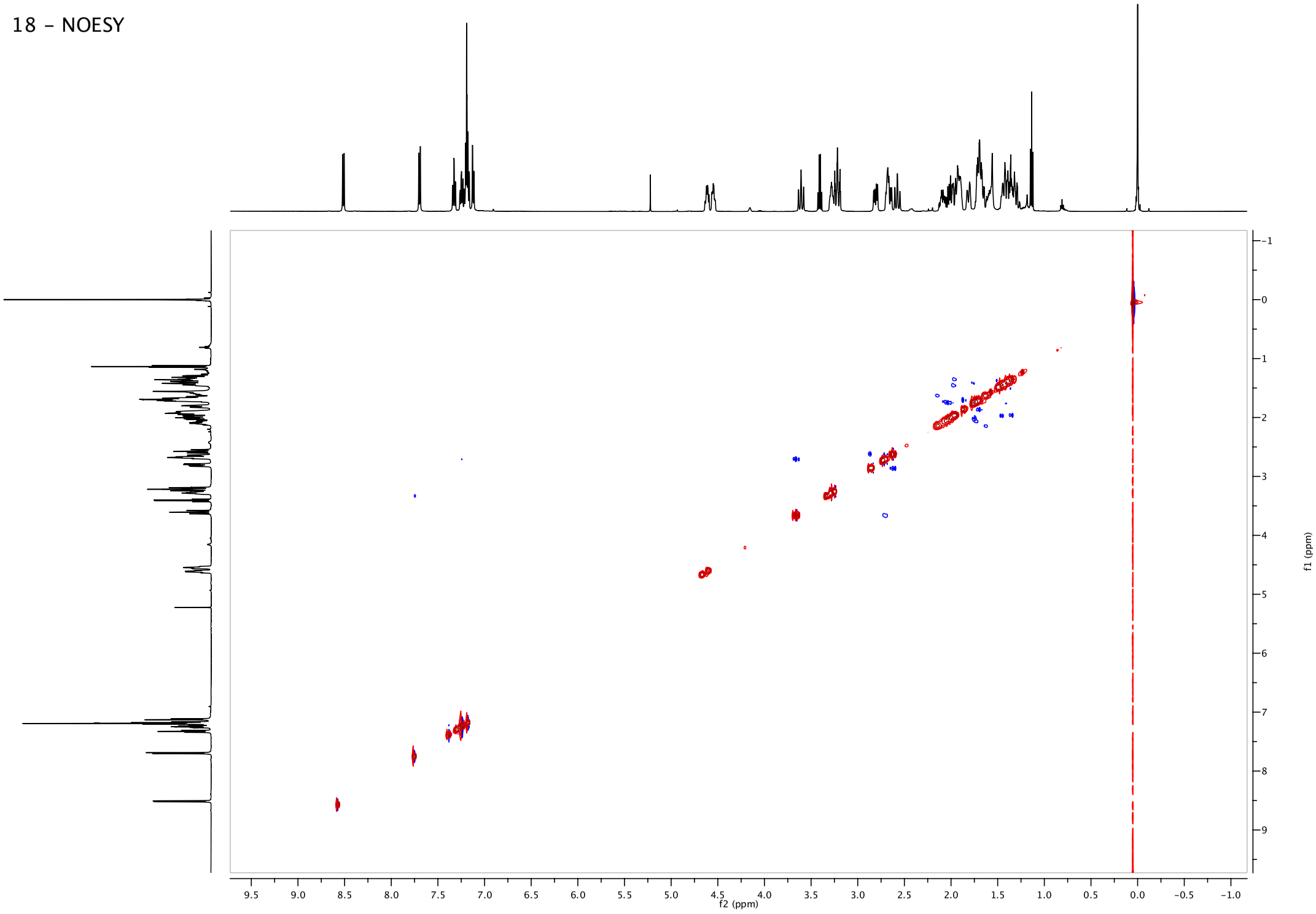
18 - HSQC



18 – HMBC



18 - NOESY



CALIFORNIA INSTITUTE OF TECHNOLOGY

BECKMAN INSTITUTE

X-RAY CRYSTALLOGRAPHY LABORATORY

Date 24 July 2008

Crystal Structure Analysis of:

ICS05

(shown below)

For Investigator: Ian Stewart ext. 6885

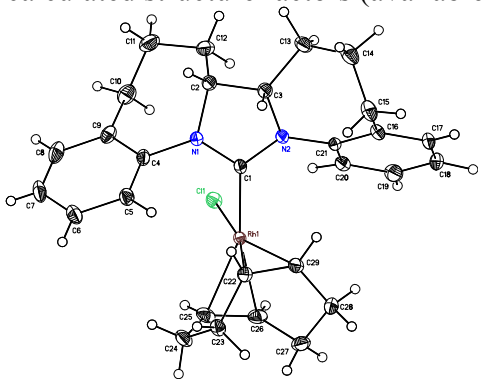
Advisor: R. H. Grubbs ext. 6003

Account Number: RHG.METAL-1-NIH.METAL2

By Michael W. Day 116 Beckman ext. 2734

e-mail: mikeday@caltech.edu

Contents



ICS05

Note: The crystallographic data have been deposited in the Cambridge Database (CCDC) and has been placed on hold pending further instructions from me. The deposition number is 696088. Ideally the CCDC would like the publication to contain a footnote of the type: "Crystallographic data have been deposited at the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK and copies can be obtained on request, free of charge, by quoting the publication citation and the deposition number 696088."

Table 1. Crystal data and structure refinement for ICS05 (CCDC 696088).

Empirical formula	$C_{29}H_{34}N_2ClRh$
Formula weight	548.94
Crystallization Solvent	Toluene/hexane
Crystal Habit	Fragment
Crystal size	0.13 x 0.11 x 0.11 mm^3
Crystal color	Light yellow

Data Collection

Type of diffractometer	Bruker KAPPA APEX II
Wavelength	0.71073 Å MoKα
Data Collection Temperature	100(2) K
θ range for 9945 reflections used in lattice determination	2.39 to 37.76°
Unit cell dimensions	a = 17.2031(7) Å b = 11.6106(5) Å β= 98.397(2)° c = 12.3438(5) Å
Volume	2439.10(18) $Å^3$
Z	4
Crystal system	Monoclinic
Space group	C*c*
Density (calculated)	1.495 Mg/m^3
F(000)	1136
Data collection program	Bruker APEX2 v2.1-0
θ range for data collection	2.12 to 37.83°
Completeness to θ = 37.83°	92.2 %
Index ranges	$-28 \le h \le 23$, $-19 \le k \le 19$, $-21 \le l \le 20$
Data collection scan type	scans; 11 settings
Data reduction program	Bruker SAINT-Plus v7.34A
Reflections collected	27779
Independent reflections	8931 [R_{int}= 0.0420]
Absorption coefficient	0.830 mm^{-1}
Absorption correction	None
Max. and min. transmission	0.9142 and 0.8998

Table 1 (cont.)

Structure solution and Refinement

Structure solution program	SHELXS-97 (Sheldrick, 2008)
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Difference Fourier map
Structure refinement program	SHELXL-97 (Sheldrick, 2008)
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	8931 / 2 / 434
Treatment of hydrogen atoms	Unrestrained
Goodness-of-fit on F^2	1.618
Final R indices [I>2σ(I), 7983 reflections]	R1 = 0.0323, *w*R2 = 0.0499
R indices (all data)	R1 = 0.0391, *w*R2 = 0.0504
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Max shift/error	0.002
Average shift/error	0.000
Absolute structure determination	Anomalous differences
Absolute structure parameter	-0.013(15)
Largest diff. peak and hole	2.588 and -1.995 e.Å^{-3}

Special Refinement Details

Crystals were mounted on a glass fiber using Paratone oil then placed on the diffractometer under a nitrogen stream at 100K.

Refinement of F^2 against ALL reflections. The weighted R-factor (*w*R) and goodness of fit (S) are based on F^2, conventional R-factors (R) are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

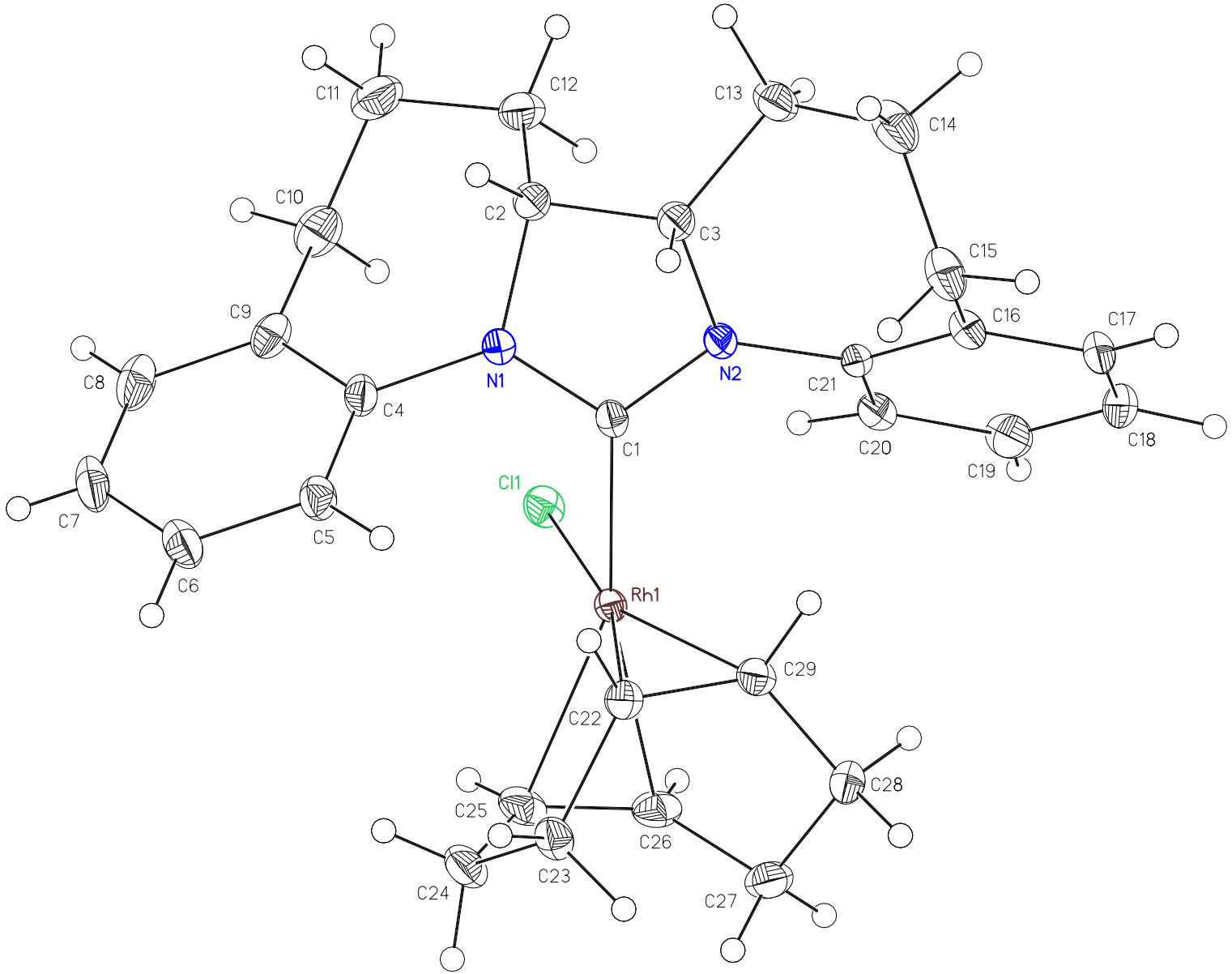

C11
C12
C13
C14
C10
C2
C3
C15
C9
C16
C17
C8
N1
N2
C21
C4
C1
C20
C18
C19
Cl1
C7
C5
C6
Rh1
C29
C22
C28
C25
C26
C23
C24
C27

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for ICS05 (CCDC 696088). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
Rh(1)	8006(1)	6711(1)	5754(1)	11(1)
Cl(1)	8046(1)	7387(1)	3919(1)	17(1)
N(1)	7702(1)	9179(2)	6184(2)	15(1)
N(2)	8904(1)	8745(2)	6852(2)	14(1)
C(1)	8231(1)	8311(2)	6292(2)	12(1)
C(2)	8080(1)	10319(2)	6478(2)	17(1)
C(3)	8766(2)	9911(2)	7312(2)	16(1)
C(4)	6889(1)	9075(2)	5722(2)	16(1)
C(5)	6395(1)	8446(2)	6278(2)	18(1)
C(6)	5592(1)	8368(2)	5868(2)	21(1)
C(7)	5305(2)	8936(2)	4916(2)	24(1)
C(8)	5804(1)	9552(2)	4356(2)	23(1)
C(9)	6606(1)	9636(2)	4737(2)	17(1)
C(10)	7144(2)	10246(3)	4075(2)	23(1)
C(11)	7617(2)	11263(2)	4619(2)	26(1)
C(12)	8312(1)	10910(2)	5480(2)	21(1)
C(13)	9505(1)	10653(2)	7531(2)	21(1)
C(14)	9998(2)	10306(2)	8610(2)	26(1)
C(15)	10033(2)	9008(2)	8795(2)	23(1)
C(16)	10216(1)	8341(2)	7828(2)	17(1)
C(17)	10944(1)	7801(2)	7836(2)	21(1)
C(18)	11112(1)	7172(2)	6963(2)	24(1)
C(19)	10556(1)	7068(2)	6025(2)	23(1)
C(20)	9833(1)	7612(2)	5979(2)	17(1)
C(21)	9661(1)	8227(2)	6879(2)	14(1)
C(22)	7707(1)	6163(2)	7283(2)	14(1)
C(23)	7096(1)	5208(2)	7184(2)	17(1)
C(24)	6714(1)	5020(2)	5990(2)	22(1)
C(25)	7262(1)	5223(2)	5172(2)	20(1)
C(26)	8027(1)	4853(2)	5248(2)	20(1)
C(27)	8430(2)	4133(2)	6182(2)	21(1)
C(28)	8908(1)	4865(2)	7084(2)	20(1)
C(29)	8519(1)	5992(2)	7255(2)	16(1)

Table 3. Selected bond lengths [Å] and angles [°] for ICS05 (CCDC 696088).

Rh(1)-C(1)	1.992(2)	C(1)-Rh(1)-C(29)	92.30(9)
Rh(1)-C(29)	2.105(2)	C(1)-Rh(1)-C(22)	92.47(8)
Rh(1)-C(22)	2.124(2)	C(29)-Rh(1)-C(22)	39.11(8)
Rh(1)-C(25)	2.207(2)	C(1)-Rh(1)-C(25)	156.04(9)
Rh(1)-C(26)	2.247(2)	C(29)-Rh(1)-C(25)	97.19(9)
Rh(1)-Cl(1)	2.4075(5)	C(22)-Rh(1)-C(25)	81.44(9)
		C(1)-Rh(1)-C(26)	167.88(8)
		C(29)-Rh(1)-C(26)	80.92(9)
		C(22)-Rh(1)-C(26)	88.58(9)
		C(25)-Rh(1)-C(26)	35.93(9)
		C(1)-Rh(1)-Cl(1)	88.78(6)
		C(29)-Rh(1)-Cl(1)	153.03(6)
		C(22)-Rh(1)-Cl(1)	167.75(6)
		C(25)-Rh(1)-Cl(1)	92.52(6)
		C(26)-Rh(1)-Cl(1)	92.75(6)

Table 4. Bond lengths [Å] and angles [°] for ICS05 (CCDC 696088).

Rh(1)-C(1)	1.992(2)	C(19)-C(20)	1.387(3)
Rh(1)-C(29)	2.105(2)	C(19)-H(19)	0.98(3)
Rh(1)-C(22)	2.124(2)	C(20)-C(21)	1.389(3)
Rh(1)-C(25)	2.207(2)	C(20)-H(20)	0.99(3)
Rh(1)-C(26)	2.247(2)	C(22)-C(29)	1.416(3)
Rh(1)-Cl(1)	2.4075(5)	C(22)-C(23)	1.521(3)
N(1)-C(1)	1.351(3)	C(22)-H(22)	0.88(2)
N(1)-C(4)	1.436(3)	C(23)-C(24)	1.538(3)
N(1)-C(2)	1.496(3)	C(23)-H(23A)	0.94(2)
N(2)-C(1)	1.356(3)	C(23)-H(23B)	0.98(2)
N(2)-C(21)	1.432(3)	C(24)-C(25)	1.497(3)
N(2)-C(3)	1.500(3)	C(24)-H(24A)	0.92(3)
C(2)-C(12)	1.514(3)	C(24)-H(24B)	0.89(3)
C(2)-C(3)	1.523(3)	C(25)-C(26)	1.375(3)
C(2)-H(2)	0.91(2)	C(25)-H(25)	0.86(3)
C(3)-C(13)	1.527(3)	C(26)-C(27)	1.508(3)
C(3)-H(3)	0.98(3)	C(26)-H(26)	1.07(2)
C(4)-C(5)	1.378(3)	C(27)-C(28)	1.540(3)
C(4)-C(9)	1.402(3)	C(27)-H(27A)	0.91(3)
C(5)-C(6)	1.402(3)	C(27)-H(27B)	0.94(2)
C(5)-H(5)	0.97(2)	C(28)-C(29)	1.499(3)
C(6)-C(7)	1.376(4)	C(28)-H(28A)	1.03(2)
C(6)-H(6)	0.97(3)	C(28)-H(28B)	0.86(3)
C(7)-C(8)	1.377(4)	C(29)-H(29)	0.95(3)
C(7)-H(7)	0.77(3)		
C(8)-C(9)	1.394(3)	C(1)-Rh(1)-C(29)	92.30(9)
C(8)-H(8)	0.85(3)	C(1)-Rh(1)-C(22)	92.47(8)
C(9)-C(10)	1.499(4)	C(29)-Rh(1)-C(22)	39.11(8)
C(10)-C(11)	1.533(4)	C(1)-Rh(1)-C(25)	156.04(9)
C(10)-H(10A)	0.96(3)	C(29)-Rh(1)-C(25)	97.19(9)
C(10)-H(10B)	0.92(3)	C(22)-Rh(1)-C(25)	81.44(9)
C(11)-C(12)	1.536(4)	C(1)-Rh(1)-C(26)	167.88(8)
C(11)-H(11A)	0.99(2)	C(29)-Rh(1)-C(26)	80.92(9)
C(11)-H(11B)	0.94(2)	C(22)-Rh(1)-C(26)	88.58(9)
C(12)-H(12A)	0.98(2)	C(25)-Rh(1)-C(26)	35.93(9)
C(12)-H(12B)	0.93(3)	C(1)-Rh(1)-Cl(1)	88.78(6)
C(13)-C(14)	1.525(4)	C(29)-Rh(1)-Cl(1)	153.03(6)
C(13)-H(13A)	1.01(2)	C(22)-Rh(1)-Cl(1)	167.75(6)
C(13)-H(13B)	0.93(2)	C(25)-Rh(1)-Cl(1)	92.52(6)
C(14)-C(15)	1.524(4)	C(26)-Rh(1)-Cl(1)	92.75(6)
C(14)-H(14A)	0.99(3)	C(1)-N(1)-C(4)	125.41(18)
C(14)-H(14B)	0.95(3)	C(1)-N(1)-C(2)	111.93(17)
C(15)-C(16)	1.495(3)	C(4)-N(1)-C(2)	122.27(17)
C(15)-H(15A)	0.94(3)	C(1)-N(2)-C(21)	124.21(18)
C(15)-H(15B)	0.90(3)	C(1)-N(2)-C(3)	110.99(17)
C(16)-C(17)	1.401(3)	C(21)-N(2)-C(3)	124.36(17)
C(16)-C(21)	1.404(3)	N(1)-C(1)-N(2)	106.57(19)
C(17)-C(18)	1.367(4)	N(1)-C(1)-Rh(1)	124.58(14)
C(17)-H(17)	0.88(3)	N(2)-C(1)-Rh(1)	128.82(15)
C(18)-C(19)	1.396(4)	N(1)-C(2)-C(12)	111.12(19)
C(18)-H(18)	0.99(3)	N(1)-C(2)-C(3)	98.93(17)

C(12)-C(2)-C(3)	114.87(19)	C(13)-C(14)-H(14A)	108.0(16)
N(1)-C(2)-H(2)	108.1(15)	C(15)-C(14)-H(14B)	106.1(16)
C(12)-C(2)-H(2)	114.5(16)	C(13)-C(14)-H(14B)	110.8(16)
C(3)-C(2)-H(2)	108.0(15)	H(14A)-C(14)-H(14B)	108(2)
N(2)-C(3)-C(2)	100.04(18)	C(16)-C(15)-C(14)	113.6(2)
N(2)-C(3)-C(13)	113.7(2)	C(16)-C(15)-H(15A)	108.9(17)
C(2)-C(3)-C(13)	119.1(2)	C(14)-C(15)-H(15A)	110.4(18)
N(2)-C(3)-H(3)	102.8(16)	C(16)-C(15)-H(15B)	109.4(18)
C(2)-C(3)-H(3)	107.5(15)	C(14)-C(15)-H(15B)	111.9(19)
C(13)-C(3)-H(3)	111.9(15)	H(15A)-C(15)-H(15B)	102(2)
C(5)-C(4)-C(9)	121.1(2)	C(17)-C(16)-C(21)	117.5(2)
C(5)-C(4)-N(1)	118.83(19)	C(17)-C(16)-C(15)	121.3(2)
C(9)-C(4)-N(1)	120.1(2)	C(21)-C(16)-C(15)	121.2(2)
C(4)-C(5)-C(6)	120.1(2)	C(18)-C(17)-C(16)	121.7(2)
C(4)-C(5)-H(5)	120.0(14)	C(18)-C(17)-H(17)	123.4(16)
C(6)-C(5)-H(5)	119.8(14)	C(16)-C(17)-H(17)	114.8(16)
C(7)-C(6)-C(5)	119.2(2)	C(17)-C(18)-C(19)	120.1(2)
C(7)-C(6)-H(6)	120.4(16)	C(17)-C(18)-H(18)	119.7(16)
C(5)-C(6)-H(6)	120.3(16)	C(19)-C(18)-H(18)	120.1(16)
C(6)-C(7)-C(8)	120.4(2)	C(20)-C(19)-C(18)	119.8(2)
C(6)-C(7)-H(7)	117(2)	C(20)-C(19)-H(19)	117.0(15)
C(8)-C(7)-H(7)	123(2)	C(18)-C(19)-H(19)	123.1(15)
C(7)-C(8)-C(9)	121.7(2)	C(19)-C(20)-C(21)	119.7(2)
C(7)-C(8)-H(8)	121.1(19)	C(19)-C(20)-H(20)	119.8(15)
C(9)-C(8)-H(8)	117.2(19)	C(21)-C(20)-H(20)	120.3(15)
C(8)-C(9)-C(4)	117.5(2)	C(20)-C(21)-C(16)	121.18(19)
C(8)-C(9)-C(10)	120.5(2)	C(20)-C(21)-N(2)	119.81(18)
C(4)-C(9)-C(10)	121.9(2)	C(16)-C(21)-N(2)	119.00(18)
C(9)-C(10)-C(11)	117.1(2)	C(29)-C(22)-C(23)	124.6(2)
C(9)-C(10)-H(10A)	108.0(15)	C(29)-C(22)-Rh(1)	69.71(12)
C(11)-C(10)-H(10A)	108.5(16)	C(23)-C(22)-Rh(1)	113.63(14)
C(9)-C(10)-H(10B)	110.0(19)	C(29)-C(22)-H(22)	119.4(15)
C(11)-C(10)-H(10B)	111.3(19)	C(23)-C(22)-H(22)	112.4(15)
H(10A)-C(10)-H(10B)	101(2)	Rh(1)-C(22)-H(22)	106.1(15)
C(10)-C(11)-C(12)	114.1(2)	C(22)-C(23)-C(24)	111.97(18)
C(10)-C(11)-H(11A)	110.7(14)	C(22)-C(23)-H(23A)	105.2(14)
C(12)-C(11)-H(11A)	110.3(14)	C(24)-C(23)-H(23A)	110.8(13)
C(10)-C(11)-H(11B)	106.9(14)	C(22)-C(23)-H(23B)	109.0(13)
C(12)-C(11)-H(11B)	106.6(14)	C(24)-C(23)-H(23B)	113.0(13)
H(11A)-C(11)-H(11B)	107.8(19)	H(23A)-C(23)-H(23B)	106.5(18)
C(2)-C(12)-C(11)	114.4(2)	C(25)-C(24)-C(23)	113.67(19)
C(2)-C(12)-H(12A)	105.9(13)	C(25)-C(24)-H(24A)	109.8(16)
C(11)-C(12)-H(12A)	111.3(13)	C(23)-C(24)-H(24A)	110.7(16)
C(2)-C(12)-H(12B)	106.8(17)	C(25)-C(24)-H(24B)	108.6(16)
C(11)-C(12)-H(12B)	111.2(17)	C(23)-C(24)-H(24B)	107.7(16)
H(12A)-C(12)-H(12B)	107(2)	H(24A)-C(24)-H(24B)	106(2)
C(14)-C(13)-C(3)	110.4(2)	C(26)-C(25)-C(24)	126.5(2)
C(14)-C(13)-H(13A)	109.9(13)	C(26)-C(25)-Rh(1)	73.63(13)
C(3)-C(13)-H(13A)	108.1(13)	C(24)-C(25)-Rh(1)	107.20(16)
C(14)-C(13)-H(13B)	113.4(14)	C(26)-C(25)-H(25)	112(2)
C(3)-C(13)-H(13B)	106.9(14)	C(24)-C(25)-H(25)	120(2)
H(13A)-C(13)-H(13B)	107.9(19)	Rh(1)-C(25)-H(25)	98(2)
C(15)-C(14)-C(13)	113.4(2)	C(25)-C(26)-C(27)	123.7(2)
C(15)-C(14)-H(14A)	110.1(15)	C(25)-C(26)-Rh(1)	70.43(14)

C(27)-C(26)-Rh(1)	110.14(15)	C(29)-C(28)-H(28A)	110.8(12)
C(25)-C(26)-H(26)	117.0(12)	C(27)-C(28)-H(28A)	111.4(12)
C(27)-C(26)-H(26)	117.7(12)	C(29)-C(28)-H(28B)	106.2(18)
Rh(1)-C(26)-H(26)	101.4(13)	C(27)-C(28)-H(28B)	110.6(17)
C(26)-C(27)-C(28)	112.58(19)	H(28A)-C(28)-H(28B)	105(2)
C(26)-C(27)-H(27A)	104.6(16)	C(22)-C(29)-C(28)	126.0(2)
C(28)-C(27)-H(27A)	111.5(17)	C(22)-C(29)-Rh(1)	71.18(12)
C(26)-C(27)-H(27B)	110.6(13)	C(28)-C(29)-Rh(1)	111.13(15)
C(28)-C(27)-H(27B)	107.1(13)	C(22)-C(29)-H(29)	119.2(16)
H(27A)-C(27)-H(27B)	111(2)	C(28)-C(29)-H(29)	110.4(16)
C(29)-C(28)-C(27)	112.44(19)	Rh(1)-C(29)-H(29)	110.2(16)

Table 5. Anisotropic displacement parameters ($Å^2 x 10^4$) for ICS05 (CCDC 696088). The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Rh(1)	111(1)	115(1)	108(1)	0(1)	-12(1)	-9(1)
Cl(1)	171(2)	188(3)	130(2)	9(2)	0(2)	-20(2)
N(1)	111(8)	126(9)	203(9)	-6(7)	-1(7)	3(7)
N(2)	109(8)	119(9)	196(9)	-31(7)	-5(7)	1(6)
C(1)	104(9)	152(9)	117(8)	13(9)	9(7)	-5(8)
C(2)	134(10)	131(10)	237(12)	-42(9)	30(9)	14(8)
C(3)	137(12)	145(13)	191(13)	-30(10)	29(9)	8(9)
C(4)	119(9)	159(11)	191(11)	-35(9)	-7(8)	27(8)
C(5)	152(10)	194(12)	172(10)	-26(9)	-17(8)	8(8)
C(6)	132(10)	250(13)	258(12)	-64(11)	27(9)	-17(10)
C(7)	123(11)	297(14)	270(13)	-80(11)	-41(10)	31(10)
C(8)	234(12)	261(14)	177(12)	-26(10)	-49(10)	93(10)
C(9)	195(11)	169(11)	157(10)	-9(9)	22(8)	56(8)
C(10)	265(15)	244(15)	194(14)	43(12)	36(11)	43(12)
C(11)	291(13)	183(12)	310(14)	82(11)	76(11)	36(10)
C(12)	206(12)	148(12)	287(13)	22(10)	60(10)	-8(9)
C(13)	163(11)	173(12)	309(13)	-67(10)	48(10)	-34(9)
C(14)	179(12)	304(14)	285(14)	-119(11)	27(10)	-41(10)
C(15)	160(12)	303(15)	207(12)	-46(11)	-14(10)	-23(10)
C(16)	127(9)	174(11)	189(10)	-7(10)	-7(8)	-28(9)
C(17)	114(10)	201(12)	288(13)	56(10)	-37(10)	-13(8)
C(18)	115(11)	187(12)	406(15)	21(11)	38(10)	35(9)
C(19)	185(12)	193(12)	323(14)	-63(10)	91(10)	1(9)
C(20)	143(10)	153(11)	208(11)	-17(9)	13(9)	-20(8)
C(21)	94(9)	109(9)	201(10)	5(9)	20(7)	0(8)
C(22)	165(10)	156(11)	104(10)	8(8)	-6(8)	-5(8)
C(23)	141(11)	217(12)	156(11)	53(9)	23(9)	-23(9)
C(24)	181(12)	241(13)	220(12)	54(10)	-49(9)	-79(10)
C(25)	241(12)	193(12)	151(11)	9(9)	-52(9)	-85(9)
C(26)	257(12)	130(11)	195(11)	-41(9)	19(9)	-27(9)
C(27)	226(12)	131(11)	277(13)	12(10)	71(10)	2(9)
C(28)	143(11)	187(12)	246(13)	48(10)	-2(9)	20(9)
C(29)	171(11)	158(11)	125(10)	24(9)	-30(8)	-30(8)

Table 6. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10^3) for ICS05 (CCDC 696088).

	x	y	z	U_{iso}
H(2)	7752(14)	10740(20)	6840(20)	14(6)
H(3)	8558(14)	9720(20)	7990(20)	13(7)
H(5)	6600(14)	8070(20)	6960(20)	14(6)
H(6)	5241(15)	7940(20)	6260(20)	24(7)
H(7)	4861(17)	8900(30)	4730(20)	36(9)
H(8)	5638(16)	9870(20)	3750(20)	29(7)
H(10A)	6834(14)	10510(20)	3410(20)	23(7)
H(10B)	7466(17)	9720(30)	3800(20)	22(8)
H(11A)	7270(14)	11810(20)	4940(19)	13(6)
H(11B)	7831(13)	11660(20)	4061(19)	11(6)
H(12A)	8661(13)	10370(20)	5175(18)	10(6)
H(12B)	8614(16)	11540(20)	5730(20)	27(7)
H(13A)	9337(13)	11480(20)	7568(18)	10(6)
H(13B)	9768(13)	10570(20)	6927(18)	9(6)
H(14A)	10532(15)	10620(20)	8610(20)	24(7)
H(14B)	9787(15)	10620(20)	9210(20)	24(7)
H(15A)	10406(16)	8830(30)	9400(20)	33(7)
H(15B)	9588(17)	8740(30)	9000(20)	31(8)
H(17)	11269(15)	7870(20)	8460(20)	20(7)
H(18)	11621(16)	6760(20)	7010(20)	30(7)
H(19)	10658(14)	6660(20)	5370(20)	22(6)
H(20)	9464(15)	7610(20)	5290(20)	27(7)
H(22)	7550(13)	6790(20)	7600(18)	9(6)
H(23A)	6721(13)	5450(20)	7617(18)	11(6)
H(23B)	7338(12)	4510(20)	7527(17)	3(5)
H(24A)	6502(15)	4290(20)	5900(20)	25(7)
H(24B)	6312(15)	5510(20)	5850(20)	22(7)
H(25)	7083(19)	5470(30)	4530(30)	42(9)
H(26)	8299(13)	4910(20)	4524(18)	11(6)
H(27A)	8743(15)	3650(20)	5870(20)	24(7)
H(27B)	8060(12)	3716(19)	6513(17)	5(5)
H(28A)	9025(12)	4414(19)	7809(19)	9(6)
H(28B)	9363(16)	5030(20)	6910(19)	18(7)
H(29)	8887(15)	6510(20)	7640(20)	28(7)

CALIFORNIA INSTITUTE OF TECHNOLOGY

BECKMAN INSTITUTE

X-RAY CRYSTALLOGRAPHY LABORATORY

Date 21 October 2009

Crystal Structure Analysis of:

ICS06

(shown below)

For Investigator: Ian Stewart ext. 6885

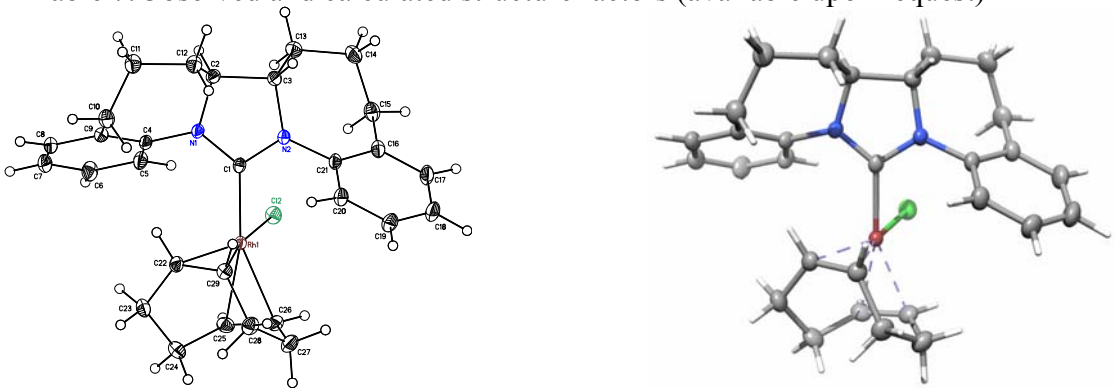
Advisor: R. H. Grubbs ext. 6003

Account Number: RHG.METAL-1-NIH.METAL2

By Michael W. Day 116 Beckman ext. 2734

e-mail: mikeday@caltech.edu

Contents



ICS06

Note: The crystallographic data have been deposited in the Cambridge Database (CCDC) and has been placed on hold pending further instructions from me. The deposition number is 752022. Ideally the CCDC would like the publication to contain a footnote of the type: "Crystallographic data have been deposited at the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK and copies can be obtained on request, free of charge, by quoting the publication citation and the deposition number 752022."

Table 1. Crystal data and structure refinement for ICS06 (CCDC 752022).

Empirical formula	$C_{29}H_{34}N_2ClRh \bullet C_7H_8$
Formula weight	641.08
Crystallization Solvent	Toluene
Crystal Habit	Fragment
Crystal size	0.27 x 0.19 x 0.17 mm^3
Crystal color	Yellow

Data Collection

Type of diffractometer	Bruker KAPPA APEX II	
Wavelength	0.71073 Å MoKα	
Data Collection Temperature	100(2) K	
θ range for 9500 reflections used in lattice determination	3.30 to 43.38°	
Unit cell dimensions	a = 10.1852(4) Å b = 11.0172(4) Å c = 14.5013(5) Å	α= 94.162(2)° β= 91.706(2)° γ = 109.954(2)°
Volume	1522.92(10) $Å^3$	
Z	2	
Crystal system	Triclinic	
Space group	P-1	
Density (calculated)	1.398 Mg/m^3	
F(000)	668	
Data collection program	Bruker APEX2 v2.1-0	
θ range for data collection	1.97 to 43.53°	
Completeness to θ = 43.53°	92.4 %	
Index ranges	$-18 \le h \le 19$, $-20 \le k \le 20$, $-24 \le l \le 27$	
Data collection scan type	ω scans; 17 settings	
Data reduction program	Bruker SAINT-Plus v7.34A	
Reflections collected	68936	
Independent reflections	21461 [R_{int}= 0.0256]	
Absorption coefficient	0.676 mm^{-1}	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7485 and 0.6719	

Table 1 (cont.)

Structure solution and Refinement

Structure solution program	SHELXS-97 (Sheldrick, 2008)
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Difference Fourier map
Structure refinement program	SHELXL-97 (Sheldrick, 2008)
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	21461 / 0 / 518
Treatment of hydrogen atoms	Unrestrained (see below)
Goodness-of-fit on F^2	1.887
Final R indices [$I>2\sigma(I)$, 18607 reflections]	R1 = 0.0274, *w*R2 = 0.0505
R indices (all data)	R1 = 0.0361, *w*R2 = 0.0513
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Max shift/error	0.002
Average shift/error	0.000
Largest diff. peak and hole	1.251 and -0.706 e.Å^{-3}

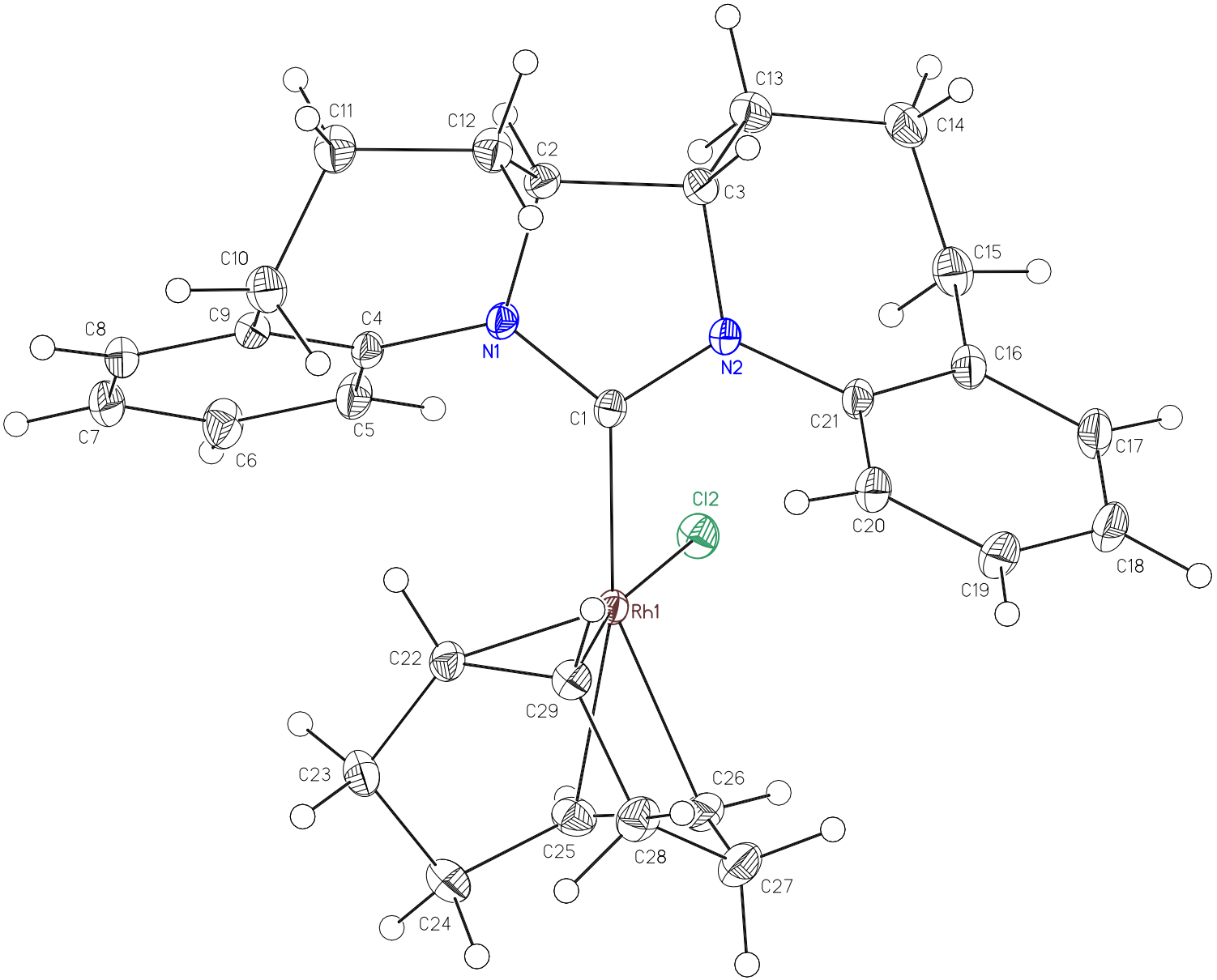
Special Refinement Details

Crystals were mounted on a glass fiber using Paratone oil then placed on the diffractometer under a nitrogen stream at 100K.

The methyl hydrogens of the toluene were refined as riding atoms. All other hydrogens were refined without restraint.

Refinement of F^2 against ALL reflections. The weighted R-factor (*w*R) and goodness of fit (S) are based on F^2, conventional R-factors (R) are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.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C1
C21
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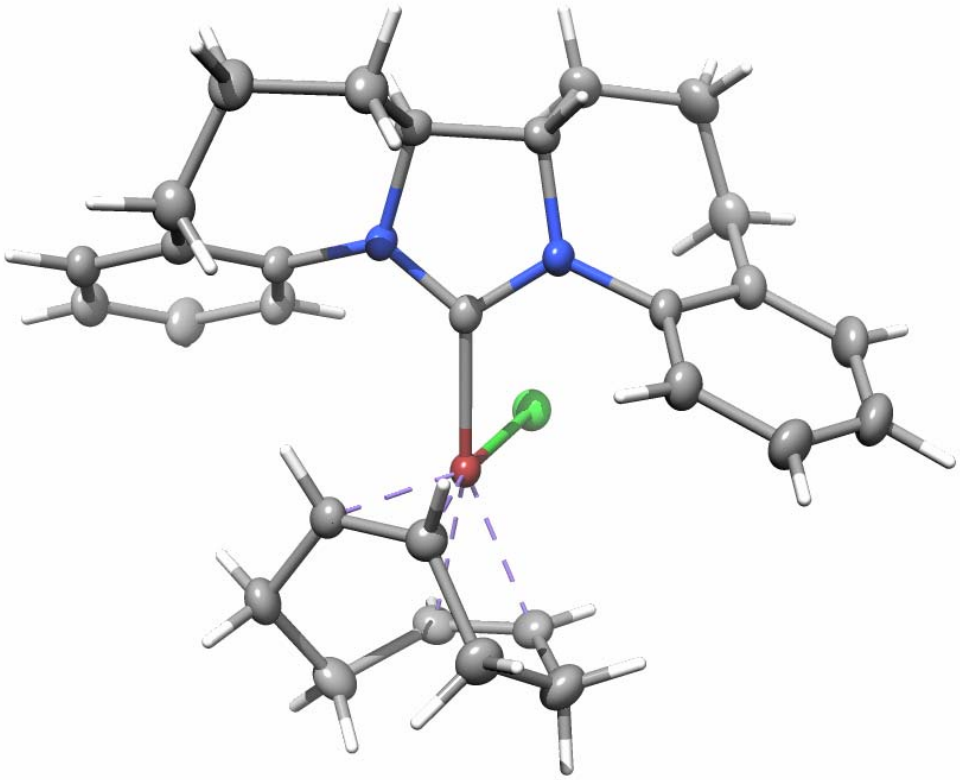

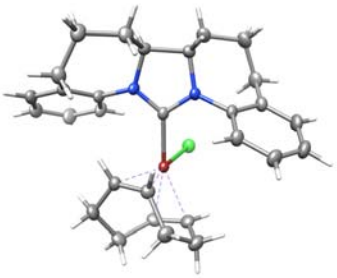

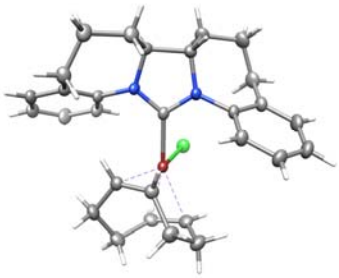

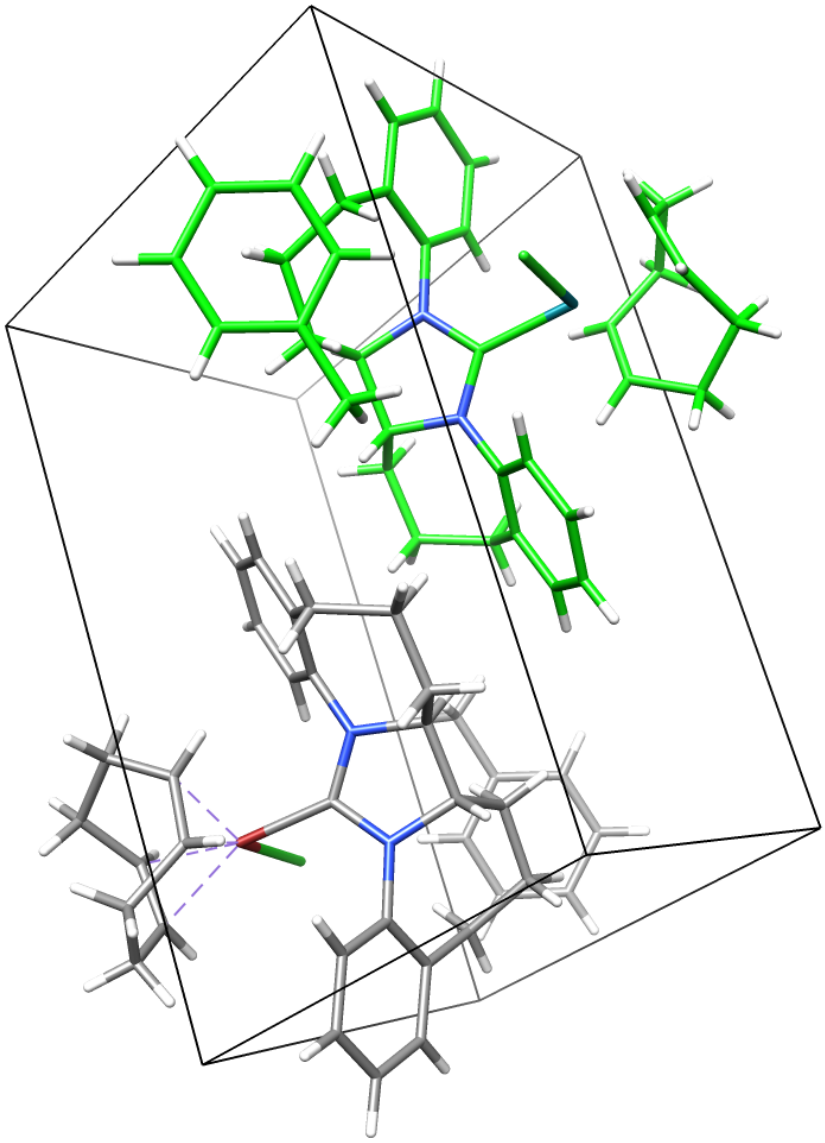

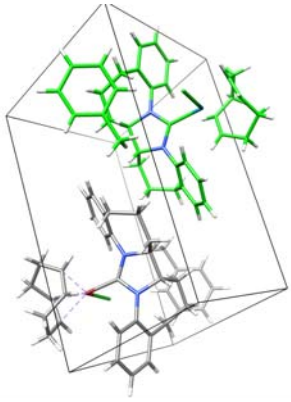

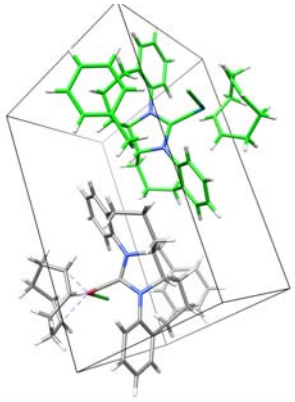

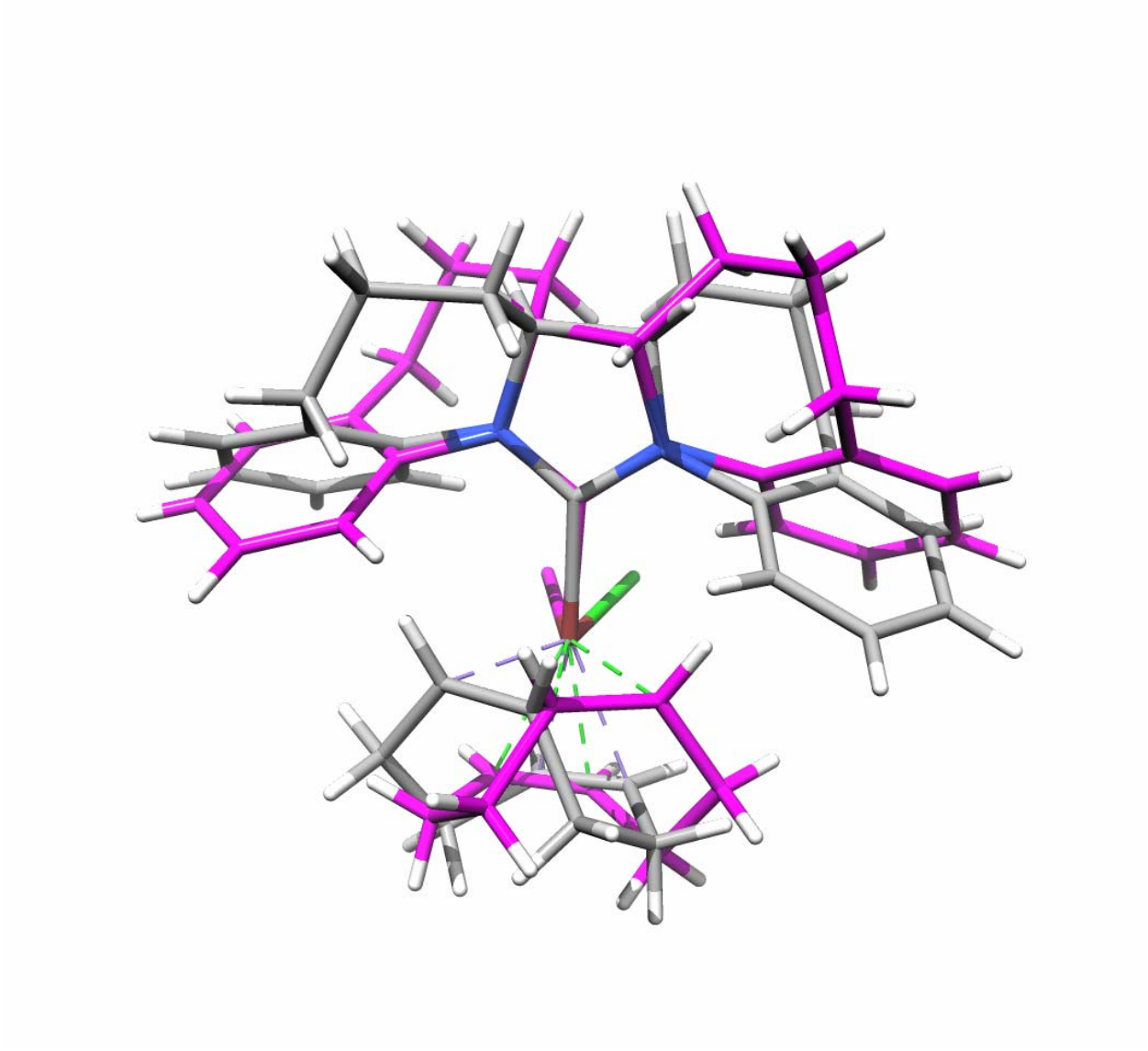

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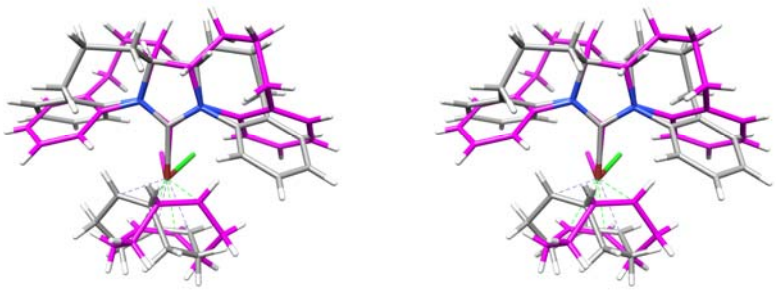

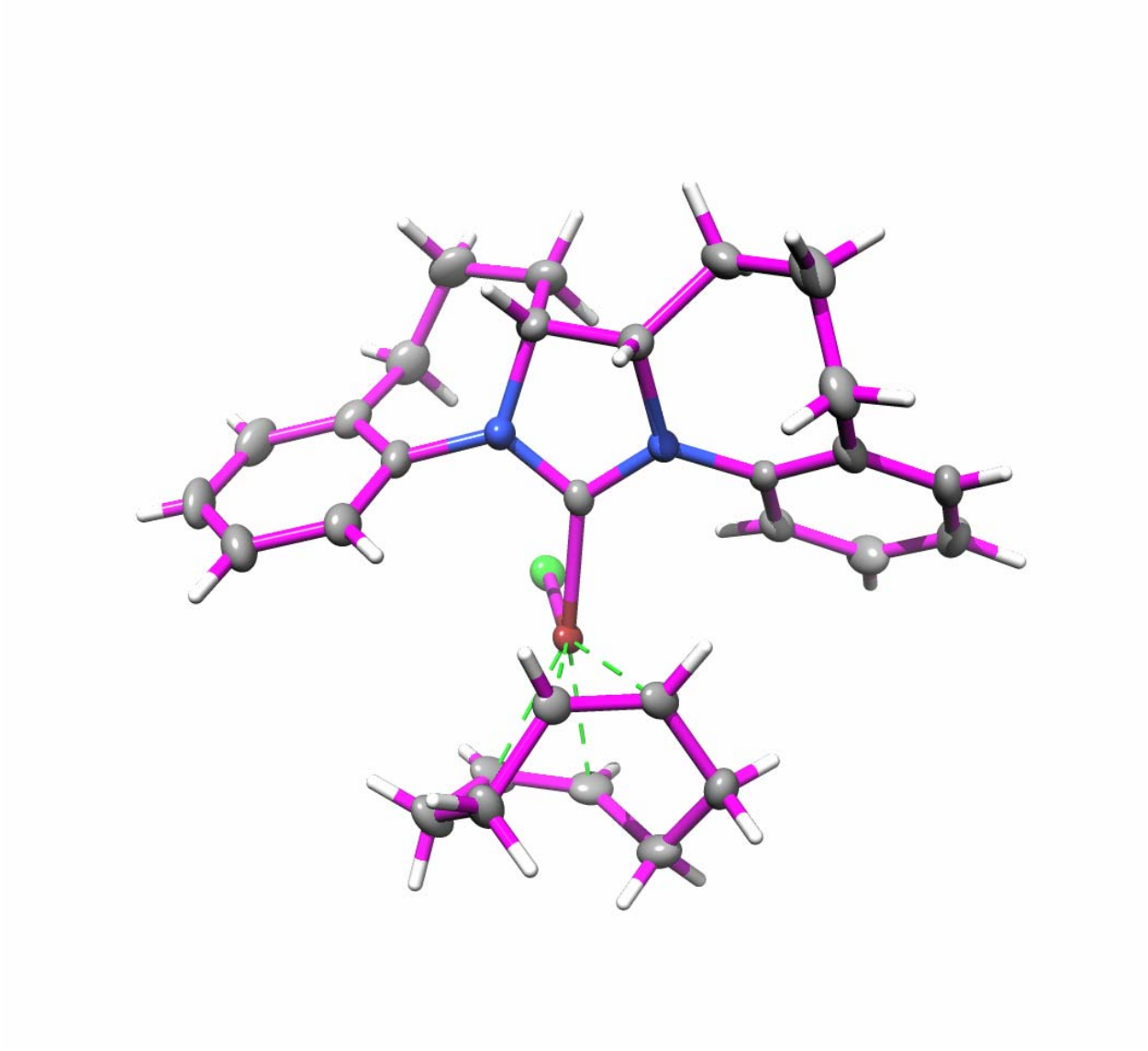

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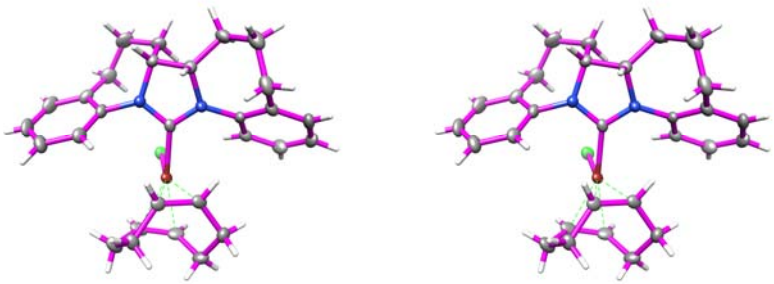

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for ICS06 (CCDC 752022). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
Rh(1)	9771(1)	7323(1)	2653(1)	11(1)
Cl(2)	10006(1)	5244(1)	2375(1)	17(1)
N(1)	6851(1)	6144(1)	3335(1)	13(1)
N(2)	6899(1)	5910(1)	1838(1)	12(1)
C(1)	7696(1)	6422(1)	2623(1)	12(1)
C(2)	5358(1)	5467(1)	3032(1)	14(1)
C(3)	5471(1)	5052(1)	2006(1)	14(1)
C(4)	7338(1)	6280(1)	4285(1)	13(1)
C(5)	8316(1)	5726(1)	4536(1)	17(1)
C(6)	8791(1)	5843(1)	5463(1)	21(1)
C(7)	8271(1)	6502(1)	6126(1)	20(1)
C(8)	7297(1)	7050(1)	5866(1)	17(1)
C(9)	6811(1)	6965(1)	4938(1)	14(1)
C(10)	5822(1)	7643(1)	4666(1)	18(1)
C(11)	4416(1)	6757(1)	4201(1)	19(1)
C(12)	4483(1)	6326(1)	3181(1)	17(1)
C(13)	5308(1)	3624(1)	1848(1)	19(1)
C(14)	5550(1)	3167(1)	868(1)	21(1)
C(15)	7054(1)	3778(1)	580(1)	20(1)
C(16)	7447(1)	5143(1)	313(1)	16(1)
C(17)	7963(1)	5458(1)	-556(1)	21(1)
C(18)	8397(1)	6721(1)	-802(1)	23(1)
C(19)	8307(1)	7710(1)	-186(1)	21(1)
C(20)	7790(1)	7422(1)	680(1)	17(1)
C(21)	7389(1)	6161(1)	929(1)	14(1)
C(22)	9753(1)	8874(1)	3574(1)	15(1)
C(23)	11065(1)	9347(1)	4207(1)	20(1)
C(24)	12375(1)	9345(1)	3717(1)	19(1)
C(25)	12056(1)	8212(1)	2984(1)	17(1)
C(26)	11768(1)	8287(1)	2057(1)	17(1)
C(27)	11678(1)	9470(1)	1633(1)	21(1)
C(28)	10798(1)	10137(1)	2169(1)	19(1)
C(29)	9622(1)	9202(1)	2660(1)	15(1)
C(41)	6888(1)	1062(1)	2658(1)	25(1)
C(42)	7894(1)	1586(1)	2030(1)	24(1)
C(43)	7728(1)	1069(1)	1116(1)	25(1)
C(44)	6549(1)	29(1)	811(1)	28(1)
C(45)	5540(1)	-504(1)	1424(1)	32(1)
C(46)	5715(1)	11(1)	2340(1)	30(1)
C(47)	7068(2)	1644(1)	3650(1)	45(1)

Table 3. Selected bond lengths [Å] and angles [°] for ICS06 (CCDC 752022).

Rh(1)-C(1)	2.0032(7)	C(1)-Rh(1)-C(22)	94.10(3)
Rh(1)-C(22)	2.0969(7)	C(1)-Rh(1)-C(29)	93.79(3)
Rh(1)-C(29)	2.1263(8)	C(22)-Rh(1)-C(29)	39.06(3)
Rh(1)-C(26)	2.1847(8)	C(1)-Rh(1)-C(26)	155.17(3)
Rh(1)-C(25)	2.2169(8)	C(22)-Rh(1)-C(26)	97.92(3)
Rh(1)-Cl(2)	2.3896(2)	C(29)-Rh(1)-C(26)	82.28(3)
		C(1)-Rh(1)-C(25)	168.18(3)
		C(22)-Rh(1)-C(25)	81.66(3)
		C(29)-Rh(1)-C(25)	89.83(3)
		C(26)-Rh(1)-C(25)	36.58(3)
		C(1)-Rh(1)-Cl(2)	88.20(2)
		C(22)-Rh(1)-Cl(2)	150.23(2)
		C(29)-Rh(1)-Cl(2)	170.374(19)
		C(26)-Rh(1)-Cl(2)	91.96(2)
		C(25)-Rh(1)-Cl(2)	90.10(2)

Table 4. Bond lengths [Å] and angles [°] for ICS06 (CCDC 752022).

Rh(1)-C(1)	2.0032(7)	C(19)-C(20)	1.3917(10)
Rh(1)-C(22)	2.0969(7)	C(19)-H(19)	0.936(11)
Rh(1)-C(29)	2.1263(8)	C(20)-C(21)	1.3863(11)
Rh(1)-C(26)	2.1847(8)	C(20)-H(20)	0.949(11)
Rh(1)-C(25)	2.2169(8)	C(22)-C(29)	1.4120(10)
Rh(1)-Cl(2)	2.3896(2)	C(22)-C(23)	1.5114(11)
N(1)-C(1)	1.3487(9)	C(22)-H(22)	0.956(10)
N(1)-C(4)	1.4294(8)	C(23)-C(24)	1.5307(13)
N(1)-C(2)	1.4831(9)	C(23)-H(23A)	1.009(10)
N(2)-C(1)	1.3465(8)	C(23)-H(23B)	0.970(11)
N(2)-C(21)	1.4359(8)	C(24)-C(25)	1.5177(11)
N(2)-C(3)	1.4780(10)	C(24)-H(24A)	1.002(11)
C(2)-C(12)	1.5156(12)	C(24)-H(24B)	0.987(11)
C(2)-C(3)	1.5440(9)	C(25)-C(26)	1.3817(11)
C(2)-H(2)	0.970(9)	C(25)-H(25)	0.936(10)
C(3)-C(13)	1.5234(11)	C(26)-C(27)	1.5102(12)
C(3)-H(3)	1.014(10)	C(26)-H(26)	0.953(10)
C(4)-C(5)	1.3871(11)	C(27)-C(28)	1.5343(13)
C(4)-C(9)	1.3992(10)	C(27)-H(27A)	0.995(10)
C(5)-C(6)	1.3970(10)	C(27)-H(27B)	0.973(12)
C(5)-H(5)	0.946(11)	C(28)-C(29)	1.5227(11)
C(6)-C(7)	1.3872(12)	C(28)-H(28A)	0.994(11)
C(6)-H(6)	0.935(12)	C(28)-H(28B)	0.950(11)
C(7)-C(8)	1.3828(13)	C(29)-H(29)	0.955(12)
C(7)-H(7)	0.967(10)	C(41)-C(46)	1.3878(14)
C(8)-C(9)	1.4054(9)	C(41)-C(42)	1.3923(13)
C(8)-H(8)	0.954(11)	C(41)-C(47)	1.5121(14)
C(9)-C(10)	1.5038(12)	C(42)-C(43)	1.3874(13)
C(10)-C(11)	1.5333(12)	C(42)-H(42)	0.899(12)
C(10)-H(10A)	0.990(10)	C(43)-C(44)	1.3805(14)
C(10)-H(10B)	0.938(11)	C(43)-H(43)	0.949(13)
C(11)-C(12)	1.5313(10)	C(44)-C(45)	1.3819(15)
C(11)-H(11A)	0.996(10)	C(44)-H(44)	0.958(13)
C(11)-H(11B)	0.988(12)	C(45)-C(46)	1.3882(15)
C(12)-H(12A)	0.950(11)	C(45)-H(45)	0.975(14)
C(12)-H(12B)	0.956(11)	C(46)-H(46)	0.906(14)
C(13)-C(14)	1.5293(11)	C(47)-H(47A)	0.9800
C(13)-H(13A)	0.964(11)	C(47)-H(47B)	0.9800
C(13)-H(13B)	1.002(10)	C(47)-H(47C)	0.9800
C(14)-C(15)	1.5325(13)		
C(14)-H(14A)	0.984(11)	C(1)-Rh(1)-C(22)	94.10(3)
C(14)-H(14B)	0.957(10)	C(1)-Rh(1)-C(29)	93.79(3)
C(15)-C(16)	1.5022(12)	C(22)-Rh(1)-C(29)	39.06(3)
C(15)-H(15A)	0.972(11)	C(1)-Rh(1)-C(26)	155.17(3)
C(15)-H(15B)	0.960(11)	C(22)-Rh(1)-C(26)	97.92(3)
C(16)-C(21)	1.4005(10)	C(29)-Rh(1)-C(26)	82.28(3)
C(16)-C(17)	1.4009(10)	C(1)-Rh(1)-C(25)	168.18(3)
C(17)-C(18)	1.3859(13)	C(22)-Rh(1)-C(25)	81.66(3)
C(17)-H(17)	0.952(11)	C(29)-Rh(1)-C(25)	89.83(3)
C(18)-C(19)	1.3881(12)	C(26)-Rh(1)-C(25)	36.58(3)
C(18)-H(18)	0.993(11)	C(1)-Rh(1)-Cl(2)	88.20(2)

C(22)-Rh(1)-Cl(2)	150.23(2)	H(11A)-C(11)-H(11B)	105.4(9)
C(29)-Rh(1)-Cl(2)	170.374(19)	C(2)-C(12)-C(11)	113.54(6)
C(26)-Rh(1)-Cl(2)	91.96(2)	C(2)-C(12)-H(12A)	109.3(7)
C(25)-Rh(1)-Cl(2)	90.10(2)	C(11)-C(12)-H(12A)	107.5(6)
C(1)-N(1)-C(4)	124.25(6)	C(2)-C(12)-H(12B)	108.8(7)
C(1)-N(1)-C(2)	113.12(5)	C(11)-C(12)-H(12B)	109.3(6)
C(4)-N(1)-C(2)	121.88(6)	H(12A)-C(12)-H(12B)	108.2(9)
C(1)-N(2)-C(21)	123.53(6)	C(3)-C(13)-C(14)	115.51(6)
C(1)-N(2)-C(3)	113.17(5)	C(3)-C(13)-H(13A)	106.0(7)
C(21)-N(2)-C(3)	123.29(5)	C(14)-C(13)-H(13A)	109.3(6)
N(2)-C(1)-N(1)	107.40(6)	C(3)-C(13)-H(13B)	108.0(6)
N(2)-C(1)-Rh(1)	123.40(5)	C(14)-C(13)-H(13B)	107.3(6)
N(1)-C(1)-Rh(1)	129.10(5)	H(13A)-C(13)-H(13B)	110.7(8)
N(1)-C(2)-C(12)	112.47(6)	C(13)-C(14)-C(15)	114.21(6)
N(1)-C(2)-C(3)	101.40(6)	C(13)-C(14)-H(14A)	109.5(6)
C(12)-C(2)-C(3)	114.32(6)	C(15)-C(14)-H(14A)	106.9(7)
N(1)-C(2)-H(2)	107.8(6)	C(13)-C(14)-H(14B)	111.7(6)
C(12)-C(2)-H(2)	111.1(6)	C(15)-C(14)-H(14B)	109.0(6)
C(3)-C(2)-H(2)	109.2(5)	H(14A)-C(14)-H(14B)	105.0(9)
N(2)-C(3)-C(13)	111.95(7)	C(16)-C(15)-C(14)	115.62(8)
N(2)-C(3)-C(2)	101.27(5)	C(16)-C(15)-H(15A)	108.2(7)
C(13)-C(3)-C(2)	112.25(6)	C(14)-C(15)-H(15A)	111.1(7)
N(2)-C(3)-H(3)	109.6(6)	C(16)-C(15)-H(15B)	108.0(6)
C(13)-C(3)-H(3)	110.8(6)	C(14)-C(15)-H(15B)	111.4(7)
C(2)-C(3)-H(3)	110.6(6)	H(15A)-C(15)-H(15B)	101.6(10)
C(5)-C(4)-C(9)	121.71(6)	C(21)-C(16)-C(17)	117.25(7)
C(5)-C(4)-N(1)	119.15(6)	C(21)-C(16)-C(15)	122.05(6)
C(9)-C(4)-N(1)	119.13(7)	C(17)-C(16)-C(15)	120.61(7)
C(4)-C(5)-C(6)	119.70(7)	C(18)-C(17)-C(16)	121.72(7)
C(4)-C(5)-H(5)	120.6(6)	C(18)-C(17)-H(17)	119.5(6)
C(6)-C(5)-H(5)	119.6(6)	C(16)-C(17)-H(17)	118.7(6)
C(7)-C(6)-C(5)	119.74(8)	C(17)-C(18)-C(19)	119.96(7)
C(7)-C(6)-H(6)	119.1(6)	C(17)-C(18)-H(18)	120.7(7)
C(5)-C(6)-H(6)	121.1(6)	C(19)-C(18)-H(18)	119.4(7)
C(8)-C(7)-C(6)	119.94(7)	C(20)-C(19)-C(18)	119.47(8)
C(8)-C(7)-H(7)	120.2(7)	C(20)-C(19)-H(19)	118.2(6)
C(6)-C(7)-H(7)	119.8(7)	C(18)-C(19)-H(19)	122.3(6)
C(7)-C(8)-C(9)	121.76(7)	C(21)-C(20)-C(19)	120.18(7)
C(7)-C(8)-H(8)	120.0(6)	C(21)-C(20)-H(20)	117.9(6)
C(9)-C(8)-H(8)	118.1(6)	C(19)-C(20)-H(20)	121.9(6)
C(8)-C(9)-C(4)	117.13(7)	C(20)-C(21)-C(16)	121.39(6)
C(8)-C(9)-C(10)	120.68(7)	C(20)-C(21)-N(2)	118.51(6)
C(4)-C(9)-C(10)	122.13(6)	C(16)-C(21)-N(2)	120.10(7)
C(9)-C(10)-C(11)	115.20(7)	C(29)-C(22)-C(23)	126.37(7)
C(9)-C(10)-H(10A)	107.7(7)	C(29)-C(22)-Rh(1)	71.60(4)
C(11)-C(10)-H(10A)	110.5(6)	C(23)-C(22)-Rh(1)	110.29(6)
C(9)-C(10)-H(10B)	108.3(7)	C(29)-C(22)-H(22)	115.9(5)
C(11)-C(10)-H(10B)	109.7(7)	C(23)-C(22)-H(22)	113.7(5)
H(10A)-C(10)-H(10B)	104.9(9)	Rh(1)-C(22)-H(22)	108.4(6)
C(12)-C(11)-C(10)	114.24(7)	C(22)-C(23)-C(24)	113.18(6)
C(12)-C(11)-H(11A)	109.3(5)	C(22)-C(23)-H(23A)	104.9(6)
C(10)-C(11)-H(11A)	109.8(6)	C(24)-C(23)-H(23A)	109.3(7)
C(12)-C(11)-H(11B)	110.6(6)	C(22)-C(23)-H(23B)	109.8(7)
C(10)-C(11)-H(11B)	107.1(6)	C(24)-C(23)-H(23B)	111.2(7)

H(23A)-C(23)-H(23B)	108.1(8)	C(22)-C(29)-C(28)	124.47(7)
C(25)-C(24)-C(23)	111.84(7)	C(22)-C(29)-Rh(1)	69.35(4)
C(25)-C(24)-H(24A)	110.7(6)	C(28)-C(29)-Rh(1)	112.76(5)
C(23)-C(24)-H(24A)	110.2(7)	C(22)-C(29)-H(29)	115.5(6)
C(25)-C(24)-H(24B)	109.3(7)	C(28)-C(29)-H(29)	114.8(6)
C(23)-C(24)-H(24B)	110.1(7)	Rh(1)-C(29)-H(29)	110.0(7)
H(24A)-C(24)-H(24B)	104.4(9)	C(46)-C(41)-C(42)	118.08(8)
C(26)-C(25)-C(24)	123.77(8)	C(46)-C(41)-C(47)	121.40(10)
C(26)-C(25)-Rh(1)	70.44(5)	C(42)-C(41)-C(47)	120.51(10)
C(24)-C(25)-Rh(1)	110.73(6)	C(43)-C(42)-C(41)	120.85(9)
C(26)-C(25)-H(25)	118.2(6)	C(43)-C(42)-H(42)	121.5(7)
C(24)-C(25)-H(25)	116.5(6)	C(41)-C(42)-H(42)	117.6(7)
Rh(1)-C(25)-H(25)	100.9(6)	C(44)-C(43)-C(42)	120.24(9)
C(25)-C(26)-C(27)	125.68(7)	C(44)-C(43)-H(43)	119.9(7)
C(25)-C(26)-Rh(1)	72.98(5)	C(42)-C(43)-H(43)	119.8(7)
C(27)-C(26)-Rh(1)	108.06(6)	C(45)-C(44)-C(43)	119.69(9)
C(25)-C(26)-H(26)	116.0(6)	C(45)-C(44)-H(44)	118.2(8)
C(27)-C(26)-H(26)	116.8(6)	C(43)-C(44)-H(44)	122.1(8)
Rh(1)-C(26)-H(26)	100.9(6)	C(44)-C(45)-C(46)	119.88(9)
C(26)-C(27)-C(28)	113.61(6)	C(44)-C(45)-H(45)	118.5(8)
C(26)-C(27)-H(27A)	109.6(7)	C(46)-C(45)-H(45)	121.6(8)
C(28)-C(27)-H(27A)	107.1(7)	C(41)-C(46)-C(45)	121.25(9)
C(26)-C(27)-H(27B)	110.1(7)	C(41)-C(46)-H(46)	117.1(9)
C(28)-C(27)-H(27B)	109.9(7)	C(45)-C(46)-H(46)	121.6(9)
H(27A)-C(27)-H(27B)	106.3(9)	C(41)-C(47)-H(47A)	109.5
C(29)-C(28)-C(27)	113.26(7)	C(41)-C(47)-H(47B)	109.5
C(29)-C(28)-H(28A)	110.7(6)	H(47A)-C(47)-H(47B)	109.5
C(27)-C(28)-H(28A)	110.7(7)	C(41)-C(47)-H(47C)	109.5
C(29)-C(28)-H(28B)	107.6(7)	H(47A)-C(47)-H(47C)	109.5
C(27)-C(28)-H(28B)	111.2(7)	H(47B)-C(47)-H(47C)	109.5
H(28A)-C(28)-H(28B)	102.9(9)		

Table 5. Anisotropic displacement parameters ($Å^2 x 10^4$) for ICS06 (CCDC 752022). The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Rh(1)	114(1)	121(1)	97(1)	3(1)	11(1)	46(1)
Cl(2)	195(1)	151(1)	180(1)	-2(1)	24(1)	85(1)
N(1)	113(3)	177(3)	92(2)	20(2)	6(2)	50(2)
N(2)	131(3)	143(2)	93(2)	13(2)	6(2)	39(2)
C(1)	139(3)	121(3)	103(2)	17(2)	13(2)	60(2)
C(2)	116(3)	163(3)	126(2)	27(2)	10(2)	43(2)
C(3)	127(3)	166(3)	122(2)	13(2)	-4(2)	41(2)
C(4)	128(3)	165(3)	95(2)	24(2)	7(2)	48(2)
C(5)	170(3)	254(4)	121(2)	23(2)	12(2)	105(3)
C(6)	195(4)	315(4)	143(3)	34(3)	-15(2)	126(3)
C(7)	202(4)	259(4)	115(3)	16(2)	-14(2)	63(3)
C(8)	187(4)	177(3)	118(2)	-5(2)	16(2)	36(3)
C(9)	139(3)	140(3)	122(2)	14(2)	14(2)	38(2)
C(10)	212(4)	169(3)	169(3)	-2(2)	8(3)	89(3)
C(11)	174(4)	255(4)	172(3)	3(3)	10(2)	123(3)
C(12)	158(3)	224(4)	153(3)	13(2)	-4(2)	94(3)
C(13)	203(4)	151(3)	178(3)	4(2)	10(3)	21(3)
C(14)	237(4)	168(3)	192(3)	-35(3)	-13(3)	38(3)
C(15)	246(4)	181(3)	180(3)	-27(3)	9(3)	90(3)
C(16)	177(3)	194(3)	124(2)	-7(2)	4(2)	79(3)
C(17)	230(4)	283(4)	129(3)	-25(3)	27(3)	95(3)
C(18)	246(4)	314(4)	121(3)	40(3)	40(3)	80(3)
C(19)	240(4)	236(4)	150(3)	74(3)	35(3)	62(3)
C(20)	204(4)	188(3)	124(2)	25(2)	10(2)	76(3)
C(21)	148(3)	172(3)	92(2)	14(2)	5(2)	62(2)
C(22)	168(3)	142(3)	137(2)	-7(2)	31(2)	53(2)
C(23)	228(4)	200(3)	134(3)	-25(2)	-10(2)	51(3)
C(24)	178(4)	194(3)	194(3)	-19(3)	-40(3)	53(3)
C(25)	132(3)	179(3)	203(3)	-3(2)	-7(2)	57(3)
C(26)	144(3)	186(3)	179(3)	0(2)	46(2)	50(3)
C(27)	215(4)	223(4)	184(3)	57(3)	66(3)	59(3)
C(28)	222(4)	164(3)	183(3)	54(2)	32(3)	66(3)
C(29)	156(3)	141(3)	155(3)	15(2)	14(2)	62(2)
C(41)	252(4)	286(4)	262(4)	66(3)	12(3)	146(4)
C(42)	195(4)	208(4)	314(4)	44(3)	-11(3)	64(3)
C(43)	231(4)	234(4)	308(4)	71(3)	62(3)	106(3)
C(44)	294(5)	249(4)	316(4)	-30(3)	-4(4)	128(4)
C(45)	219(5)	221(4)	484(5)	-12(4)	7(4)	45(3)
C(46)	224(5)	293(5)	413(5)	120(4)	107(4)	96(4)
C(47)	532(8)	612(8)	258(4)	30(5)	-15(5)	268(7)

Table 6. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10^3) for ICS06 (CCDC 752022).

	x	y	z	U_{iso}
H(2)	5013(11)	4700(9)	3367(6)	12(2)
H(3)	4765(12)	5259(10)	1596(6)	18(2)
H(5)	8662(12)	5266(10)	4085(6)	20(3)
H(6)	9423(13)	5448(11)	5650(7)	25(3)
H(7)	8612(13)	6597(11)	6766(7)	30(3)
H(8)	6969(12)	7536(10)	6317(6)	19(3)
H(10A)	6313(12)	8316(10)	4257(6)	20(3)
H(10B)	5667(12)	8104(11)	5196(7)	22(3)
H(11A)	4029(11)	5978(10)	4549(6)	14(2)
H(11B)	3751(12)	7231(11)	4269(7)	23(3)
H(12A)	3552(12)	5870(10)	2941(6)	21(3)
H(12B)	4858(12)	7074(11)	2846(6)	20(3)
H(13A)	4366(12)	3145(10)	1997(6)	21(3)
H(13B)	6004(12)	3459(10)	2279(6)	18(2)
H(14A)	5337(12)	2223(11)	819(7)	23(3)
H(14B)	4923(12)	3307(10)	417(6)	20(3)
H(15A)	7725(12)	3760(11)	1065(7)	24(3)
H(15B)	7254(12)	3261(11)	79(7)	24(3)
H(17)	8062(12)	4790(10)	-973(6)	20(3)
H(18)	8789(13)	6931(11)	-1413(7)	27(3)
H(19)	8543(12)	8565(11)	-340(6)	20(3)
H(20)	7663(12)	8063(10)	1106(6)	19(3)
H(22)	8908(11)	8603(9)	3894(6)	12(2)
H(23A)	10882(12)	8720(11)	4702(7)	22(3)
H(23B)	11185(12)	10200(11)	4503(7)	21(3)
H(24A)	12812(13)	10187(11)	3439(7)	26(3)
H(24B)	13098(13)	9310(11)	4172(7)	26(3)
H(25)	12268(11)	7493(10)	3153(6)	14(2)
H(26)	11835(11)	7603(10)	1641(6)	15(2)
H(27A)	11241(12)	9218(11)	993(7)	25(3)
H(27B)	12612(13)	10088(11)	1575(7)	30(3)
H(28A)	11403(13)	10853(11)	2613(7)	26(3)
H(28B)	10388(12)	10572(11)	1770(7)	22(3)
H(29)	8702(13)	9136(11)	2448(7)	23(3)
H(47A)	6774	2404	3687	54
H(47B)	8054	1904	3867	54
H(47C)	6494	999	4040	54
H(42)	8627(14)	2296(12)	2233(7)	32(3)
H(43)	8424(14)	1437(12)	697(8)	35(3)
H(44)	6407(15)	-349(13)	184(9)	46(4)
H(45)	4704(15)	-1224(13)	1193(8)	45(4)
H(46)	5080(16)	-332(14)	2759(8)	48(4)