Supporting Information for

Catalytic Arene H/D Exchange with Novel Rhodium and Iridium Complexes

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Complete reference 41:

(41) Gaussian 09, Revision A.1, Frisch, M. J.; Trucks, G. W.; Schlegel, H. B.; Scuseria, G. E.; Robb, M. A.; Cheeseman, J. R.; Scalmani, G.; Barone, V.; Mennucci, B.; Petersson, G. A.; Nakatsuji, H.; Caricato, M.; Li, X.; Hratchian, H. P.; Izmaylov, A. F.; Bloino, J.; Zheng, G.; Sonnenberg, J. L.; Hada, M.; Ehara, M.; Toyota, K.; Fukuda, R.; Hasegawa, J.; Ishida, M.; Nakajima, T.; Honda, Y.; Kitao, O.; Nakai, H.; Vreven, T.; Montgomery, Jr., J. A.; Peralta, J. E.; Ogliaro, F.; Bearpark, M.; Heyd, J. J.; Brothers, E.; Kudin, K. N.; Staroverov, V. N.; Kobayashi, R.; Normand, J.; Raghavachari, K.; Rendell, A.; Burant, J. C.; Iyengar, S. S.; Tomasi, J.; Cossi, M.; Rega, N.; Millam, N. J.; Klene, M.; Knox, J. E.; Cross, J. B.; Bakken, V.; Adamo, C.; Jaramillo, J.; Gomperts, R.; Stratmann, R. E.; Yazyev, O.; Austin, A. J.; Cammi, R.; Pomelli, C.; Ochterski, J. W.; Martin, R. L.; Morokuma, K.; Zakrzewski, V. G.; Voth, G. A.; Salvador, P.; Dannenberg, J. J.; Dapprich, S.; Daniels, A. D.; Farkas, Ö.; Foresman, J. B.; Ortiz, J. V.; Cioslowski, J.; Fox, D. J. Gaussian, Inc., Wallingford CT, 2009.

H/D exchange data for all substrates with **Na-2** and **3b**

Selected ^{2}H NMR spectra for arene alkyl β-C–H bond exchange

DFT calculated energies and structures.

X-ray structural procedures and data for **Li-1**, **Et_4N-1**, **Na-2**, and **3a**.

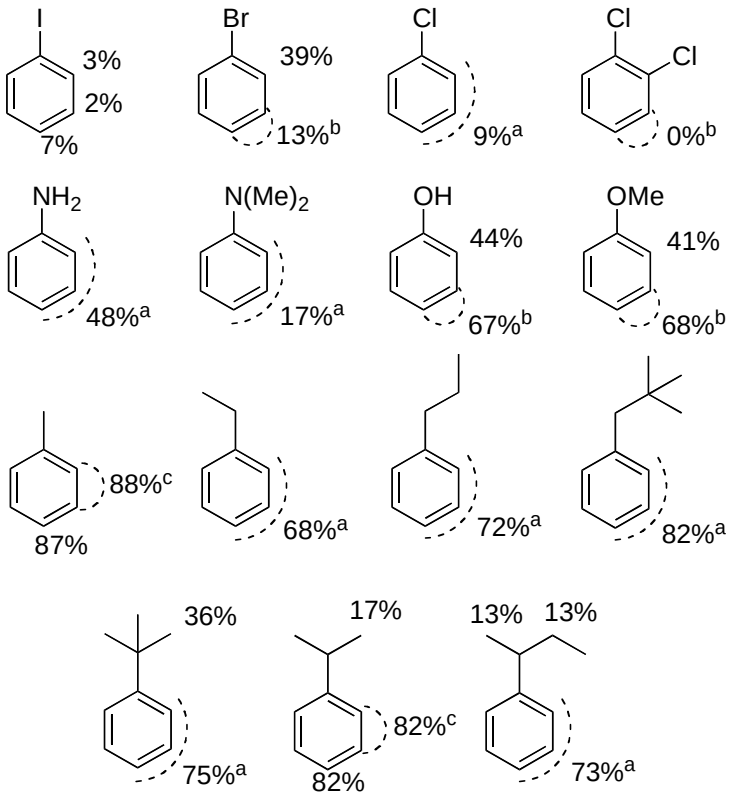


Figure 1S. Results of H/D exchange of substituted benzenes with **Na-2**. Average percentage of deuterium incorporation for a) all, b) meta plus para, and c) ortho plus para positions on the ring.

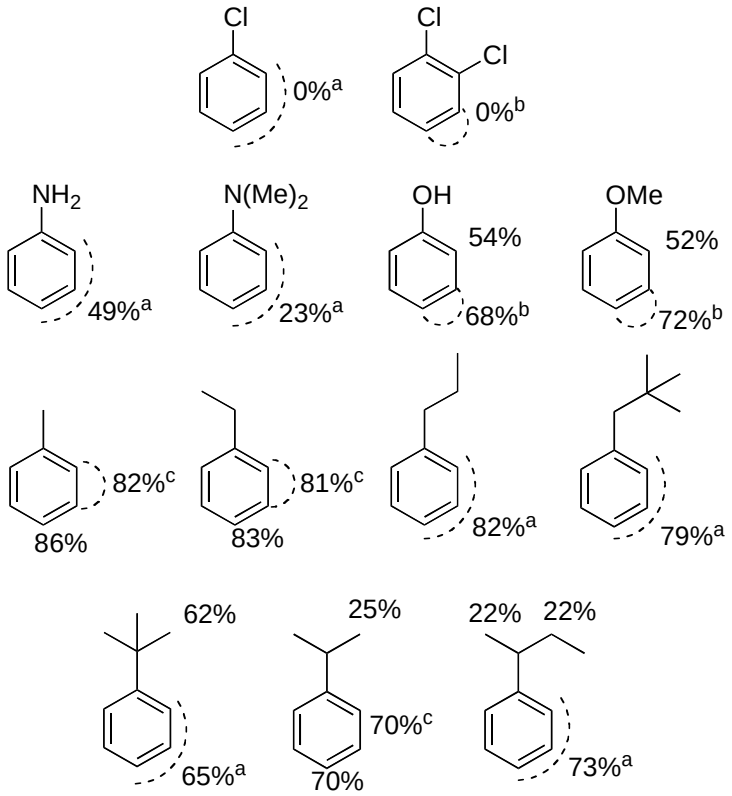


Figure 2S. Results of H/D exchange of substituted benzenes with **3b**. Average percentage of deuterium incorporation for a)all, b) meta plus para, and c) ortho plus para positions on the ring.

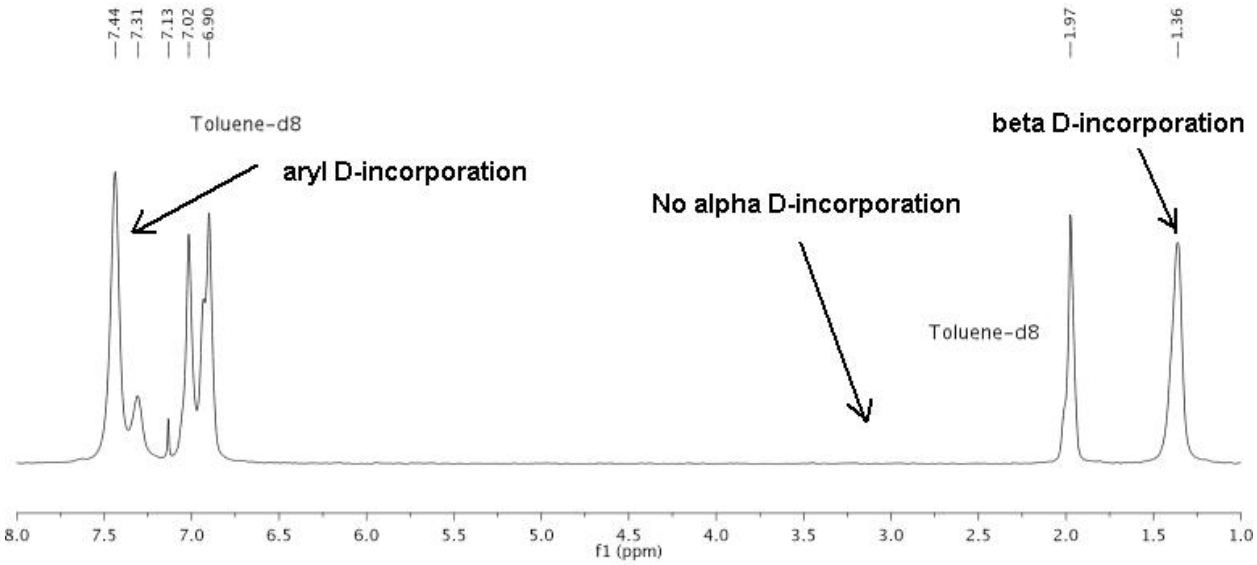


Figure 3S. ^{2}H NMR of cumene after catalytic H/D exchange facilitated by **Na-1**.

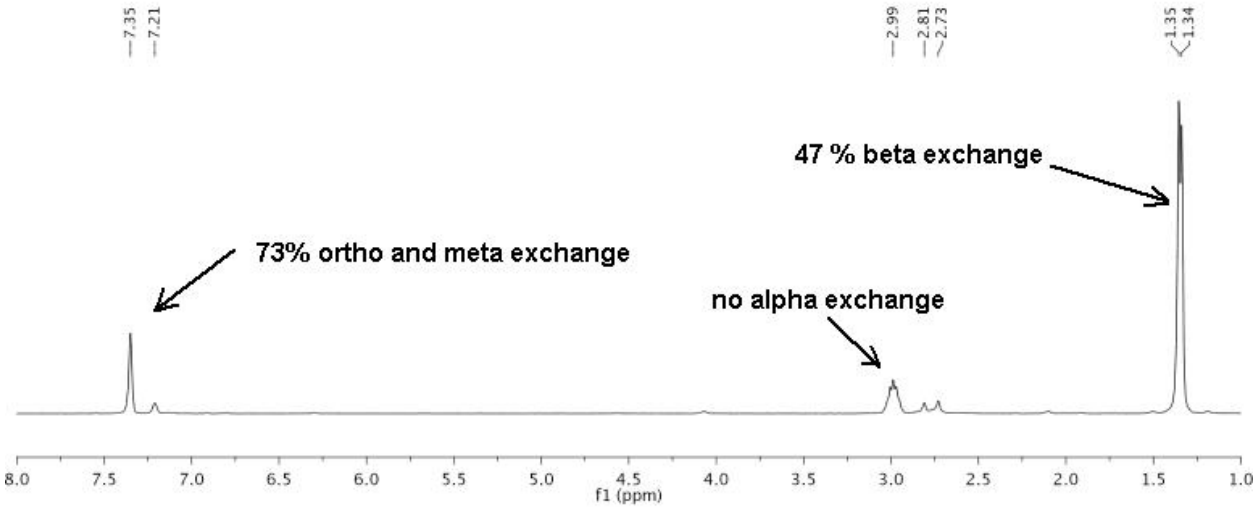


Figure 4S. ^{1}H NMR of cumene after catalytic H/D exchange facilitated by **Na-1**.

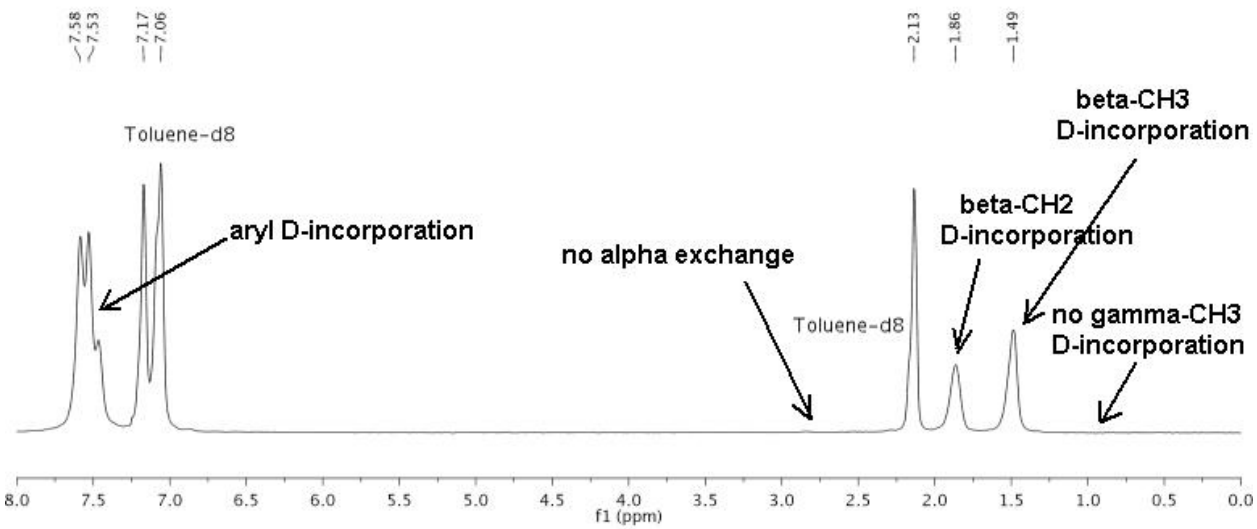


Figure 5S. [2]H NMR of *sec*-butylbenzene after catalytic H/D exchange facilitated by **Na-1**.

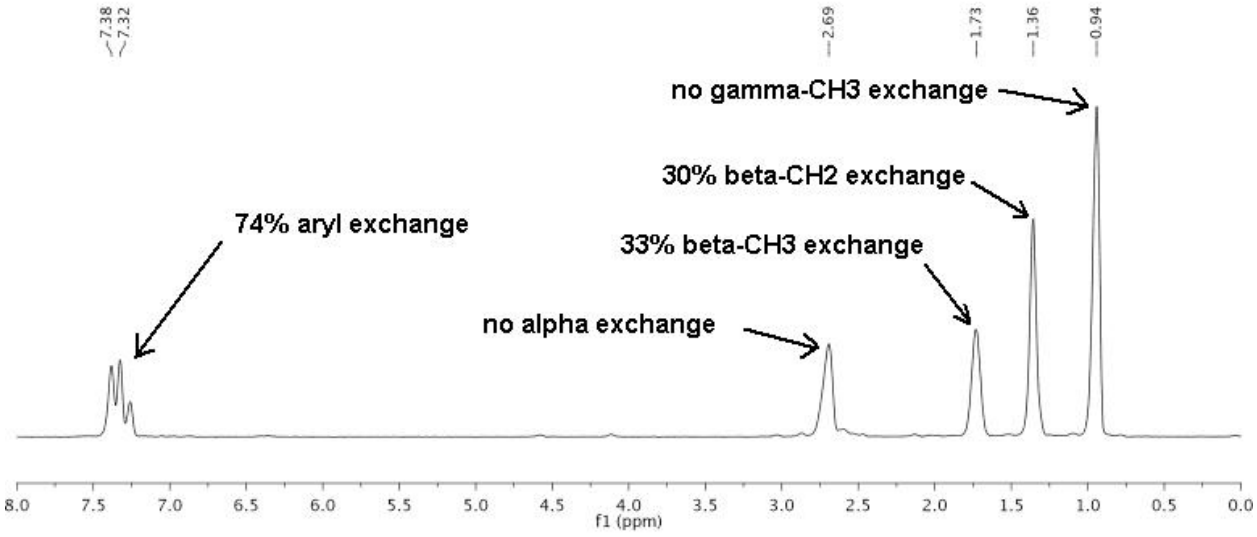
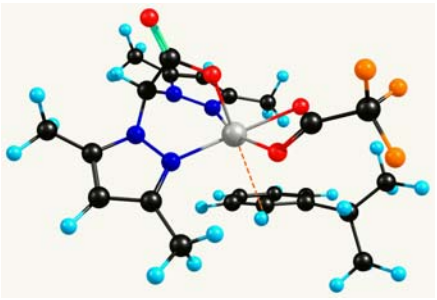


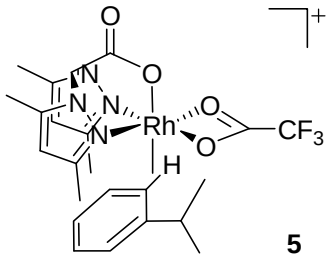
Figure 6S. [1]H NMR of *sec*-butylbenzene after catalytic H/D exchange facilitated by **Na-1**.

Table S-1. DFT calculated free energies (kcal/mol) in the gas phase and acetic acid solvent for **5**, **TS-6**, **7**, **TS-8**, **9**, **TS-10**, and **11**.

compound	G, gas phase	G, HOAc (PCM)	Gas phase	HOAc solvent
5	-1143435.3435	-1143469.93	0.00	0.00
TS-6	-1143403.2840	-1143437.62	32.06	32.31
7	-1143420.3196	-1143454.36	15.02	15.57
5	-1143435.3435	-1143469.93	0.00	0.00
TS-8	-1143409.2541	-1143446.60	26.09	23.32
9	-1143415.0724	-1143451.32	20.27	18.61
5	-1143435.3435	-1143469.93	0.00	0.00
TS-10	-1143391.4900	-1143425.87	43.85	44.06
11	-1143418.6103	-1143453.87	16.73	16.06

Table S-1. DFT Coordinates for cumene adduct **5**.



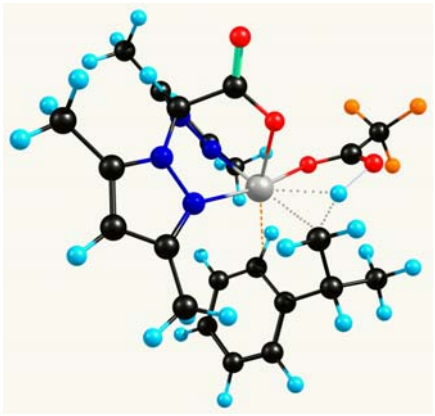


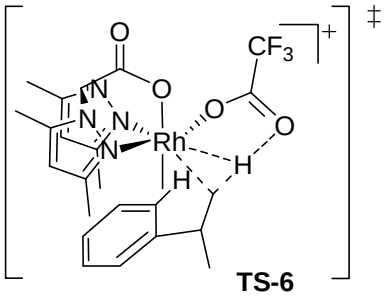
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8	2.454116000	5.425508000	15.175056000
7	5.035003000	4.706269000	15.906292000
7	4.442583000	4.345086000	17.086476000
7	3.095606000	2.757479000	14.950717000
7	2.869522000	2.716247000	16.303369000
6	6.166529000	5.362463000	16.218139000
6	6.292428000	5.403438000	17.620588000
1	7.103946000	5.850602000	18.175021000
6	5.179722000	4.777553000	18.152705000
6	3.044952000	3.952672000	17.044117000
1	2.699716000	3.798080000	18.062620000
6	2.175369000	5.096557000	16.418387000
6	2.085866000	1.639184000	16.624921000
6	1.847852000	0.968438000	15.442351000
1	1.283977000	0.054919000	15.325223000
6	2.468525000	1.704840000	14.409117000
6	7.098016000	5.945999000	15.203974000
1	7.727697000	5.177964000	14.742444000
1	7.761366000	6.661775000	15.692963000
1	6.549921000	6.472482000	14.418591000
6	4.761163000	4.574618000	19.572562000
1	3.846833000	5.131316000	19.806589000
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1	4.589573000	3.517446000	19.800561000
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8	2.788812000	4.806759000	12.471318000
6	2.840417000	6.964037000	11.320384000
9	3.906200000	7.493993000	10.699229000
9	2.154721000	7.939707000	11.922071000
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1	6.208601000	0.476657000	14.060169000
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1	6.446236000	2.854938000	14.724424000
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6	7.009972000	4.523319000	9.691375000
1	7.799471000	4.699620000	10.428538000
1	7.253942000	3.604155000	9.148870000
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1	4.503487000	5.111064000	8.633136000
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Table S-2. DFT Coordinates for transition state **TS-6**.

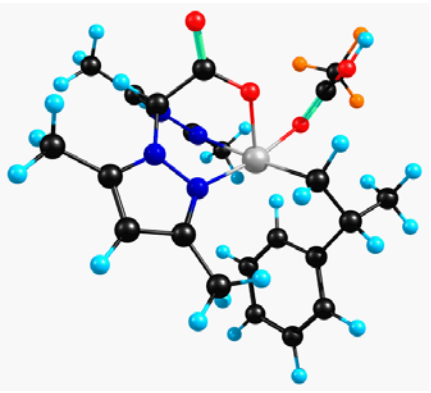
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7	-2.642190000	-3.642833000	-17.147850000
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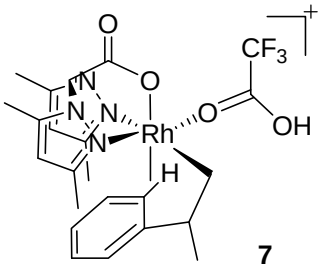




TS-6

Table S-3. DFT Coordinates for β-C–H activation product **7**.



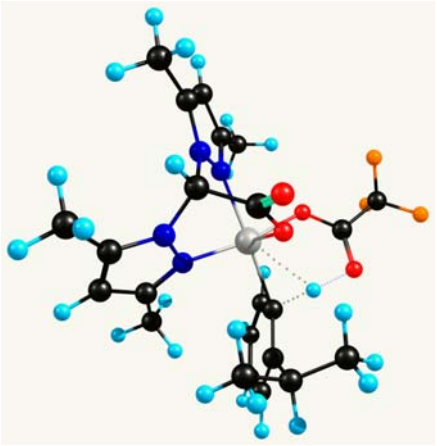


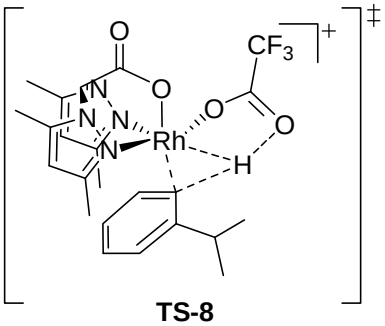
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Table S-4. DFT Coordinates for transition state **TS-8**.

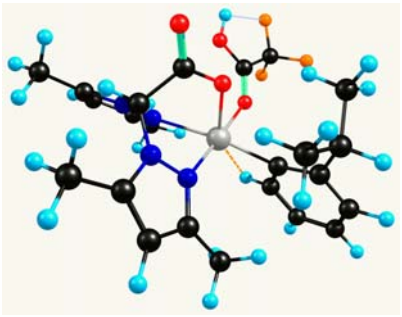
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7	-4.444710000	-5.006481000	-16.901360000
7	-3.539285000	-4.758112000	-17.892568000
7	-2.921598000	-3.055396000	-15.563988000
7	-2.238970000	-3.105371000	-16.753949000
6	-5.565807000	-5.439214000	-17.495156000
6	-5.360169000	-5.466784000	-18.890281000
1	-6.078803000	-5.765422000	-19.638887000
6	-4.063705000	-5.040730000	-19.122369000
6	-2.200556000	-4.355384000	-17.496668000
1	-1.600877000	-4.205157000	-18.390611000
6	-1.517915000	-5.478828000	-16.669770000
6	-1.839561000	-1.857552000	-17.141247000
6	-2.283039000	-0.992246000	-16.159142000
1	-2.134242000	0.076830000	-16.130235000
6	-2.962453000	-1.764862000	-15.196626000
6	-6.790763000	-5.825794000	-16.729296000
1	-6.662187000	-6.808689000	-16.267401000
1	-7.008416000	-5.107963000	-15.933084000
1	-7.652728000	-5.865110000	-17.397739000
6	-3.297970000	-4.891255000	-20.396858000
1	-2.400182000	-5.518706000	-20.406910000
1	-3.926661000	-5.197978000	-21.234058000
1	-2.994281000	-3.853577000	-20.574411000
6	-1.066636000	-1.593530000	-18.392675000
1	-0.133111000	-2.165183000	-18.422035000
1	-1.645421000	-1.834143000	-19.291361000
1	-0.810746000	-0.534071000	-18.441134000
6	-3.639393000	-1.277149000	-13.958317000
1	-3.080373000	-1.558397000	-13.062239000
1	-3.713402000	-0.188522000	-13.987459000
1	-4.648858000	-1.684960000	-13.864750000
8	-4.604042000	-6.658609000	-14.571236000
6	-4.102602000	-7.483359000	-13.743116000
8	-3.212291000	-7.238097000	-12.901160000
6	-4.729581000	-8.895913000	-13.778818000
9	-4.893980000	-9.291702000	-15.048435000
9	-3.970167000	-9.777084000	-13.136511000
9	-5.935772000	-8.834189000	-13.187516000
6	-0.092536000	-3.491928000	-13.584040000
1	0.991019000	-3.624179000	-13.658245000
1	-0.290372000	-2.426267000	-13.428682000
6	-0.636706000	-4.349381000	-12.423623000
1	-0.096370000	-4.026977000	-11.525253000
1	-0.730763000	-6.229576000	-13.556071000
1	0.777345000	-5.964111000	-12.679131000
6	-0.308662000	-5.839830000	-12.627640000
1	-0.674549000	-6.448561000	-11.795944000
6	-2.108797000	-4.074392000	-12.120685000
1	-1.556321000	-2.879053000	-10.419993000
6	-2.388536000	-3.277863000	-10.993774000
6	-3.222906000	-4.617663000	-12.832555000
6	-3.688527000	-2.985519000	-10.583586000
1	-3.846942000	-2.361916000	-9.708469000
6	-4.538991000	-4.330873000	-12.369542000
1	-5.393782000	-4.812693000	-12.852232000
6	-4.785282000	-3.511618000	-11.275454000
1	-5.799335000	-3.319631000	-10.939792000
1	-3.078210000	-5.922337000	-12.935753000
1	-0.530322000	-3.792500000	-14.537598000

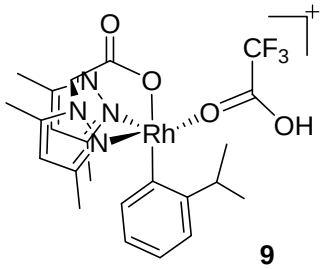




TS-8

Table S-5. DFT Coordinates for arene C–H activation product **9**.





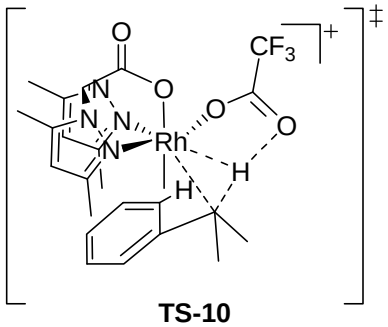
9

45	-3.595766000	-4.718722000	-14.765769000
8	-0.370746000	-5.934397000	-16.878589000
8	-1.856609000	-5.540529000	-15.257079000
7	-4.289232000	-5.168116000	-16.856293000
7	-3.359676000	-4.874446000	-17.809102000
7	-2.985171000	-2.957340000	-15.550214000
7	-2.258777000	-3.046571000	-16.712672000
6	-5.353802000	-5.649075000	-17.506100000
6	-5.088211000	-5.678041000	-18.893914000
1	-5.750626000	-6.019826000	-19.675498000
6	-3.809247000	-5.180400000	-19.064600000
6	-2.084309000	-4.336692000	-17.367396000
1	-1.453537000	-4.185132000	-18.239209000
6	-1.350126000	-5.358615000	-16.452399000
6	-1.905484000	-1.805361000	-17.158523000
6	-2.423813000	-0.906155000	-16.246428000
1	-2.327607000	0.168778000	-16.278329000
6	-3.095598000	-1.649907000	-15.257175000
6	-6.602576000	-6.055057000	-16.788595000
1	-6.473345000	-5.992285000	-15.705580000
1	-7.439952000	-5.408010000	-17.069497000
1	-6.885689000	-7.080524000	-17.045609000
6	-2.992601000	-4.991167000	-20.302139000
1	-2.050381000	-5.547812000	-20.256835000
1	-3.552838000	-5.354023000	-21.165258000
1	-2.757789000	-3.936042000	-20.482241000
6	-1.101329000	-1.573283000	-18.396710000
1	-0.137761000	-2.092352000	-18.359519000
1	-1.631333000	-1.898245000	-19.298830000
1	-0.900948000	-0.505752000	-18.499752000
6	-3.824496000	-1.106990000	-14.070715000
1	-3.311327000	-1.351660000	-13.137287000
1	-3.888440000	-0.020532000	-14.155777000
1	-4.839298000	-1.506629000	-14.004299000
8	-4.195293000	-6.610580000	-13.981916000
6	-3.890354000	-7.758227000	-14.273035000
8	-3.263242000	-8.105334000	-15.368333000
6	-4.288114000	-8.886978000	-13.288343000
9	-3.720048000	-8.675451000	-12.106864000
9	-5.612670000	-8.921511000	-13.156703000
9	-3.862564000	-10.072739000	-13.780545000
6	-0.224648000	-3.183912000	-13.284959000
1	0.864799000	-3.291216000	-13.281605000
1	-0.455675000	-2.117200000	-13.195202000
6	-0.832883000	-3.990357000	-12.123484000
1	-0.380824000	-3.603174000	-11.201856000
1	-0.843069000	-5.928727000	-13.135107000
1	0.626639000	-5.600052000	-12.209848000
6	-0.462036000	-5.482944000	-12.213030000
1	-0.862303000	-6.037962000	-11.358516000
6	-2.336296000	-3.787602000	-11.957149000
1	-2.067826000	-2.847891000	-10.046176000
6	-2.802774000	-3.178445000	-10.776017000
6	-3.330883000	-4.181758000	-12.867620000
6	-4.159601000	-2.978353000	-10.502382000
1	-4.455966000	-2.506768000	-9.570525000
6	-4.690514000	-4.053473000	-12.570280000
1	-5.454114000	-4.474326000	-13.243331000
6	-5.127183000	-3.415660000	-11.402620000
1	-6.186177000	-3.312381000	-11.188801000
1	-3.093573000	-9.065346000	-15.379527000
1	-0.589751000	-3.540830000	-14.249951000

Table S-5. DFT Coordinates for transition state **TS-10**.

45	-3.722277000	-5.341894000	-15.013279000
8	-1.805522000	-5.807199000	-15.389255000
8	-0.169737000	-6.088863000	-16.890464000
7	-4.283095000	-5.997261000	-16.925738000
7	-3.368811000	-5.812031000	-17.921831000
7	-3.343383000	-3.480267000	-16.094547000
7	-2.547270000	-3.692449000	-17.205918000
6	-5.229296000	-6.814060000	-17.417552000
6	-4.903120000	-7.142730000	-18.747232000
1	-5.474206000	-7.776843000	-19.408743000
6	-3.712386000	-6.503395000	-19.046248000
6	-2.181951000	-5.028659000	-17.633737000
1	-1.603164000	-4.941922000	-18.549031000
6	-1.279783000	-5.711900000	-16.583016000
6	-2.317817000	-2.539263000	-17.895334000
6	-2.990676000	-1.549608000	-17.214081000
1	-3.030621000	-0.501152000	-17.468288000
6	-3.628215000	-2.160240000	-16.119907000
6	-6.420159000	-7.260339000	-16.634038000
1	-7.068593000	-6.413194000	-16.392447000
1	-7.000715000	-7.973881000	-17.221269000
1	-6.116797000	-7.746459000	-15.703678000
6	-2.887972000	-6.509253000	-20.292717000
1	-1.879466000	-6.894812000	-20.109230000
1	-3.361386000	-7.153330000	-21.035233000
1	-2.799390000	-5.509023000	-20.731189000
6	-1.478359000	-2.463832000	-19.130035000
1	-0.467986000	-2.850109000	-18.960171000
1	-1.922444000	-3.017103000	-19.965214000
1	-1.389640000	-1.421129000	-19.438688000
6	-4.515246000	-1.424796000	-15.173325000
1	-4.059991000	-1.305877000	-14.188122000
1	-4.698528000	-0.425887000	-15.574381000
1	-5.474367000	-1.925573000	-15.050286000
8	-3.875420000	-7.326925000	-14.494717000
6	-2.958946000	-7.730395000	-13.707799000
8	-2.242582000	-6.979879000	-13.012957000
6	-2.773387000	-9.259864000	-13.634604000
9	-3.929294000	-9.820912000	-13.243205000
9	-2.459869000	-9.718862000	-14.853715000
9	-1.813784000	-9.586944000	-12.776579000
6	-2.526783000	-3.485400000	-12.668567000
1	-1.514110000	-3.884502000	-12.557454000
1	-2.712755000	-2.846557000	-11.796051000
6	-3.525732000	-4.642366000	-12.737759000
1	-4.190539000	-6.252743000	-11.337483000
1	-2.552969000	-5.648367000	-11.014126000
6	-3.553626000	-5.365516000	-11.342822000
1	-3.968965000	-4.649917000	-10.624465000
6	-4.957617000	-4.295788000	-13.053234000
1	-5.564843000	-6.320437000	-13.606370000
1	-4.876156000	-2.292892000	-12.244708000
6	-5.507034000	-3.038873000	-12.714057000
6	-5.840080000	-5.272727000	-13.594043000
6	-6.849952000	-2.763609000	-12.918476000
1	-7.254233000	-1.806836000	-12.600677000
6	-7.191762000	-4.953294000	-13.858413000
1	-7.849381000	-5.722906000	-14.249325000
6	-7.695955000	-3.708390000	-13.528407000
1	-8.743872000	-3.477155000	-13.689815000
1	-2.807675000	-5.825355000	-13.171165000
1	-2.535310000	-2.865948000	-13.565682000

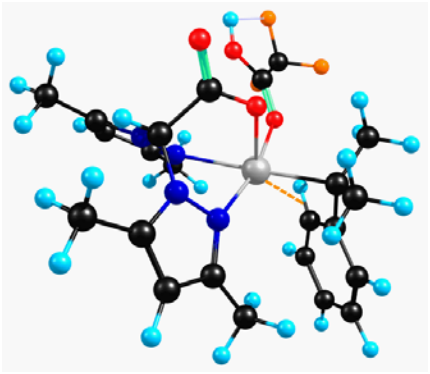


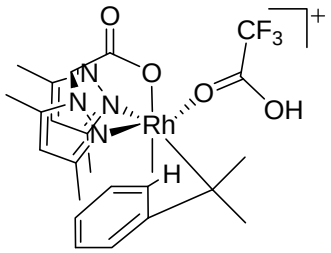


TS-10

Table S-7. DFT Coordinates for α-C–H activation product **11**.

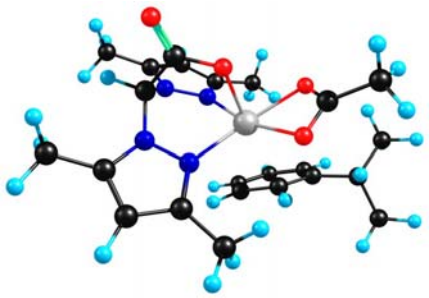
45	-3.766640000	-5.100518000	-14.855865000
8	-0.086312000	-5.957562000	-16.341833000
8	-1.797973000	-5.486954000	-14.974536000
7	-4.105360000	-6.066247000	-16.892653000
7	-3.065656000	-5.838839000	-17.745195000
7	-3.505428000	-3.447812000	-16.049842000
7	-2.546781000	-3.608721000	-17.029937000
6	-4.910381000	-6.938272000	-17.508230000
6	-4.365778000	-7.282771000	-18.765679000
1	-4.786641000	-7.959454000	-19.494850000
6	-3.186536000	-6.569968000	-18.894733000
6	-2.005991000	-4.930094000	-17.322768000
1	-1.308435000	-4.822795000	-18.148927000
6	-1.198884000	-5.514880000	-16.132385000
6	-2.509087000	-2.517749000	-17.854841000
6	-3.460657000	-1.640215000	-17.383493000
1	-3.693868000	-0.664328000	-17.782558000
6	-4.079903000	-2.255543000	-16.276983000
6	-6.176262000	-7.420118000	-16.869922000
1	-6.487302000	-6.743434000	-16.070283000
1	-6.982224000	-7.477516000	-17.606260000
1	-6.055699000	-8.422061000	-16.442254000
6	-2.181661000	-6.539505000	-20.001640000
1	-1.179436000	-6.803094000	-19.647529000
1	-2.466217000	-7.259844000	-20.770301000
1	-2.126031000	-5.554846000	-20.479677000
6	-1.570944000	-2.392280000	-19.011743000
1	-0.530428000	-2.551942000	-18.711274000
1	-1.807871000	-3.099985000	-19.814172000
1	-1.648565000	-1.387092000	-19.429155000
6	-5.220336000	-1.696471000	-15.495201000
1	-4.875819000	-1.135825000	-14.622764000
1	-5.781064000	-1.007194000	-16.131128000
1	-5.897308000	-2.477762000	-15.149320000
8	-3.982203000	-7.067416000	-14.021125000
6	-3.426264000	-8.128562000	-14.263230000
8	-2.465683000	-8.289170000	-15.137853000
6	-3.931410000	-9.393152000	-13.525882000
9	-3.998953000	-9.174499000	-12.218807000
9	-5.143174000	-9.711315000	-13.993797000
9	-3.088591000	-10.420003000	-13.767545000
6	-2.479163000	-3.105132000	-12.963017000
1	-1.495819000	-3.464938000	-13.276168000
1	-2.362297000	-2.675815000	-11.958593000
6	-3.464117000	-4.267717000	-12.940990000
1	-3.688218000	-6.156848000	-11.823243000
1	-2.017087000	-5.757625000	-12.294140000
6	-2.975218000	-5.344116000	-11.966368000
1	-2.814005000	-4.878976000	-10.984800000
6	-4.909025000	-3.909314000	-12.807837000
1	-5.658493000	-5.912685000	-13.254050000
1	-4.609616000	-1.878482000	-12.126902000
6	-5.343854000	-2.621566000	-12.418287000
6	-5.909309000	-4.866164000	-13.134581000
6	-6.693871000	-2.305739000	-12.360063000
1	-7.000917000	-1.324072000	-12.011010000
6	-7.270914000	-4.513956000	-13.138471000
1	-8.014594000	-5.263735000	-13.391618000
6	-7.665981000	-3.240297000	-12.752510000
1	-8.718769000	-2.978355000	-12.718837000
1	-2.130628000	-9.204236000	-15.123908000
1	-2.779345000	-2.302112000	-13.639336000





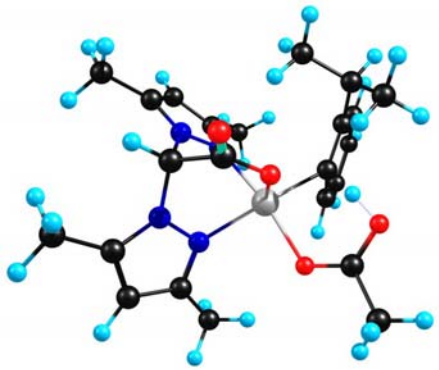
11

Table S-8. DFT Coordinates for acetate adduct **5'**.



45	3.920371000	4.514667000	14.182126000
8	1.318646000	5.654657000	17.067682000
8	2.465175000	5.470696000	15.153396000
7	5.041709000	4.721484000	15.898541000
7	4.436458000	4.357860000	17.070691000
7	3.089169000	2.786519000	14.918523000
7	2.850419000	2.748701000	16.268392000
6	6.180637000	5.354472000	16.225863000
6	6.298536000	5.380581000	17.629926000
1	7.112514000	5.809590000	18.194996000
6	5.172234000	4.766942000	18.147244000
6	3.034874000	3.979954000	17.015025000
1	2.680794000	3.824232000	18.030260000
6	2.180846000	5.136658000	16.392008000
6	2.070398000	1.669377000	16.589713000
6	1.846408000	0.991371000	15.408614000
1	1.286952000	0.075203000	15.291086000
6	2.475324000	1.726040000	14.378724000
6	7.124764000	5.931483000	15.219430000
1	7.748269000	5.158366000	14.757825000
1	7.793372000	6.639096000	15.713319000
1	6.584294000	6.464004000	14.432799000
6	4.739719000	4.555757000	19.561866000
1	3.830756000	5.121747000	19.794512000
1	5.525688000	4.899735000	20.235795000
1	4.552228000	3.498785000	19.778299000
6	1.602285000	1.386751000	17.980320000
1	0.904611000	2.153650000	18.335555000
1	2.436133000	1.325223000	18.687480000
1	1.078195000	0.430008000	17.998485000
6	2.450710000	1.445264000	12.913013000
1	3.011435000	2.188566000	12.348825000
1	1.414651000	1.457385000	12.559341000
1	2.861193000	0.454504000	12.700894000
8	4.272550000	6.305508000	13.195773000
6	3.322485000	6.041492000	12.371437000
8	2.814260000	4.867842000	12.465860000
6	2.814383000	7.051757000	11.400728000
1	3.602111000	7.760261000	11.139166000
1	1.999440000	7.604620000	11.881994000
1	2.417018000	6.561293000	10.510778000
6	5.588783000	1.933071000	11.076408000
6	5.807847000	0.936093000	12.035051000
6	5.659756000	3.286956000	11.411250000
1	5.770308000	-0.107374000	11.734550000
6	6.098446000	1.265377000	13.359025000
6	5.933010000	3.622241000	12.766711000
1	6.291350000	0.488031000	14.091091000
1	6.208214000	4.650627000	12.985019000
6	6.180432000	2.609625000	13.719840000
1	6.467570000	2.877686000	14.730040000
1	5.397654000	1.640844000	10.048961000
6	5.597229000	4.371874000	10.346963000
1	5.430317000	5.324551000	10.863638000
6	4.459977000	4.168770000	9.330870000
6	6.962385000	4.480560000	9.630521000
1	7.775744000	4.670920000	10.337512000
1	7.195013000	3.557101000	9.090297000
1	6.943995000	5.300170000	8.905612000
1	4.622366000	3.279442000	8.713637000
1	4.412363000	5.023839000	8.649666000
1	3.489915000	4.066988000	9.825846000

Table S-9. DFT Coordinates for arene C–H activation transition state **TS-8'**.



45	3.712003723	4.900448723	14.910749446
8	0.539396000	6.074506000	17.098736000
8	2.089030000	5.831906277	15.515440000
7	4.458069277	5.035974000	16.877110277
7	3.558741277	4.784514000	17.871336000
7	2.927678000	3.090981277	15.561311000
7	2.232403000	3.146986000	16.743320000
6	5.604860000	5.395285000	17.467973277
6	5.420565000	5.379427000	18.866430277
1	6.160228000	5.626120000	19.613494277
6	4.112577000	4.994420000	19.103469277
6	2.210183000	4.396892000	17.486792000
1	1.618151000	4.251388000	18.386484000
6	1.529256000	5.526498000	16.668097000
6	1.824936000	1.902745000	17.135059000
6	2.275123000	1.030371000	16.162849000
1	2.122153000	-0.038238000	16.138016000
6	2.967873000	1.796593000	15.203768000
6	6.848280000	5.709412000	16.697062000
1	6.618054277	6.285959000	15.800133000
1	7.364856000	4.789249000	16.399921000
1	7.535984000	6.285581000	17.319351000
6	3.360171000	4.830333000	20.382507000
1	2.491055000	5.495196000	20.430653000
1	4.014586000	5.077082000	21.219866000
1	3.013227000	3.801158000	20.526949000
6	1.038119000	1.649148000	18.380127000
1	0.110694000	2.230969000	18.400646000
1	1.610939000	1.884673000	19.284077000
1	0.770411000	0.592556000	18.428078000
6	3.656446000	1.300505000	13.975559000
1	3.096785277	1.560458277	13.074143000
1	3.749007000	0.213765000	14.021408000
1	4.658377000	1.725213000	13.877128000
8	4.610811723	6.695413831	14.551139723
6	4.089446109	7.563986446	13.764915386
8	3.162339000	7.277566951	12.950324446
6	4.619616169	8.967598723	13.848765446
1	4.091104169	9.486111000	14.657341446
1	4.442277169	9.504703723	12.917056446
1	5.682918169	8.952412446	14.093835446
6	0.089051000	3.507008000	13.570398000
1	-0.994753000	3.639390000	13.643757277
1	0.288193000	2.439695000	13.428249000
6	0.635192000	4.352069000	12.403006277
1	0.100536277	4.017601000	11.504598000
1	0.722213446	6.241099723	13.516129277
1	-0.781902000	5.968081277	12.637003000
6	0.304571000	5.843233000	12.589928000
1	0.672832723	6.444547000	11.754938000
6	2.111923554	4.080744109	12.112778554
1	1.567909000	2.788508831	10.479365554
6	2.397488277	3.226702831	11.027027000
6	3.217351277	4.665926000	12.804101217
6	3.699717000	2.927244000	10.629279000
1	3.866085000	2.257731446	9.790956554
6	4.532367723	4.381329000	12.343479831
1	5.382078831	4.895228783	12.800191663
6	4.787256723	3.508212277	11.287586723
1	5.804849000	3.318428831	10.957938723
1	3.070020494	6.066465330	12.945167734
1	0.526086000	3.819498000	14.520797000

REFERENCE NUMBER: jonjlm12

CRYSTAL STRUCTURE REPORT

$C_{16}\ H_{25}\ Cl_3\ Li\ N_4\ O_4\ Rh$

or

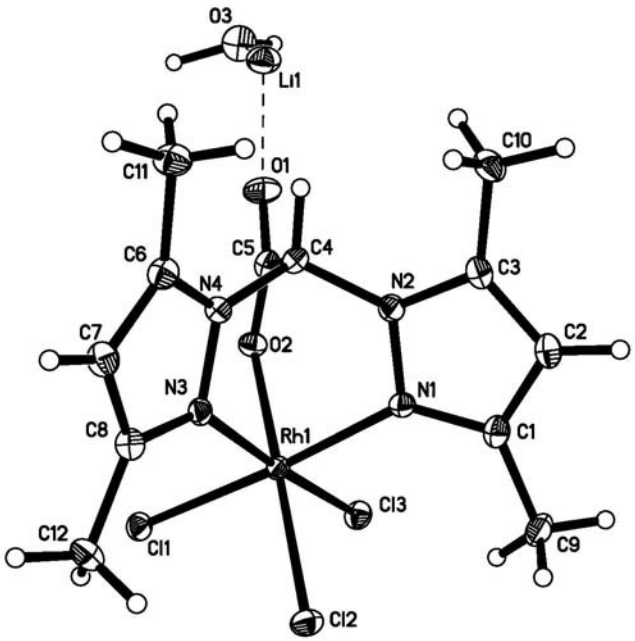
$\{RhCl_3[Li(OH_2)\ bdmpza] \cdot THF\}_n$

Li-1

Report prepared for:

J. Miller, Prof. W. Jones

December 10, 2008



Geoffrey Lippa and William Brennessel

X-ray Crystallographic Facility

Department of Chemistry, University of Rochester

120 Trustee Road

Rochester, NY 14627

Data collection

A crystal (0.22 x 0.08 x 0.06 mm^3) was placed onto the tip of a 0.1 mm diameter glass capillary tube or fiber and mounted on a Bruker SMART APEX II CCD Platform diffractometer for a data collection at 100.0(1) K.[1] A preliminary set of cell constants and an orientation matrix were calculated from reflections harvested from three orthogonal wedges of reciprocal space. The full data collection was carried out using MoKα radiation (graphite monochromator) with a frame time of 45 seconds and a detector distance of 4.01 cm. A randomly oriented region of reciprocal space was surveyed: four major sections of frames were collected with 0.50º steps in ω at four different ϕ settings and a detector position of -38º in 2θ. The intensity data were corrected for absorption.[2] Final cell constants were calculated from the xyz centroids of 7044 strong reflections from the actual data collection after integration.[3] See Table 1 for additional crystal and refinement information.

Structure solution and refinement

The structure was solved using SHELXS-97[4] and refined using SHELXL-97.[4] The space group $P2_1/c$ was determined based on systematic absences and intensity statistics. A direct-methods solution was calculated which provided most non-hydrogen atoms from the E-map. Full-matrix least squares / difference Fourier cycles were performed which located the remaining non-hydrogen atoms. All non-hydrogen atoms were refined with anisotropic displacement parameters. Water hydrogen atoms were found from the difference Fourier map and refined independently from the oxygen atom with individual isotropic displacement parameters. All other hydrogen atoms were placed in ideal positions and refined as riding atoms with relative isotropic displacement parameters. The final full matrix least squares refinement converged to $R1 = 0.0576$ (F^2, $I > 2\sigma(I)$) and $wR2 = 0.0970$ (F^2, all data).

Structure description

The structure is similar to the one suggested. The asymmetric unit consists of one complex rhodium anion, one aqualithium cation, and one co-crystallized THF solvent molecule. The cation and anion form a contact pair, while the THF molecule is connected only by hydrogen bonding (see Table 7 for full details). The structure is polymeric via Li…Cl and Li…O linkages (see diagram).

Unless noted otherwise all structural diagrams containing thermal displacement ellipsoids are drawn at the 50 % probability level.

Data collection, structure solution, and structure refinement were conducted at the X-ray Crystallographic Facility, B51 Hutchison Hall, Department of Chemistry, University of Rochester.

[1] *APEX2*, version 2008.3-0; Bruker AXS: Madison, WI, 2008.

[2] Sheldrick, G. M. *SADABS*, version 2008/1; University of Göttingen: Göttingen, Germany, 2008.

[3] *SAINT*, version 7.53A; Bruker AXS: Madison, WI, 2007.

[4] Sheldrick, G. M. *Acta. Cryst.* **2008**, *A64*, 112-122.

Some equations of interest:

$$R_{int} = \Sigma |F_o^2 - < F_o^2 >| / \Sigma |F_o^2|$$

$$R1 = \Sigma ||F_o| - |F_c|| / \Sigma |F_o|$$

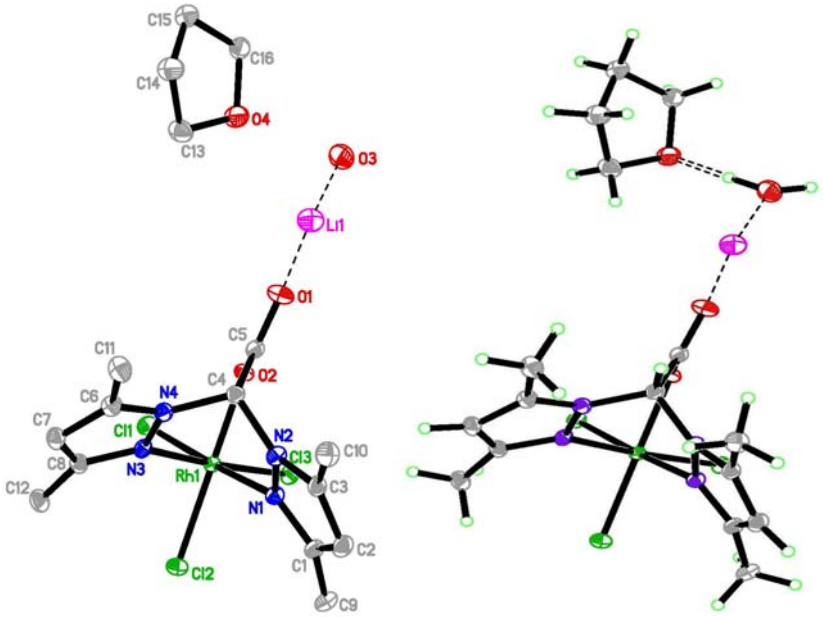
$$wR2 = [\Sigma [w(F_o^2 - F_c^2)^2] / \Sigma [w(F_o^2)^2]]^{1/2}$$

where $w = 1 / [\sigma^2 (F_o^2) + (aP)^2 + bP]$ and

$$P = 1/3 \max (0, F_o^2) + 2/3 F_c^2$$

$$GOF = S = [\Sigma [w(F_o^2 - F_c^2)^2] / (m-n)]^{1/2}$$

where m = number of reflections and n = number of parameters



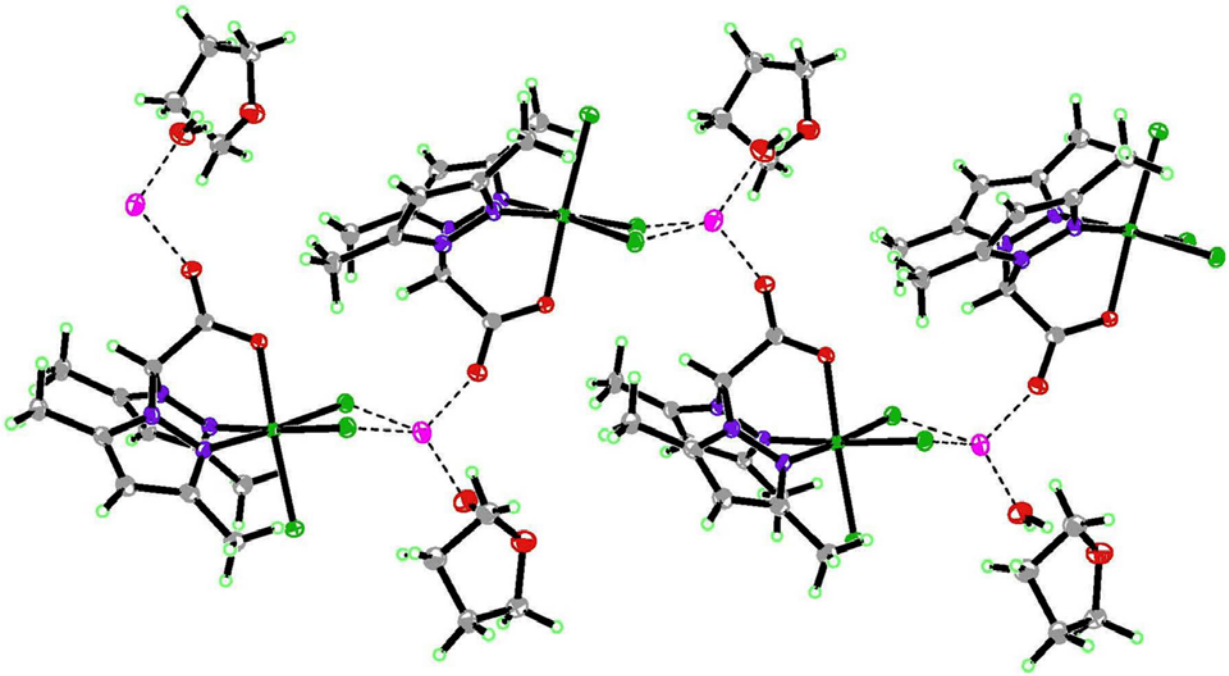


Table 1. Crystal data and structure refinement for **Li-1**.

Identification code	jonjlm12	
Empirical formula	C16 H25 Cl3 Li N4 O4 Rh	
Formula weight	553.60	
Temperature	100.0(1) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 8.3096(9) Å	α = 90°
	b = 12.7502(14) Å	β = 99.447(2)°
	c = 20.107(2) Å	γ = 90°
Volume	2101.5(4) Å^3	
Z	4	
Density (calculated)	1.750 Mg/m^3	
Absorption coefficient	1.225 mm^{-1}	
F(000)	1120	
Crystal color, morphology	yellow-orange, rectangular prism	
Crystal size	0.22 x 0.08 x 0.06 mm^3	
Theta range for data collection	1.90 to 37.03°	
Index ranges	$-14 \le h \le 13$, $-21 \le k \le 21$, $-34 \le l \le 34$	
Reflections collected	48567	
Independent reflections	10605 [R(int) = 0.1005]	
Observed reflections	7044	
Completeness to theta = 37.03°	98.9%	
Absorption correction	Multi-scan	
Max. and min. transmission	0.9301 and 0.7744	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	10605 / 0 / 266	
Goodness-of-fit on F^2	1.034	
Final R indices [I>2sigma(I)]	R1 = 0.0576, wR2 = 0.0856	
R indices (all data)	R1 = 0.1041, wR2 = 0.0970	
Largest diff. peak and hole	1.378 and -1.478 e.Å^{-3}	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **Li-1**. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Rh1	842(1)	7250(1)	1354(1)	11(1)
Cl1	-1308(1)	8438(1)	1104(1)	15(1)
Cl2	1681(1)	7649(1)	343(1)	17(1)
Cl3	2469(1)	8607(1)	1882(1)	16(1)
O1	-197(3)	5818(2)	3038(1)	20(1)
O2	92(2)	6980(1)	2251(1)	14(1)
N1	2643(3)	6166(2)	1618(1)	13(1)
N2	2167(3)	5252(2)	1894(1)	14(1)
N3	-597(3)	6030(2)	961(1)	12(1)
N4	-589(3)	5173(2)	1373(1)	13(1)
C1	4194(3)	6022(2)	1543(1)	16(1)
C2	4688(3)	5012(2)	1768(1)	18(1)
C3	3390(3)	4533(2)	1977(1)	15(1)
C4	507(3)	5158(2)	2012(1)	13(1)
C5	94(3)	6053(2)	2478(1)	14(1)
C6	-1504(3)	4382(2)	1055(1)	15(1)
C7	-2157(3)	4766(2)	434(1)	16(1)
C8	-1574(3)	5785(2)	388(1)	14(1)
C9	5194(3)	6827(2)	1264(2)	19(1)
C10	3187(3)	3457(2)	2234(2)	19(1)
C11	-1634(4)	3333(2)	1364(1)	19(1)
C12	-1931(4)	6513(2)	-197(1)	19(1)
Li1	-560(6)	4861(4)	3710(3)	21(1)
O3	-1059(3)	5664(2)	4439(1)	25(1)
O4	-4015(3)	6683(2)	4028(1)	25(1)
C13	-4728(4)	6058(2)	3462(2)	24(1)
C14	-5601(4)	5190(3)	3767(2)	25(1)
C15	-6315(4)	5776(2)	4308(2)	21(1)
C16	-5011(3)	6594(2)	4547(2)	20(1)

Table 3. Bond lengths [Å] and angles [°] for **Li-1**.

Rh(1)-O(2)	2.0322(18)	C(9)-H(9C)	0.9800
Rh(1)-N(3)	2.041(2)	C(10)-H(10A)	0.9800
Rh(1)-N(1)	2.043(2)	C(10)-H(10B)	0.9800
Rh(1)-Cl(2)	2.3108(7)	C(10)-H(10C)	0.9800
Rh(1)-Cl(1)	2.3326(7)	C(11)-H(11A)	0.9800
Rh(1)-Cl(3)	2.3395(7)	C(11)-H(11B)	0.9800
Rh(1)-Li(1)#1	3.337(5)	C(11)-H(11C)	0.9800
Cl(1)-Li(1)#1	2.377(5)	C(12)-H(12A)	0.9800
Cl(3)-Li(1)#1	2.423(5)	C(12)-H(12B)	0.9800
O(1)-C(5)	1.227(3)	C(12)-H(12C)	0.9800
O(1)-Li(1)	1.882(5)	Li(1)-O(3)	1.888(6)
O(2)-C(5)	1.267(3)	Li(1)-Cl(1)#2	2.377(5)
N(1)-C(1)	1.334(3)	Li(1)-Cl(3)#2	2.423(5)
N(1)-N(2)	1.376(3)	Li(1)-Rh(1)#2	3.337(5)
N(2)-C(3)	1.359(3)	O(3)-H(3B)	0.9069
N(2)-C(4)	1.442(3)	O(3)-H(3A)	1.0400
N(3)-C(8)	1.333(3)	O(4)-C(13)	1.436(4)
N(3)-N(4)	1.371(3)	O(4)-C(16)	1.439(4)
N(4)-C(6)	1.357(3)	C(13)-C(14)	1.508(4)
N(4)-C(4)	1.448(3)	C(13)-H(13A)	0.9900
C(1)-C(2)	1.403(4)	C(13)-H(13B)	0.9900
C(1)-C(9)	1.488(4)	C(14)-C(15)	1.518(4)
C(2)-C(3)	1.364(4)	C(14)-H(14A)	0.9900
C(2)-H(2)	0.9500	C(14)-H(14B)	0.9900
C(3)-C(10)	1.486(4)	C(15)-C(16)	1.524(4)
C(4)-C(5)	1.551(4)	C(15)-H(15A)	0.9900
C(4)-H(4)	1.0000	C(15)-H(15B)	0.9900
C(6)-C(7)	1.368(4)	C(16)-H(16A)	0.9900
C(6)-C(11)	1.486(4)	C(16)-H(16B)	0.9900
C(7)-C(8)	1.395(4)	O(2)-Rh(1)-N(3)	88.24(8)
C(7)-H(7)	0.9500	O(2)-Rh(1)-N(1)	88.48(8)
C(8)-C(12)	1.490(4)	N(3)-Rh(1)-N(1)	86.70(9)
C(9)-H(9A)	0.9800	O(2)-Rh(1)-Cl(2)	177.07(5)
C(9)-H(9B)	0.9800	N(3)-Rh(1)-Cl(2)	93.95(7)

N(1)-Rh(1)-Cl(2)	93.60(6)	C(3)-C(2)-H(2)	126.1
O(2)-Rh(1)-Cl(1)	88.05(5)	C(1)-C(2)-H(2)	126.1
N(3)-Rh(1)-Cl(1)	91.81(6)	N(2)-C(3)-C(2)	105.9(2)
N(1)-Rh(1)-Cl(1)	176.26(6)	N(2)-C(3)-C(10)	122.7(2)
Cl(2)-Rh(1)-Cl(1)	89.92(2)	C(2)-C(3)-C(10)	131.4(2)
O(2)-Rh(1)-Cl(3)	87.79(5)	N(2)-C(4)-N(4)	109.5(2)
N(3)-Rh(1)-Cl(3)	175.91(7)	N(2)-C(4)-C(5)	110.5(2)
N(1)-Rh(1)-Cl(3)	92.21(6)	N(4)-C(4)-C(5)	110.9(2)
Cl(2)-Rh(1)-Cl(3)	90.06(2)	N(2)-C(4)-H(4)	108.6
Cl(1)-Rh(1)-Cl(3)	89.04(2)	N(4)-C(4)-H(4)	108.6
O(2)-Rh(1)-Li(1)#1	100.00(11)	C(5)-C(4)-H(4)	108.6
N(3)-Rh(1)-Li(1)#1	135.40(11)	O(1)-C(5)-O(2)	124.7(3)
N(1)-Rh(1)-Li(1)#1	136.79(11)	O(1)-C(5)-C(4)	118.2(2)
Cl(2)-Rh(1)-Li(1)#1	77.07(9)	O(2)-C(5)-C(4)	117.1(2)
Cl(1)-Rh(1)-Li(1)#1	45.42(9)	N(4)-C(6)-C(7)	105.8(2)
Cl(3)-Rh(1)-Li(1)#1	46.53(9)	N(4)-C(6)-C(11)	123.0(2)
Rh(1)-Cl(1)-Li(1)#1	90.24(13)	C(7)-C(6)-C(11)	131.1(2)
Rh(1)-Cl(3)-Li(1)#1	88.97(12)	C(6)-C(7)-C(8)	107.6(2)
C(5)-O(1)-Li(1)	153.6(3)	C(6)-C(7)-H(7)	126.2
C(5)-O(2)-Rh(1)	119.69(17)	C(8)-C(7)-H(7)	126.2
C(1)-N(1)-N(2)	106.0(2)	N(3)-C(8)-C(7)	109.3(2)
C(1)-N(1)-Rh(1)	138.12(19)	N(3)-C(8)-C(12)	123.4(2)
N(2)-N(1)-Rh(1)	115.50(16)	C(7)-C(8)-C(12)	127.3(2)
C(3)-N(2)-N(1)	111.2(2)	C(1)-C(9)-H(9A)	109.5
C(3)-N(2)-C(4)	129.8(2)	C(1)-C(9)-H(9B)	109.5
N(1)-N(2)-C(4)	118.9(2)	H(9A)-C(9)-H(9B)	109.5
C(8)-N(3)-N(4)	106.0(2)	C(1)-C(9)-H(9C)	109.5
C(8)-N(3)-Rh(1)	138.52(18)	H(9A)-C(9)-H(9C)	109.5
N(4)-N(3)-Rh(1)	115.42(16)	H(9B)-C(9)-H(9C)	109.5
C(6)-N(4)-N(3)	111.2(2)	C(3)-C(10)-H(10A)	109.5
C(6)-N(4)-C(4)	129.0(2)	C(3)-C(10)-H(10B)	109.5
N(3)-N(4)-C(4)	119.1(2)	H(10A)-C(10)-H(10B)	109.5
N(1)-C(1)-C(2)	109.1(2)	C(3)-C(10)-H(10C)	109.5
N(1)-C(1)-C(9)	123.8(2)	H(10A)-C(10)-H(10C)	109.5
C(2)-C(1)-C(9)	127.0(2)	H(10B)-C(10)-H(10C)	109.5
C(3)-C(2)-C(1)	107.8(2)	C(6)-C(11)-H(11A)	109.5

C(6)-C(11)-H(11B)	109.5	O(4)-C(13)-C(14)	104.3(2)
H(11A)-C(11)-H(11B)	109.5	O(4)-C(13)-H(13A)	110.9
C(6)-C(11)-H(11C)	109.5	C(14)-C(13)-H(13A)	110.9
H(11A)-C(11)-H(11C)	109.5	O(4)-C(13)-H(13B)	110.9
H(11B)-C(11)-H(11C)	109.5	C(14)-C(13)-H(13B)	110.9
C(8)-C(12)-H(12A)	109.5	H(13A)-C(13)-H(13B)	108.9
C(8)-C(12)-H(12B)	109.5	C(13)-C(14)-C(15)	101.6(2)
H(12A)-C(12)-H(12B)	109.5	C(13)-C(14)-H(14A)	111.4
C(8)-C(12)-H(12C)	109.5	C(15)-C(14)-H(14A)	111.4
H(12A)-C(12)-H(12C)	109.5	C(13)-C(14)-H(14B)	111.4
H(12B)-C(12)-H(12C)	109.5	C(15)-C(14)-H(14B)	111.4
O(1)-Li(1)-O(3)	106.6(3)	H(14A)-C(14)-H(14B)	109.3
O(1)-Li(1)-Cl(1)#2	115.5(3)	C(14)-C(15)-C(16)	102.8(2)
O(3)-Li(1)-Cl(1)#2	120.9(3)	C(14)-C(15)-H(15A)	111.2
O(1)-Li(1)-Cl(3)#2	104.2(2)	C(16)-C(15)-H(15A)	111.2
O(3)-Li(1)-Cl(3)#2	122.0(3)	C(14)-C(15)-H(15B)	111.2
Cl(1)#2-Li(1)-Cl(3)#2	86.07(17)	C(16)-C(15)-H(15B)	111.2
O(1)-Li(1)-Rh(1)#2	129.6(2)	H(15A)-C(15)-H(15B)	109.1
O(3)-Li(1)-Rh(1)#2	123.2(2)	O(4)-C(16)-C(15)	106.8(2)
Cl(1)#2-Li(1)-Rh(1)#2	44.34(9)	O(4)-C(16)-H(16A)	110.4
Cl(3)#2-Li(1)-Rh(1)#2	44.50(8)	C(15)-C(16)-H(16A)	110.4
Li(1)-O(3)-H(3B)	132.5	O(4)-C(16)-H(16B)	110.4
Li(1)-O(3)-H(3A)	105.9	C(15)-C(16)-H(16B)	110.4
H(3B)-O(3)-H(3A)	116.8	H(16A)-C(16)-H(16B)	108.6
C(13)-O(4)-C(16)	108.6(2)		

Symmetry transformations used to generate equivalent atoms:

#1 -x,y+1/2,-z+1/2 #2 -x,y-1/2,-z+1/2

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **Li-1**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U_{11} + ... + 2\ h\ k\ a^*\ b^*\ U_{12}\ ]$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Rh1	10(1)	10(1)	12(1)	0(1)	3(1)	0(1)
Cl1	13(1)	13(1)	20(1)	1(1)	3(1)	2(1)
Cl2	19(1)	17(1)	16(1)	2(1)	7(1)	0(1)
Cl3	15(1)	14(1)	19(1)	-1(1)	2(1)	-2(1)
O1	30(1)	18(1)	16(1)	1(1)	10(1)	-1(1)
O2	17(1)	12(1)	12(1)	0(1)	4(1)	1(1)
N1	13(1)	12(1)	14(1)	2(1)	3(1)	0(1)
N2	13(1)	11(1)	17(1)	1(1)	3(1)	1(1)
N3	10(1)	11(1)	16(1)	1(1)	2(1)	1(1)
N4	12(1)	13(1)	14(1)	2(1)	2(1)	1(1)
C1	13(1)	18(1)	17(1)	-2(1)	3(1)	1(1)
C2	14(1)	18(1)	20(1)	0(1)	3(1)	4(1)
C3	14(1)	16(1)	14(1)	-1(1)	1(1)	3(1)
C4	12(1)	13(1)	14(1)	0(1)	3(1)	-2(1)
C5	12(1)	16(1)	15(1)	-2(1)	3(1)	0(1)
C6	16(1)	13(1)	17(1)	-2(1)	3(1)	-1(1)
C7	17(1)	16(1)	15(1)	-3(1)	0(1)	-1(1)
C8	14(1)	14(1)	15(1)	-2(1)	2(1)	0(1)
C9	13(1)	20(1)	25(1)	1(1)	7(1)	-1(1)
C10	20(1)	15(1)	21(1)	3(1)	2(1)	4(1)
C11	24(1)	13(1)	19(1)	-1(1)	1(1)	-5(1)
C12	20(1)	21(1)	15(1)	4(1)	-1(1)	3(1)
Li1	25(3)	16(2)	24(3)	3(2)	7(2)	1(2)
O3	28(1)	27(1)	21(1)	-5(1)	4(1)	0(1)
O4	26(1)	28(1)	23(1)	-8(1)	8(1)	-10(1)
C13	29(2)	22(1)	20(1)	-3(1)	6(1)	-5(1)
C14	29(2)	23(1)	25(2)	-6(1)	8(1)	-7(1)
C15	22(1)	17(1)	23(1)	-2(1)	5(1)	-2(1)
C16	18(1)	22(1)	19(1)	-4(1)	3(1)	1(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for **Li-1**.

	x	y	z	U(eq)
H2	5739	4716	1775	21
H4	385	4471	2238	15
H7	-2877	4403	95	19
H9A	4704	7520	1297	29
H9B	5233	6667	790	29
H9C	6303	6824	1521	29
H10A	2642	3493	2630	28
H10B	4260	3129	2359	28
H10C	2524	3039	1882	28
H11A	-1865	3419	1824	29
H11B	-604	2953	1378	29
H11C	-2520	2936	1095	29
H12A	-2021	7231	-34	29
H12B	-2960	6310	-479	29
H12C	-1045	6477	-464	29
H3B	-424	5976	4794	30
H3A	-2189	6004	4270	30
H13A	-3876	5770	3222	28
H13B	-5504	6477	3141	28
H14A	-4833	4636	3965	30
H14B	-6466	4872	3431	30
H15A	-6484	5302	4681	25
H15B	-7366	6111	4120	25
H16A	-5523	7277	4618	24
H16B	-4341	6371	4977	24

Table 6. Torsion angles [°] for **Li-1**.

O2-Rh1-Cl1-Li1#1	106.11(14)	Cl2-Rh1-N3-C8	-36.5(3)
N3-Rh1-Cl1-Li1#1	-165.72(14)	Cl1-Rh1-N3-C8	53.5(3)
N1-Rh1-Cl1-Li1#1	127.8(10)	Cl3-Rh1-N3-C8	155.4(7)
Cl2-Rh1-Cl1-Li1#1	-71.77(13)	Li1#1-Rh1-N3-C8	39.0(3)
Cl3-Rh1-Cl1-Li1#1	18.29(13)	O2-Rh1-N3-N4	-40.49(17)
O2-Rh1-Cl3-Li1#1	-106.03(14)	N1-Rh1-N3-N4	48.08(17)
N3-Rh1-Cl3-Li1#1	-119.9(9)	Cl2-Rh1-N3-N4	141.47(16)
N1-Rh1-Cl3-Li1#1	165.59(14)	Cl1-Rh1-N3-N4	-128.49(17)
Cl2-Rh1-Cl3-Li1#1	71.98(13)	Cl3-Rh1-N3-N4	-26.6(10)
Cl1-Rh1-Cl3-Li1#1	-17.94(13)	Li1#1-Rh1-N3-N4	-142.98(19)
N3-Rh1-O2-C5	47.15(19)	C8-N3-N4-C6	2.2(3)
N1-Rh1-O2-C5	-39.6(2)	Rh1-N3-N4-C6	-176.47(17)
Cl2-Rh1-O2-C5	-174.7(10)	C8-N3-N4-C4	173.5(2)
Cl1-Rh1-O2-C5	139.02(19)	Rh1-N3-N4-C4	-5.1(3)
Cl3-Rh1-O2-C5	-131.87(19)	N2-N1-C1-C2	0.5(3)
Li1#1-Rh1-O2-C5	-177.0(2)	Rh1-N1-C1-C2	-171.6(2)
O2-Rh1-N1-C1	-143.7(3)	N2-N1-C1-C9	-179.4(3)
N3-Rh1-N1-C1	127.9(3)	Rh1-N1-C1-C9	8.5(5)
Cl2-Rh1-N1-C1	34.2(3)	N1-C1-C2-C3	0.7(3)
Cl1-Rh1-N1-C1	-165.4(8)	C9-C1-C2-C3	-179.4(3)
Cl3-Rh1-N1-C1	-56.0(3)	N1-N2-C3-C2	1.9(3)
Li1#1-Rh1-N1-C1	-40.7(3)	C4-N2-C3-C2	177.6(3)
O2-Rh1-N1-N2	44.73(18)	N1-N2-C3-C10	-176.9(2)
N3-Rh1-N1-N2	-43.59(18)	C4-N2-C3-C10	-1.2(4)
Cl2-Rh1-N1-N2	-137.35(17)	C1-C2-C3-N2	-1.6(3)
Cl1-Rh1-N1-N2	23.0(11)	C1-C2-C3-C10	177.1(3)
Cl3-Rh1-N1-N2	132.46(17)	C3-N2-C4-N4	-110.5(3)
Li1#1-Rh1-N1-N2	147.76(19)	N1-N2-C4-N4	64.9(3)
C1-N1-N2-C3	-1.5(3)	C3-N2-C4-C5	127.1(3)
Rh1-N1-N2-C3	172.59(17)	N1-N2-C4-C5	-57.5(3)
C1-N1-N2-C4	-177.8(2)	C6-N4-C4-N2	110.1(3)
Rh1-N1-N2-C4	-3.6(3)	N3-N4-C4-N2	-59.5(3)
O2-Rh1-N3-C8	141.5(3)	C6-N4-C4-C5	-127.7(3)
N1-Rh1-N3-C8	-129.9(3)	N3-N4-C4-C5	62.7(3)

Li1-O1-C5-O2	174.2(5)
Li1-O1-C5-C4	-6.3(7)
Rh1-O2-C5-O1	173.3(2)
Rh1-O2-C5-C4	-6.2(3)
N2-C4-C5-O1	-114.5(3)
N4-C4-C5-O1	123.9(3)
N2-C4-C5-O2	65.0(3)
N4-C4-C5-O2	-56.6(3)
N3-N4-C6-C7	-2.3(3)
C4-N4-C6-C7	-172.6(2)
N3-N4-C6-C11	176.1(2)
C4-N4-C6-C11	5.8(4)
N4-C6-C7-C8	1.5(3)
C11-C6-C7-C8	-176.7(3)
N4-N3-C8-C7	-1.2(3)
Rh1-N3-C8-C7	177.0(2)
N4-N3-C8-C12	179.5(2)
Rh1-N3-C8-C12	-2.4(4)
C6-C7-C8-N3	-0.2(3)
C6-C7-C8-C12	179.1(3)
C5-O1-Li1-O3	-176.5(5)
C5-O1-Li1-Cl1#2	46.1(7)
C5-O1-Li1-Cl3#2	-46.3(7)
C5-O1-Li1-Rh1#2	-4.5(8)
C16-O4-C13-C14	-28.8(3)
O4-C13-C14-C15	39.5(3)
C13-C14-C15-C16	-34.9(3)
C13-O4-C16-C15	6.0(3)
C14-C15-C16-O4	18.8(3)

Symmetry transformations used to generate equivalent atoms:

#1 -x,y+1/2,-z+1/2 #2 -x,y-1/2,-z+1/2

Table 7. Hydrogen bonds and close contacts for **Li-1** [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O3-H3B...Cl1#3	0.91	2.95	3.577(2)	128.1
O3-H3B...Cl2#3	0.91	2.59	3.429(2)	153.8
O3-H3A...O4	1.04	1.75	2.782(3)	174.5

Symmetry transformations used to generate equivalent atoms:

#1 -x,y+1/2,-z+1/2 #2 -x,y-1/2,-z+1/2 #3 x,-y+3/2,z+1/2

REFERENCE NUMBER: jonjlm15

CRYSTAL STRUCTURE REPORT

$C_{20}\ H_{35}\ Cl_3\ N_5\ O_2\ Rh$

or

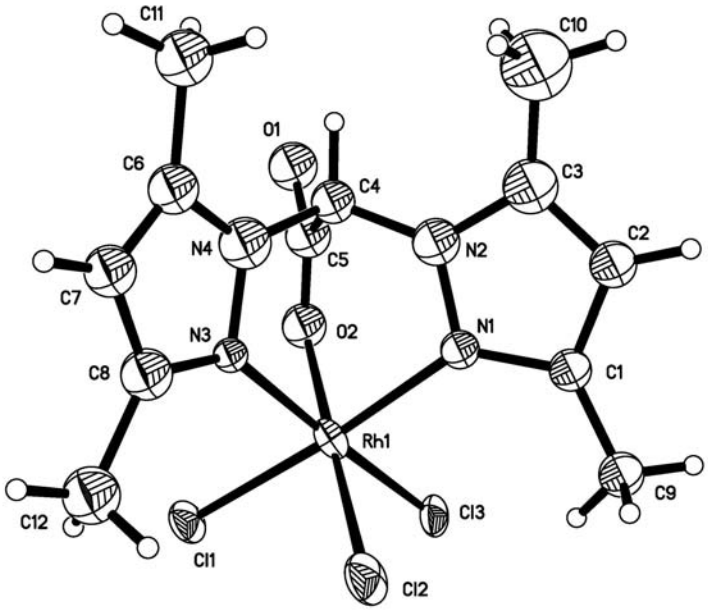
$[NEt_4][RhCl_3(bdmpza)]$

Et_4N-1

Report prepared for:

J. Miller, Prof. W. Jones

January 15, 2008



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Data collection

A crystal (0.16 x 0.16 x 0.03 mm^3) was placed onto the tip of a 0.1 mm diameter glass capillary tube or fiber and mounted on a Bruker SMART APEX II CCD Platform diffractometer for a data collection at 100.0(1) K.[1] A preliminary set of cell constants and an orientation matrix were calculated from reflections harvested from three orthogonal wedges of reciprocal space. The full data collection was carried out using MoKα radiation (graphite monochromator) with a frame time of 120 seconds and a detector distance of 4.02 cm. A randomly oriented region of reciprocal space was surveyed: three major sections of frames were collected with 1.0º steps in ω at three different ϕ settings and a detector position of -38º in 2θ. The intensity data were corrected for absorption.[2] Final cell constants were calculated from the xyz centroids of 1243 strong reflections from the actual data collection after integration.[3] See Table 1 for additional crystal and refinement information.

Structure solution and refinement

The structure was solved using SIR97[4] and refined using SHELXL-97.[5] The space group $P2_1/c$ was determined based on systematic absences and intensity statistics. A direct-methods solution was calculated which provided most non-hydrogen atoms from the E-map. Full-matrix least squares / difference Fourier cycles were performed which located the remaining non-hydrogen atoms. The Rh and Cl atoms were refined with anisotropic displacement parameters; all other non-hydrogen atoms were refined with isotropic displacement parameters. All hydrogen atoms were placed in ideal positions and refined as riding atoms with relative isotropic displacement parameters. The final full matrix least squares refinement converged to $R1 = 0.1326$ (F^2, $I > 2\sigma(I)$) and $wR2 = 0.3113$ (F^2, all data). *The intensity data were of poor quality and very weak. This experiment is intended solely for proof of solid-state structure. Bond lengths and angles are for advice only.*

Structure description

The structure is the one suggested with all atoms in general positions. The NEt_4^+ cation was modeled as disordered over two positions (50:50).

Unless noted otherwise all structural diagrams containing thermal displacement ellipsoids are drawn at the 50 % probability level.

Data collection, structure solution, and structure refinement were conducted at the X-ray Crystallographic Facility, B51 Hutchison Hall, Department of Chemistry, University of Rochester.

[1] *APEX2*, version 2008.3-0; Bruker AXS: Madison, WI, 2008.

[2] Sheldrick, G. M. *SADABS*, version 2008/1; University of Göttingen: Göttingen, Germany, 2008.

[3] *SAINT*, version 7.53A; Bruker AXS: Madison, WI, 2007.

[4] Altomare, A.; Burla, M. C.; Camalli, M.; Cascarano, G. L.; Giacovazzo, C.; Guagliardi, A.; Moliterni, A. G. G.; Polidori, G.; Spagna, R. *SIR97: A new program for solving and refining crystal structures*; Istituto di Cristallografia, CNR: Bari, Italy, 1999.

[5] Sheldrick, G. M. *Acta. Cryst.* **2008**, *A64*, 112-122.

Some equations of interest:

$$R_{int} = \Sigma\, |F_o^2 - < F_o^2 >| \,/\, \Sigma |F_o^2|$$

$$R1 = \Sigma ||F_o| - |F_c|| \,/\, \Sigma |F_o|$$

$$wR2 = [\Sigma [w(F_o^2 - F_c^2)^2] \,/\, \Sigma\, [w(F_o^2)^2]]^{1/2}$$

where $w = 1 \,/\, [\sigma^2 (F_o^2) + (aP)^2 + bP]$ and

$$P = 1/3 \max (0, F_o^2) + 2/3\, F_c^2$$

$$\text{GOF} = S = [\Sigma [w(F_o^2 - F_c^2)^2] \,/\, (m-n)]^{1/2}$$

where m = number of reflections and n = number of parameters

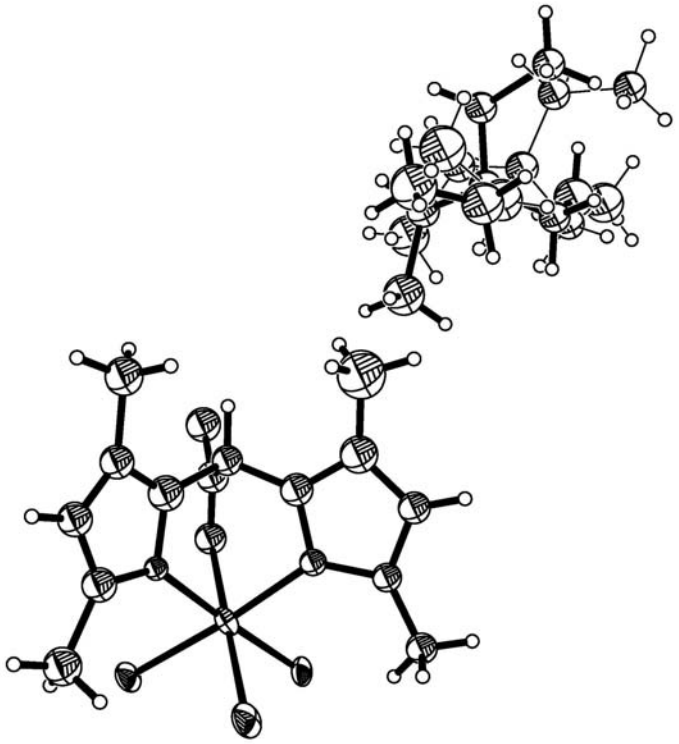


Table 1. Crystal data and structure refinement for **Et_4N-1**.

Identification code	jonjlm15	
Empirical formula	C20 H35 Cl3 N5 O2 Rh	
Formula weight	586.79	
Temperature	100.0(1) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 10.402(8) Å	α = 90°
	b = 12.261(10) Å	β = 100.660(11)°
	c = 19.677(15) Å	γ = 90°
Volume	2466(3) Å^3	
Z	4	
Density (calculated)	1.580 Mg/m^3	
Absorption coefficient	1.044 mm^{-1}	
F(000)	1208	
Crystal color, morphology	yellow, needle	
Crystal size	0.16 x 0.16 x 0.03 mm^3	
Theta range for data collection	1.97 to 25.03°	
Index ranges	$-12 \le h \le 12$, $-14 \le k \le 14$, $-23 \le l \le 23$	
Reflections collected	18744	
Independent reflections	4363 [R(int) = 0.3145]	
Observed reflections	1994	
Completeness to theta = 25.03°	100.0%	
Absorption correction	Multi-scan	
Max. and min. transmission	0.9693 and 0.8507	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4363 / 56 / 175	
Goodness-of-fit on F^2	1.219	
Final R indices [I>2sigma(I)]	R1 = 0.1326, wR2 = 0.2673	
R indices (all data)	R1 = 0.2489, wR2 = 0.3113	
Largest diff. peak and hole	1.746 and -1.354 e.Å^{-3}	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **Et_4N-1**. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Rh1	8265(2)	2352(1)	6220(1)	30(1)
Cl1	7843(4)	1930(4)	7323(2)	29(1)
Cl2	10489(5)	1986(4)	6521(2)	36(1)
Cl3	7828(5)	557(4)	5866(2)	29(1)
N1	8502(13)	2799(11)	5235(7)	24(3)
N2	7833(15)	3724(12)	5009(8)	36(4)
N3	8548(13)	3956(10)	6503(7)	18(3)
N4	7898(16)	4710(13)	6062(8)	41(4)
C1	9108(16)	2345(14)	4729(9)	28(4)
C2	8852(18)	3122(14)	4188(10)	33(5)
C3	8110(20)	3970(17)	4358(11)	46(6)
C4	7041(18)	4292(15)	5413(10)	31(5)
C5	6020(20)	3570(16)	5607(10)	37(5)
O1	4815(14)	3874(10)	5408(7)	45(4)
O2	6307(12)	2710(10)	5942(6)	36(3)
C6	8250(20)	5749(14)	6281(11)	36(5)
C7	9052(19)	5616(16)	6858(10)	37(5)
C8	9230(20)	4528(16)	6998(10)	38(5)
C9	9847(18)	1356(14)	4801(9)	32(5)
C10	7710(30)	4990(20)	3973(13)	76(8)
C11	7730(20)	6711(16)	5823(11)	45(5)
C12	10120(20)	4050(17)	7647(11)	46(6)
N5	4560(20)	5396(18)	1898(11)	38(6)
C13	5940(30)	5810(50)	2140(30)	59(9)
C14	6200(40)	6690(30)	2660(30)	69(11)
C15	3700(30)	6270(30)	1519(19)	35(5)
C16	4320(40)	6700(30)	850(16)	35(5)
C17	4740(50)	4450(30)	1440(20)	35(6)
C18	3430(50)	3950(40)	1030(30)	61(10)
C19	3810(40)	5130(40)	2460(20)	40(8)
C20	4820(50)	4410(40)	3079(18)	62(11)

N5'	4480(20)	5366(18)	1498(11)	38(6)
C13'	5770(40)	5640(50)	1950(30)	59(9)
C14'	5810(50)	6880(30)	2240(20)	69(11)
C15'	4110(40)	6110(30)	887(16)	35(5)
C16'	5090(40)	6090(30)	358(15)	35(5)
C17'	4660(50)	4180(20)	1330(30)	35(6)
C18'	3530(50)	3680(40)	790(30)	61(10)
C19'	3410(30)	5400(30)	1920(19)	40(8)
C20'	3980(50)	4920(50)	2709(19)	62(11)

Table 3. Bond lengths [Å] and angles [°] for **Et_4N-1.**

Rh(1)-N(3)	2.051(13)	C(11)-H(11A)	0.9800
Rh(1)-O(2)	2.057(12)	C(11)-H(11B)	0.9800
Rh(1)-N(1)	2.074(13)	C(11)-H(11C)	0.9800
Rh(1)-Cl(2)	2.323(5)	C(12)-H(12A)	0.9800
Rh(1)-Cl(3)	2.328(5)	C(12)-H(12B)	0.9800
Rh(1)-Cl(1)	2.350(5)	C(12)-H(12C)	0.9800
N(1)-N(2)	1.362(19)	N(5)-C(17)	1.503(14)
N(1)-C(1)	1.39(2)	N(5)-C(19)	1.504(14)
N(2)-C(3)	1.39(3)	N(5)-C(15)	1.505(14)
N(2)-C(4)	1.43(2)	N(5)-C(13)	1.506(14)
N(3)-C(8)	1.30(2)	C(13)-C(14)	1.48(6)
N(3)-N(4)	1.360(19)	C(13)-H(13A)	0.9900
N(4)-C(6)	1.37(2)	C(13)-H(13B)	0.9900
N(4)-C(4)	1.51(2)	C(14)-H(14A)	0.9800
C(1)-C(2)	1.42(2)	C(14)-H(14B)	0.9800
C(1)-C(9)	1.43(2)	C(14)-H(14C)	0.9800
C(2)-C(3)	1.37(3)	C(15)-C(16)	1.66(4)
C(2)-H(2)	0.9500	C(15)-H(15A)	0.9900
C(3)-C(10)	1.48(3)	C(15)-H(15B)	0.9900
C(4)-C(5)	1.49(3)	C(16)-H(16A)	0.9800
C(4)-H(4)	1.0000	C(16)-H(16B)	0.9800
C(5)-O(2)	1.25(2)	C(16)-H(16C)	0.9800
C(5)-O(1)	1.29(2)	C(17)-C(18)	1.57(6)
C(6)-C(7)	1.29(2)	C(17)-H(17A)	0.9900
C(6)-C(11)	1.52(3)	C(17)-H(17B)	0.9900
C(7)-C(8)	1.37(3)	C(18)-H(18A)	0.9800
C(7)-H(7)	0.9500	C(18)-H(18B)	0.9800
C(8)-C(12)	1.54(3)	C(18)-H(18C)	0.9800
C(9)-H(9A)	0.9800	C(19)-C(20)	1.70(5)
C(9)-H(9B)	0.9800	C(19)-H(19A)	0.9900
C(9)-H(9C)	0.9800	C(19)-H(19B)	0.9900
C(10)-H(10A)	0.9800	C(20)-H(20A)	0.9800
C(10)-H(10B)	0.9800	C(20)-H(20B)	0.9800
C(10)-H(10C)	0.9800	C(20)-H(20C)	0.9800

N(5')-C(19')	1.504(14)	N(1)-Rh(1)-Cl(3)	91.2(4)
N(5')-C(13')	1.505(14)	Cl(2)-Rh(1)-Cl(3)	91.38(18)
N(5')-C(15')	1.505(14)	N(3)-Rh(1)-Cl(1)	90.2(4)
N(5')-C(17')	1.505(14)	O(2)-Rh(1)-Cl(1)	87.0(4)
C(13')-C(14')	1.62(7)	N(1)-Rh(1)-Cl(1)	175.4(4)
C(13')-H(13C)	0.9900	Cl(2)-Rh(1)-Cl(1)	94.00(16)
C(13')-H(13D)	0.9900	Cl(3)-Rh(1)-Cl(1)	90.54(16)
C(14')-H(14D)	0.9800	N(2)-N(1)-C(1)	111.9(14)
C(14')-H(14E)	0.9800	N(2)-N(1)-Rh(1)	112.5(10)
C(14')-H(14F)	0.9800	C(1)-N(1)-Rh(1)	135.4(11)
C(15')-C(16')	1.59(4)	N(1)-N(2)-C(3)	107.5(15)
C(15')-H(15C)	0.9900	N(1)-N(2)-C(4)	122.5(15)
C(15')-H(15D)	0.9900	C(3)-N(2)-C(4)	130.0(16)
C(16')-H(16D)	0.9800	C(8)-N(3)-N(4)	104.5(14)
C(16')-H(16E)	0.9800	C(8)-N(3)-Rh(1)	138.8(13)
C(16')-H(16F)	0.9800	N(4)-N(3)-Rh(1)	116.7(11)
C(17')-C(18')	1.56(6)	N(3)-N(4)-C(6)	111.0(16)
C(17')-H(17C)	0.9900	N(3)-N(4)-C(4)	117.2(14)
C(17')-H(17D)	0.9900	C(6)-N(4)-C(4)	131.6(17)
C(18')-H(18D)	0.9800	N(1)-C(1)-C(2)	102.8(15)
C(18')-H(18E)	0.9800	N(1)-C(1)-C(9)	125.2(16)
C(18')-H(18F)	0.9800	C(2)-C(1)-C(9)	131.9(16)
C(19')-C(20')	1.66(5)	C(3)-C(2)-C(1)	111.2(18)
C(19')-H(19C)	0.9900	C(3)-C(2)-H(2)	124.4
C(19')-H(19D)	0.9900	C(1)-C(2)-H(2)	124.4
C(20')-H(20D)	0.9800	C(2)-C(3)-N(2)	106.3(18)
C(20')-H(20E)	0.9800	C(2)-C(3)-C(10)	129(2)
C(20')-H(20F)	0.9800	N(2)-C(3)-C(10)	125(2)
N(3)-Rh(1)-O(2)	87.3(5)	N(2)-C(4)-C(5)	111.5(15)
N(3)-Rh(1)-N(1)	87.9(5)	N(2)-C(4)-N(4)	109.0(15)
O(2)-Rh(1)-N(1)	88.7(5)	C(5)-C(4)-N(4)	109.0(15)
N(3)-Rh(1)-Cl(2)	91.7(4)	N(2)-C(4)-H(4)	109.1
O(2)-Rh(1)-Cl(2)	178.6(4)	C(5)-C(4)-H(4)	109.1
N(1)-Rh(1)-Cl(2)	90.2(4)	N(4)-C(4)-H(4)	109.1
N(3)-Rh(1)-Cl(3)	176.8(4)	O(2)-C(5)-O(1)	121.9(18)
O(2)-Rh(1)-Cl(3)	89.6(4)	O(2)-C(5)-C(4)	121.5(18)

O(1)-C(5)-C(4)	116.7(17)	C(17)-N(5)-C(15)	112(3)
C(5)-O(2)-Rh(1)	116.8(12)	C(19)-N(5)-C(15)	100(3)
C(7)-C(6)-N(4)	104.5(17)	C(17)-N(5)-C(13)	104(3)
C(7)-C(6)-C(11)	136.2(19)	C(19)-N(5)-C(13)	116(3)
N(4)-C(6)-C(11)	119.2(18)	C(15)-N(5)-C(13)	111(3)
C(6)-C(7)-C(8)	110.1(19)	C(14)-C(13)-N(5)	120(4)
C(6)-C(7)-H(7)	125.0	C(14)-C(13)-H(13A)	107.3
C(8)-C(7)-H(7)	125.0	N(5)-C(13)-H(13A)	107.3
N(3)-C(8)-C(7)	109.9(18)	C(14)-C(13)-H(13B)	107.3
N(3)-C(8)-C(12)	125.0(17)	N(5)-C(13)-H(13B)	107.3
C(7)-C(8)-C(12)	125.1(19)	H(13A)-C(13)-H(13B)	106.9
C(1)-C(9)-H(9A)	109.5	C(13)-C(14)-H(14A)	109.5
C(1)-C(9)-H(9B)	109.5	C(13)-C(14)-H(14B)	109.5
H(9A)-C(9)-H(9B)	109.5	H(14A)-C(14)-H(14B)	109.5
C(1)-C(9)-H(9C)	109.5	C(13)-C(14)-H(14C)	109.5
H(9A)-C(9)-H(9C)	109.5	H(14A)-C(14)-H(14C)	109.5
H(9B)-C(9)-H(9C)	109.5	H(14B)-C(14)-H(14C)	109.5
C(3)-C(10)-H(10A)	109.5	N(5)-C(15)-C(16)	110(2)
C(3)-C(10)-H(10B)	109.5	N(5)-C(15)-H(15A)	109.7
H(10A)-C(10)-H(10B)	109.5	C(16)-C(15)-H(15A)	109.7
C(3)-C(10)-H(10C)	109.5	N(5)-C(15)-H(15B)	109.7
H(10A)-C(10)-H(10C)	109.5	C(16)-C(15)-H(15B)	109.7
H(10B)-C(10)-H(10C)	109.5	H(15A)-C(15)-H(15B)	108.2
C(6)-C(11)-H(11A)	109.5	C(15)-C(16)-H(16A)	109.5
C(6)-C(11)-H(11B)	109.5	C(15)-C(16)-H(16B)	109.5
H(11A)-C(11)-H(11B)	109.5	H(16A)-C(16)-H(16B)	109.5
C(6)-C(11)-H(11C)	109.5	C(15)-C(16)-H(16C)	109.5
H(11A)-C(11)-H(11C)	109.5	H(16A)-C(16)-H(16C)	109.5
H(11B)-C(11)-H(11C)	109.5	H(16B)-C(16)-H(16C)	109.5
C(8)-C(12)-H(12A)	109.5	N(5)-C(17)-C(18)	115(3)
C(8)-C(12)-H(12B)	109.5	N(5)-C(17)-H(17A)	108.5
H(12A)-C(12)-H(12B)	109.5	C(18)-C(17)-H(17A)	108.5
C(8)-C(12)-H(12C)	109.5	N(5)-C(17)-H(17B)	108.5
H(12A)-C(12)-H(12C)	109.5	C(18)-C(17)-H(17B)	108.5
H(12B)-C(12)-H(12C)	109.5	H(17A)-C(17)-H(17B)	107.5
C(17)-N(5)-C(19)	114(3)	C(17)-C(18)-H(18A)	109.5

C(17)-C(18)-H(18B)	109.5	N(5')-C(15')-H(15C)	108.6
H(18A)-C(18)-H(18B)	109.5	C(16')-C(15')-H(15C)	108.6
C(17)-C(18)-H(18C)	109.5	N(5')-C(15')-H(15D)	108.6
H(18A)-C(18)-H(18C)	109.5	C(16')-C(15')-H(15D)	108.6
H(18B)-C(18)-H(18C)	109.5	H(15C)-C(15')-H(15D)	107.6
N(5)-C(19)-C(20)	108(3)	C(15')-C(16')-H(16D)	109.5
N(5)-C(19)-H(19A)	110.2	C(15')-C(16')-H(16E)	109.5
C(20)-C(19)-H(19A)	110.2	H(16D)-C(16')-H(16E)	109.5
N(5)-C(19)-H(19B)	110.2	C(15')-C(16')-H(16F)	109.5
C(20)-C(19)-H(19B)	110.2	H(16D)-C(16')-H(16F)	109.5
H(19A)-C(19)-H(19B)	108.5	H(16E)-C(16')-H(16F)	109.5
C(19)-C(20)-H(20A)	109.5	N(5')-C(17')-C(18')	115(4)
C(19)-C(20)-H(20B)	109.5	N(5')-C(17')-H(17C)	108.5
H(20A)-C(20)-H(20B)	109.5	C(18')-C(17')-H(17C)	108.5
C(19)-C(20)-H(20C)	109.5	N(5')-C(17')-H(17D)	108.5
H(20A)-C(20)-H(20C)	109.5	C(18')-C(17')-H(17D)	108.5
H(20B)-C(20)-H(20C)	109.5	H(17C)-C(17')-H(17D)	107.5
C(19')-N(5')-C(13')	110(3)	C(17')-C(18')-H(18D)	109.5
C(19')-N(5')-C(15')	109(3)	C(17')-C(18')-H(18E)	109.5
C(13')-N(5')-C(15')	114(3)	H(18D)-C(18')-H(18E)	109.5
C(19')-N(5')-C(17')	106(3)	C(17')-C(18')-H(18F)	109.5
C(13')-N(5')-C(17')	102(3)	H(18D)-C(18')-H(18F)	109.5
C(15')-N(5')-C(17')	116(3)	H(18E)-C(18')-H(18F)	109.5
N(5')-C(13')-C(14')	112(4)	N(5')-C(19')-C(20')	110(3)
N(5')-C(13')-H(13C)	109.1	N(5')-C(19')-H(19C)	109.8
C(14')-C(13')-H(13C)	109.1	C(20')-C(19')-H(19C)	109.8
N(5')-C(13')-H(13D)	109.1	N(5')-C(19')-H(19D)	109.8
C(14')-C(13')-H(13D)	109.1	C(20')-C(19')-H(19D)	109.8
H(13C)-C(13')-H(13D)	107.9	H(19C)-C(19')-H(19D)	108.2
C(13')-C(14')-H(14D)	109.5	C(19')-C(20')-H(20D)	109.5
C(13')-C(14')-H(14E)	109.5	C(19')-C(20')-H(20E)	109.5
H(14D)-C(14')-H(14E)	109.5	H(20D)-C(20')-H(20E)	109.5
C(13')-C(14')-H(14F)	109.5	C(19')-C(20')-H(20F)	109.5
H(14D)-C(14')-H(14F)	109.5	H(20D)-C(20')-H(20F)	109.5
H(14E)-C(14')-H(14F)	109.5	H(20E)-C(20')-H(20F)	109.5
N(5')-C(15')-C(16')	114(3)		

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **Et_4N-1**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U_{11} + ... + 2\ h\ k\ a^*\ b^*\ U_{12}\ ]$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Rh1	39(1)	36(1)	14(1)	2(1)	4(1)	4(1)
Cl1	32(3)	39(3)	19(2)	4(2)	11(2)	0(2)
Cl2	35(3)	51(3)	24(2)	5(2)	15(2)	0(2)
Cl3	30(3)	40(3)	19(2)	-3(2)	11(2)	5(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3) for **Et_4N-1**.

	x	y	z	U(eq)
H2	9155	3065	3763	40
H4	6610	4924	5140	37
H7	9460	6190	7143	44
H9A	9553	889	5147	48
H9B	10777	1525	4950	48
H9C	9717	975	4356	48
H10A	6782	5128	3968	114
H10B	7857	4912	3497	114
H10C	8233	5597	4198	114
H11A	7888	7389	6090	67
H11B	6785	6621	5659	67
H11C	8173	6744	5426	67
H12A	9613	3531	7872	69
H12B	10436	4641	7969	69
H12C	10861	3675	7510	69
H13A	6483	5176	2322	71
H13B	6261	6063	1721	71
H14A	7142	6831	2774	104
H14B	5884	6469	3079	104
H14C	5744	7358	2474	104
H15A	3631	6885	1836	42
H15B	2810	5973	1357	42
H16A	3750	7272	605	53
H16B	4373	6094	536	53
H16C	5193	7004	1014	53
H17A	5222	3865	1730	42
H17B	5286	4684	1106	42
H18A	3637	3340	746	92
H18B	2959	4513	727	92
H18C	2892	3695	1353	92

H19A	3027	4696	2272	48
H19B	3529	5812	2661	48
H20A	4348	4213	3449	93
H20B	5584	4853	3270	93
H20C	5105	3744	2874	93
H13C	6478	5545	1679	71
H13D	5937	5132	2345	71
H14D	6684	7036	2504	104
H14E	5160	6963	2535	104
H14F	5614	7387	1848	104
H15C	4059	6869	1058	42
H15D	3226	5910	637	42
H16D	4773	6588	-27	53
H16E	5139	5349	180	53
H16F	5959	6324	594	53
H17C	4742	3757	1766	42
H17D	5485	4105	1159	42
H18D	3735	2910	710	92
H18E	3444	4080	354	92
H18F	2708	3717	963	92
H19C	2664	4949	1694	48
H19D	3105	6157	1950	48
H20D	3289	4946	2985	93
H20E	4722	5368	2931	93
H20F	4273	4164	2677	93

Table 6. Torsion angles [°] for **Et_4N-1.**

N3-Rh1-N1-N2	-44.6(11)	C1-C2-C3-N2	-2(2)
O2-Rh1-N1-N2	42.8(11)	C1-C2-C3-C10	174(2)
Cl2-Rh1-N1-N2	-136.3(11)	N1-N2-C3-C2	5(2)
Cl3-Rh1-N1-N2	132.4(11)	C4-N2-C3-C2	-176.2(18)
Cl1-Rh1-N1-N2	21(6)	N1-N2-C3-C10	-172(2)
N3-Rh1-N1-C1	140.7(16)	C4-N2-C3-C10	7(3)
O2-Rh1-N1-C1	-131.9(16)	N1-N2-C4-C5	-57(2)
Cl2-Rh1-N1-C1	49.0(15)	C3-N2-C4-C5	125(2)
Cl3-Rh1-N1-C1	-42.4(15)	N1-N2-C4-N4	64(2)
Cl1-Rh1-N1-C1	-154(4)	C3-N2-C4-N4	-115(2)
C1-N1-N2-C3	-6(2)	N3-N4-C4-N2	-62(2)
Rh1-N1-N2-C3	177.6(12)	C6-N4-C4-N2	112(2)
C1-N1-N2-C4	174.8(15)	N3-N4-C4-C5	60(2)
Rh1-N1-N2-C4	-1(2)	C6-N4-C4-C5	-126(2)
O2-Rh1-N3-C8	139.0(19)	N2-C4-C5-O2	58(2)
N1-Rh1-N3-C8	-132.2(19)	N4-C4-C5-O2	-62(2)
Cl2-Rh1-N3-C8	-42.1(19)	N2-C4-C5-O1	-121.3(19)
Cl3-Rh1-N3-C8	154(6)	N4-C4-C5-O1	118.3(19)
Cl1-Rh1-N3-C8	51.9(19)	O1-C5-O2-Rh1	-179.8(14)
O2-Rh1-N3-N4	-43.7(12)	C4-C5-O2-Rh1	1(2)
N1-Rh1-N3-N4	45.1(12)	N3-Rh1-O2-C5	44.4(13)
Cl2-Rh1-N3-N4	135.3(12)	N1-Rh1-O2-C5	-43.6(13)
Cl3-Rh1-N3-N4	-28(8)	Cl2-Rh1-O2-C5	-2(16)
Cl1-Rh1-N3-N4	-130.7(12)	Cl3-Rh1-O2-C5	-134.7(13)
C8-N3-N4-C6	3(2)	Cl1-Rh1-O2-C5	134.7(13)
Rh1-N3-N4-C6	-175.1(12)	N3-N4-C6-C7	-3(2)
C8-N3-N4-C4	178.2(16)	C4-N4-C6-C7	-176.8(18)
Rh1-N3-N4-C4	0.0(19)	N3-N4-C6-C11	174.4(16)
N2-N1-C1-C2	4.9(18)	C4-N4-C6-C11	0(3)
Rh1-N1-C1-C2	179.7(13)	N4-C6-C7-C8	1(2)
N2-N1-C1-C9	-177.9(17)	C11-C6-C7-C8	-175(2)
Rh1-N1-C1-C9	-3(3)	N4-N3-C8-C7	-2(2)
N1-C1-C2-C3	-2(2)	Rh1-N3-C8-C7	175.3(14)
C9-C1-C2-C3	-178.5(19)	N4-N3-C8-C12	178.0(18)

Rh1-N3-C8-C12	-4(3)
C6-C7-C8-N3	1(2)
C6-C7-C8-C12	-179.6(19)
C17-N5-C13-C14	-174(5)
C19-N5-C13-C14	-48(7)
C15-N5-C13-C14	65(6)
C17-N5-C15-C16	-58(4)
C19-N5-C15-C16	-179(3)
C13-N5-C15-C16	58(4)
C19-N5-C17-C18	62(5)
C15-N5-C17-C18	-51(5)
C13-N5-C17-C18	-171(4)
C17-N5-C19-C20	75(4)
C15-N5-C19-C20	-165(3)
C13-N5-C19-C20	-45(5)
C19'-N5'-C13'-C14'	-65(5)
C15'-N5'-C13'-C14'	57(5)
C17'-N5'-C13'-C14'	-177(4)
C19'-N5'-C15'-C16'	-175(3)
C13'-N5'-C15'-C16'	62(4)
C17'-N5'-C15'-C16'	-56(4)
C19'-N5'-C17'-C18'	72(5)
C13'-N5'-C17'-C18'	-173(4)
C15'-N5'-C17'-C18'	-48(5)
C13'-N5'-C19'-C20'	-37(4)
C15'-N5'-C19'-C20'	-162(3)
C17'-N5'-C19'-C20'	73(4)

REFERENCE NUMBER: jonjlm19

CRYSTAL STRUCTURE REPORT

$C_{12}\ H_{15}\ Cl_3\ Ir\ N_4\ Na\ O_2$

or

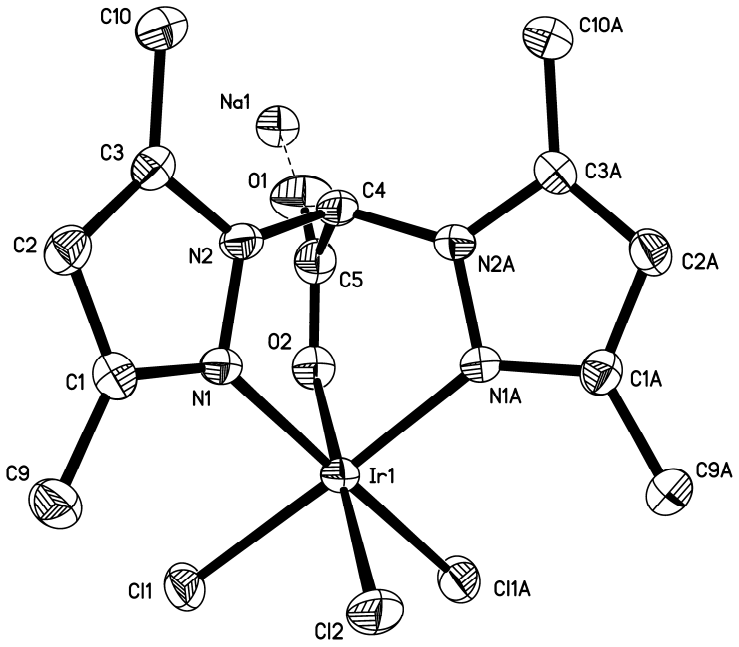
$\{Na[IrCl_3(bdmpza)]\}_n$

Na-2

Report prepared for:

J. Miller, Prof. W. Jones

June 24, 2009



William W. Brennessel

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Data collection

A crystal (0.18 x 0.12 x 0.04 mm^3) was placed onto the tip of a 0.1 mm diameter glass capillary tube or fiber and mounted on a Bruker SMART APEX II CCD Platform diffractometer for a data collection at 100.0(1) K.[1] A preliminary set of cell constants and an orientation matrix were calculated from reflections harvested from three orthogonal wedges of reciprocal space. The full data collection was carried out using MoKα radiation (graphite monochromator) with a frame time of 60 seconds and a detector distance of 4.00 cm. A randomly oriented region of reciprocal space was surveyed: four major sections of frames were collected with 0.50º steps in ω at four different ϕ settings and a detector position of -38º in 2θ. The intensity data were corrected for absorption.[2] Final cell constants were calculated from the xyz centroids of 4020 strong reflections from the actual data collection after integration.[3] See Table 1 for additional crystal and refinement information.

Structure solution and refinement

The structure was solved using SIR97[4] and refined using SHELXL-97.[5] The space group *Pnma* was determined based on systematic absences and intensity statistics. A direct-methods solution was calculated which provided most non-hydrogen atoms from the E-map. Full-matrix least squares / difference Fourier cycles were performed which located the remaining non-hydrogen atoms. All non-hydrogen atoms were refined with anisotropic displacement parameters. All hydrogen atoms were placed in ideal positions and refined as riding atoms with relative isotropic displacement parameters. The final full matrix least squares refinement converged to $R1 = 0.0410$ (F^2, $I > 2\sigma(I)$) and $wR2 = 0.1023$ (F^2, all data).

Structure description

The structure is the one suggested. The cation and anion lie on a crystallographic mirror plane; thus one half of each is unique. Sodium cations link the anions to form infinite chains parallel to the *a*-axis (see diagram).

Unless noted otherwise all structural diagrams containing thermal displacement ellipsoids are drawn at the 50 % probability level.

Data collection, structure solution, and structure refinement were conducted at the X-ray Crystallographic Facility, B51 Hutchison Hall, Department of Chemistry, University of Rochester.

[1] *APEX2*, version 2009.3-0; Bruker AXS: Madison, WI, 2009.

[2] Sheldrick, G. M. *SADABS*, version 2008/1; University of Göttingen: Göttingen, Germany, 2008.

[3] *SAINT*, version 7.60A; Bruker AXS: Madison, WI, 2008.

[4] Altomare, A.; Burla, M. C.; Camalli, M.; Cascarano, G. L.; Giacovazzo, C.; Guagliardi, A.; Moliterni, A. G. G.; Polidori, G.; Spagna, R. *SIR97: A new program for solving and refining crystal structures*; Istituto di Cristallografia, CNR: Bari, Italy, 1999.

[5] Sheldrick, G. M. *Acta. Cryst.* **2008**, *A64*, 112-122.

Some equations of interest:

$$R_{int} = \Sigma\, |F_o^2 - < F_o^2 >| \,/\, \Sigma |F_o^2|$$

$$R1 = \Sigma ||F_o| - |F_c|| \,/\, \Sigma |F_o|$$

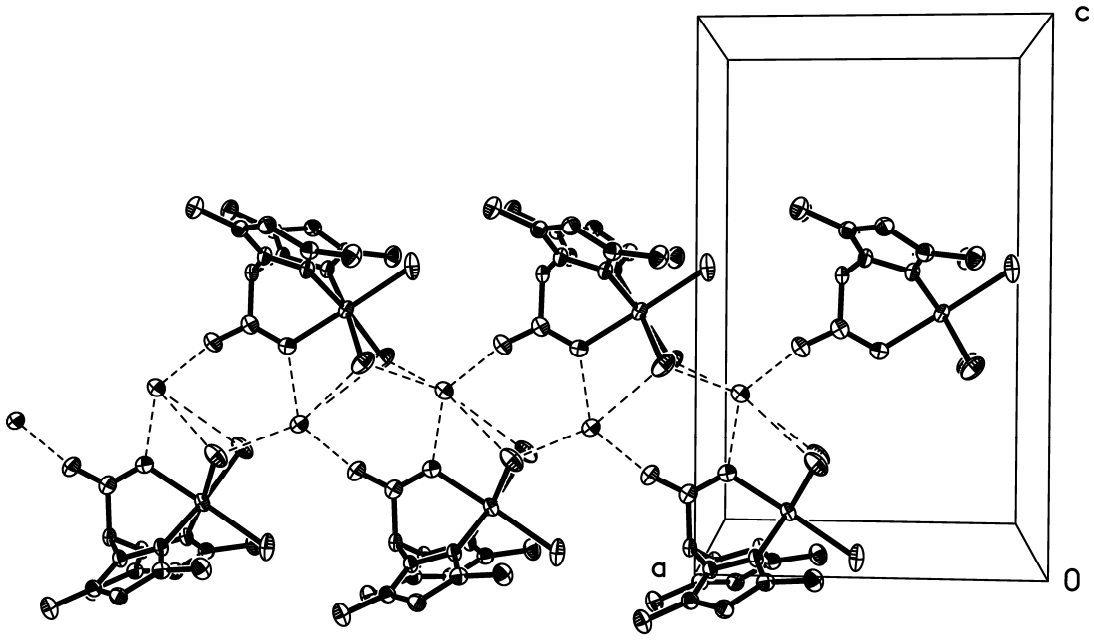
$$wR2 = [\Sigma [w(F_o^2 - F_c^2)^2] \,/\, \Sigma\, [w(F_o^2)^2]]^{1/2}$$

where $w = 1 \,/\, [\sigma^2 (F_o^2) + (aP)^2 + bP]$ and

$$P = 1/3 \max (0, F_o^2) + 2/3\, F_c^2$$

$$\text{GOF} = S = [\Sigma [w(F_o^2 - F_c^2)^2] \,/\, (m-n)]^{1/2}$$

where m = number of reflections and n = number of parameters



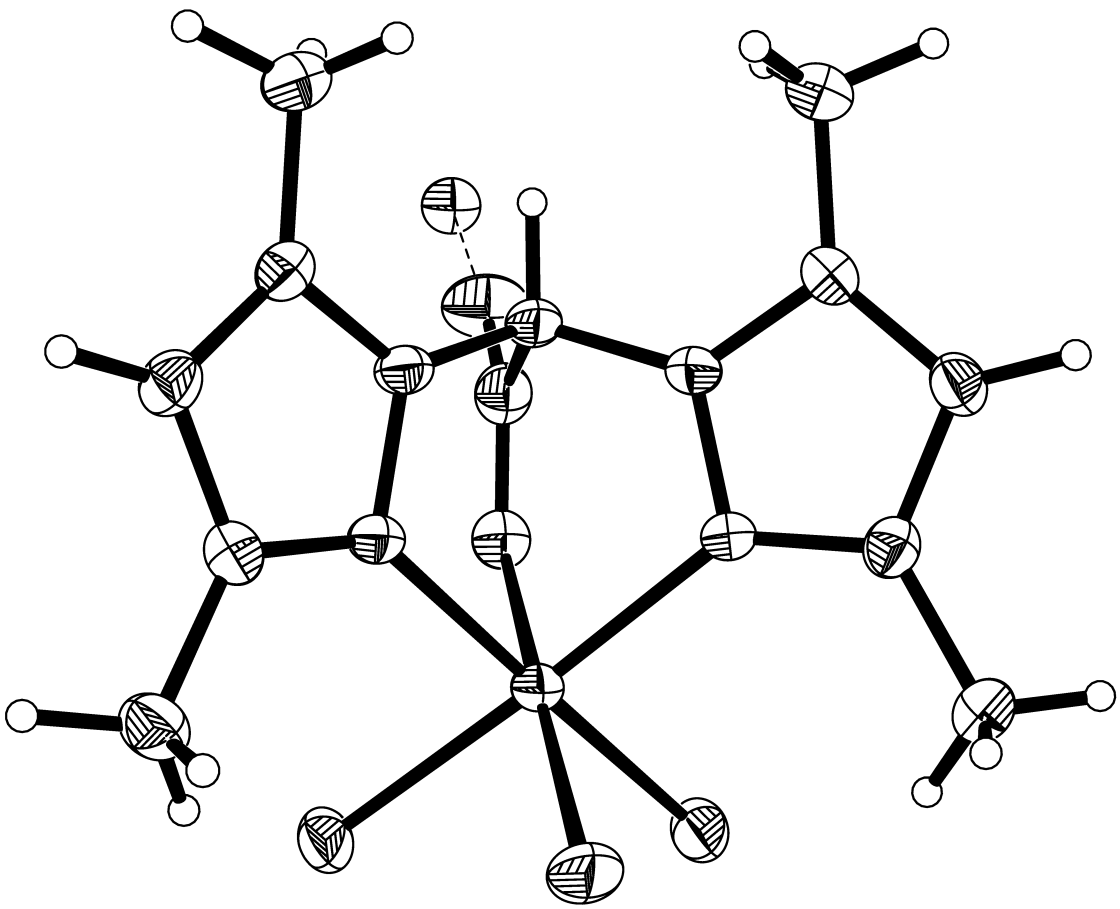


Table 1. Crystal data and structure refinement for **Na-2**.

Identification code	jonjlm19	
Empirical formula	C12 H15 Cl3 Ir N4 Na O2	
Formula weight	568.82	
Temperature	100.0(1) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	*Pn*m*a*	
Unit cell dimensions	a = 8.5743(10) Å	α = 90°
	b = 14.2503(17) Å	β = 90°
	c = 13.7914(16) Å	γ = 90°
Volume	1685.1(3) Å^3	
Z	4	
Density (calculated)	2.242 Mg/m^3	
Absorption coefficient	8.436 mm^{-1}	
F(000)	1080	
Crystal color, morphology	yellow, plate	
Crystal size	0.18 x 0.12 x 0.04 mm^3	
Theta range for data collection	2.05 to 37.78°	
Index ranges	$-14 \le h \le 14$, $-24 \le k \le 24$, $-23 \le l \le 23$	
Reflections collected	39500	
Independent reflections	4649 [R(int) = 0.0809]	
Observed reflections	3236	
Completeness to theta = 37.78°	99.8%	
Absorption correction	Multi-scan	
Max. and min. transmission	0.7190 and 0.3020	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4649 / 0 / 117	
Goodness-of-fit on F^2	1.025	
Final R indices [I>2sigma(I)]	R1 = 0.0410, wR2 = 0.0904	
R indices (all data)	R1 = 0.0744, wR2 = 0.1023	
Largest diff. peak and hole	4.622 and -1.285 e.Å^{-3}	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **Na-2**. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Na1	9305(3)	2500	2876(2)	29(1)
Ir1	2686(1)	2500	4575(1)	22(1)
Cl1	1740(1)	3639(1)	3485(1)	35(1)
Cl2	421(2)	2500	5502(1)	38(1)
N1	3683(3)	3507(2)	5430(2)	21(1)
N2	5197(3)	3332(2)	5706(2)	22(1)
C1	3311(5)	4337(3)	5818(3)	24(1)
C2	4605(4)	4683(3)	6327(3)	25(1)
C3	5770(4)	4037(3)	6261(3)	23(1)
C4	5962(6)	2500	5350(4)	21(1)
C5	6024(7)	2500	4232(4)	28(1)
O1	7309(5)	2500	3848(4)	41(1)
O2	4722(5)	2500	3782(3)	26(1)
C9	1757(5)	4783(3)	5709(3)	32(1)
C10	7331(4)	3974(3)	6698(4)	32(1)

Table 3. Bond lengths [Å] and angles [°] for **Na-2**.

Na(1)-O(1)	2.174(5)	C(10)-H(10C)	0.9800
Na(1)-O(2)#1	2.314(5)	O(1)-Na(1)-O(2)#1	137.0(2)
Na(1)-Cl(1)#2	2.775(2)	O(1)-Na(1)-Cl(1)#2	113.91(13)
Na(1)-Cl(1)#3	2.775(2)	O(2)#1-Na(1)-Cl(1)#2	100.55(11)
Na(1)-Cl(1)#4	3.316(2)	O(1)-Na(1)-Cl(1)#3	113.91(13)
Na(1)-Cl(1)#1	3.316(2)	O(2)#1-Na(1)-Cl(1)#3	100.55(11)
Ir(1)-N(1)#5	2.044(3)	Cl(1)#2-Na(1)-Cl(1)#3	71.61(7)
Ir(1)-N(1)	2.044(3)	O(1)-Na(1)-Cl(1)#4	80.04(13)
Ir(1)-O(2)	2.059(4)	O(2)#1-Na(1)-Cl(1)#4	62.83(10)
Ir(1)-Cl(2)	2.3258(15)	Cl(1)#2-Na(1)-Cl(1)#4	163.12(8)
Ir(1)-Cl(1)#5	2.3565(10)	Cl(1)#3-Na(1)-Cl(1)#4	112.59(4)
Ir(1)-Cl(1)	2.3566(10)	O(1)-Na(1)-Cl(1)#1	80.04(13)
Cl(1)-Na(1)#6	2.775(2)	O(2)#1-Na(1)-Cl(1)#1	62.83(10)
Cl(1)-Na(1)#7	3.316(2)	Cl(1)#2-Na(1)-Cl(1)#1	112.59(4)
N(1)-C(1)	1.338(5)	Cl(1)#3-Na(1)-Cl(1)#1	163.12(8)
N(1)-N(2)	1.375(4)	Cl(1)#4-Na(1)-Cl(1)#1	58.62(5)
N(2)-C(3)	1.355(5)	N(1)#5-Ir(1)-N(1)	89.12(17)
N(2)-C(4)	1.441(4)	N(1)#5-Ir(1)-O(2)	87.25(12)
C(1)-C(2)	1.402(5)	N(1)-Ir(1)-O(2)	87.25(12)
C(1)-C(9)	1.484(6)	N(1)#5-Ir(1)-Cl(2)	91.81(9)
C(2)-C(3)	1.361(5)	N(1)-Ir(1)-Cl(2)	91.81(9)
C(2)-H(2)	0.9500	O(2)-Ir(1)-Cl(2)	178.69(12)
C(3)-C(10)	1.471(5)	N(1)#5-Ir(1)-Cl(1)#5	91.65(9)
C(4)-N(2)#5	1.441(4)	N(1)-Ir(1)-Cl(1)#5	174.49(9)
C(4)-C(5)	1.543(8)	O(2)-Ir(1)-Cl(1)#5	87.33(8)
C(4)-H(4)	1.0000	Cl(2)-Ir(1)-Cl(1)#5	93.62(5)
C(5)-O(1)	1.222(7)	N(1)#5-Ir(1)-Cl(1)	174.49(9)
C(5)-O(2)	1.277(7)	N(1)-Ir(1)-Cl(1)	91.65(9)
O(2)-Na(1)#7	2.314(5)	O(2)-Ir(1)-Cl(1)	87.33(8)
C(9)-H(9A)	0.9800	Cl(2)-Ir(1)-Cl(1)	93.62(4)
C(9)-H(9B)	0.9800	Cl(1)#5-Ir(1)-Cl(1)	87.08(5)
C(9)-H(9C)	0.9800	Ir(1)-Cl(1)-Na(1)#6	92.83(5)
C(10)-H(10A)	0.9800	Ir(1)-Cl(1)-Na(1)#7	78.18(5)
C(10)-H(10B)	0.9800	Na(1)#6-Cl(1)-Na(1)#7	92.37(3)

C(1)-N(1)-N(2)	105.9(3)	C(5)-C(4)-H(4)	108.1
C(1)-N(1)-Ir(1)	138.8(3)	O(1)-C(5)-O(2)	125.3(6)
N(2)-N(1)-Ir(1)	115.3(2)	O(1)-C(5)-C(4)	117.6(5)
C(3)-N(2)-N(1)	111.4(3)	O(2)-C(5)-C(4)	117.1(5)
C(3)-N(2)-C(4)	129.6(3)	C(5)-O(1)-Na(1)	167.5(5)
N(1)-N(2)-C(4)	119.0(3)	C(5)-O(2)-Ir(1)	118.9(4)
N(1)-C(1)-C(2)	108.8(3)	C(5)-O(2)-Na(1)#7	127.9(4)
N(1)-C(1)-C(9)	123.5(4)	Ir(1)-O(2)-Na(1)#7	113.17(18)
C(2)-C(1)-C(9)	127.7(4)	C(1)-C(9)-H(9A)	109.5
C(3)-C(2)-C(1)	108.1(3)	C(1)-C(9)-H(9B)	109.5
C(3)-C(2)-H(2)	126.0	H(9A)-C(9)-H(9B)	109.5
C(1)-C(2)-H(2)	126.0	C(1)-C(9)-H(9C)	109.5
N(2)-C(3)-C(2)	105.8(3)	H(9A)-C(9)-H(9C)	109.5
N(2)-C(3)-C(10)	121.1(3)	H(9B)-C(9)-H(9C)	109.5
C(2)-C(3)-C(10)	132.9(4)	C(3)-C(10)-H(10A)	109.5
N(2)#5-C(4)-N(2)	110.7(4)	C(3)-C(10)-H(10B)	109.5
N(2)#5-C(4)-C(5)	110.9(3)	H(10A)-C(10)-H(10B)	109.5
N(2)-C(4)-C(5)	110.9(3)	C(3)-C(10)-H(10C)	109.5
N(2)#5-C(4)-H(4)	108.1	H(10A)-C(10)-H(10C)	109.5
N(2)-C(4)-H(4)	108.1	H(10B)-C(10)-H(10C)	109.5

Symmetry transformations used to generate equivalent atoms:

#1 x+1/2,y,-z+1/2 #2 x+1,y,z #3 x+1,-y+1/2,z

#4 x+1/2,-y+1/2,-z+1/2 #5 x,-y+1/2,z #6 x-1,y,z

#7 x-1/2,y,-z+1/2

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **Na-2**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U_{11} + ... + 2\ h\ k\ a^*\ b^*\ U_{12}\ ]$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Na1	28(1)	33(1)	26(1)	0	3(1)	0
Ir1	19(1)	19(1)	27(1)	0	-6(1)	0
Cl1	43(1)	23(1)	40(1)	3(1)	-18(1)	0(1)
Cl2	17(1)	35(1)	62(1)	0	4(1)	0
N1	16(1)	21(1)	26(1)	-1(1)	-3(1)	1(1)
N2	16(1)	23(1)	26(2)	-2(1)	-2(1)	0(1)
C1	26(2)	22(2)	23(2)	1(1)	0(1)	2(1)
C2	27(2)	24(2)	25(2)	-5(1)	0(1)	-2(1)
C3	24(2)	22(2)	23(2)	-2(1)	-1(1)	-3(1)
C4	14(2)	23(2)	27(2)	0	0(2)	0
C5	27(3)	25(3)	31(3)	0	3(2)	0
O1	26(2)	65(4)	32(2)	0	8(2)	0
O2	28(2)	27(2)	24(2)	0	1(2)	0
C9	28(2)	29(2)	39(2)	-5(2)	-4(2)	7(2)
C10	25(2)	31(2)	38(2)	-3(2)	-7(2)	-2(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for **Na-2**.

	x	y	z	U(eq)
H2	4660	5266	6659	30
H4	7058	2500	5598	26
H9A	1296	4594	5088	48
H9B	1871	5467	5726	48
H9C	1076	4581	6241	48
H10A	7603	4578	6993	47
H10B	8097	3818	6196	47
H10C	7334	3485	7198	47

Table 6. Torsion angles [°] for **Na-2**.

N1#5-Ir1-Cl1-Na1#6	108.0(9)	C4-N2-C3-C10	-7.9(6)
N1-Ir1-Cl1-Na1#6	-154.12(10)	C1-C2-C3-N2	1.4(4)
O2-Ir1-Cl1-Na1#6	118.70(9)	C1-C2-C3-C10	-174.7(4)
Cl2-Ir1-Cl1-Na1#6	-62.20(6)	C3-N2-C4-N2#5	119.7(4)
Cl1#5-Ir1-Cl1-Na1#6	31.24(7)	N1-N2-C4-N2#5	-64.1(5)
N1#5-Ir1-Cl1-Na1#7	16.1(9)	C3-N2-C4-C5	-116.8(5)
N1-Ir1-Cl1-Na1#7	114.05(9)	N1-N2-C4-C5	59.4(5)
O2-Ir1-Cl1-Na1#7	26.88(8)	N2#5-C4-C5-O1	-118.3(3)
Cl2-Ir1-Cl1-Na1#7	-154.03(4)	N2-C4-C5-O1	118.3(3)
Cl1#5-Ir1-Cl1-Na1#7	-60.58(5)	N2#5-C4-C5-O2	61.7(3)
N1#5-Ir1-N1-C1	-139.5(3)	N2-C4-C5-O2	-61.7(3)
O2-Ir1-N1-C1	133.2(4)	O2-C5-O1-Na1	0.000(3)
Cl2-Ir1-N1-C1	-47.8(4)	C4-C5-O1-Na1	180.000(3)
Cl1#5-Ir1-N1-C1	122.4(8)	O2#1-Na1-O1-C5	0.000(4)
Cl1-Ir1-N1-C1	45.9(4)	Cl1#2-Na1-O1-C5	140.21(7)
N1#5-Ir1-N1-N2	41.8(3)	Cl1#3-Na1-O1-C5	-140.21(7)
O2-Ir1-N1-N2	-45.5(2)	Cl1#4-Na1-O1-C5	-29.81(3)
Cl2-Ir1-N1-N2	133.6(2)	Cl1#1-Na1-O1-C5	29.81(3)
Cl1#5-Ir1-N1-N2	-56.3(10)	O1-C5-O2-Ir1	180.0
Cl1-Ir1-N1-N2	-132.8(2)	C4-C5-O2-Ir1	0.0
C1-N1-N2-C3	0.3(4)	O1-C5-O2-Na1#7	0.000(1)
Ir1-N1-N2-C3	179.4(2)	C4-C5-O2-Na1#7	180.0
C1-N1-N2-C4	-176.6(3)	N1#5-Ir1-O2-C5	-44.62(8)
Ir1-N1-N2-C4	2.5(4)	N1-Ir1-O2-C5	44.62(8)
N2-N1-C1-C2	0.7(4)	Cl2-Ir1-O2-C5	0.000(12)
Ir1-N1-C1-C2	-178.1(3)	Cl1#5-Ir1-O2-C5	-136.40(3)
N2-N1-C1-C9	-178.9(4)	Cl1-Ir1-O2-C5	136.40(3)
Ir1-N1-C1-C9	2.3(6)	N1#5-Ir1-O2-Na1#7	135.38(8)
N1-C1-C2-C3	-1.3(5)	N1-Ir1-O2-Na1#7	-135.38(8)
C9-C1-C2-C3	178.2(4)	Cl2-Ir1-O2-Na1#7	180.000(12)
N1-N2-C3-C2	-1.1(4)	Cl1#5-Ir1-O2-Na1#7	43.60(3)
C4-N2-C3-C2	175.4(4)	Cl1-Ir1-O2-Na1#7	-43.60(3)
N1-N2-C3-C10	175.7(3)		

Symmetry transformations used to generate equivalent atoms:

#1 x+1/2,y,-z+1/2 #2 x+1,y,z #3 x+1,-y+1/2,z

#4 x+1/2,-y+1/2,-z+1/2 #5 x,-y+1/2,z #6 x-1,y,z

#7 x-1/2,y,-z+1/2

REFERENCE NUMBER: jonjlm06

CRYSTAL STRUCTURE REPORT

$C_{17}\ H_{20}\ Cl_2\ N_5\ O_2\ Rh$

or

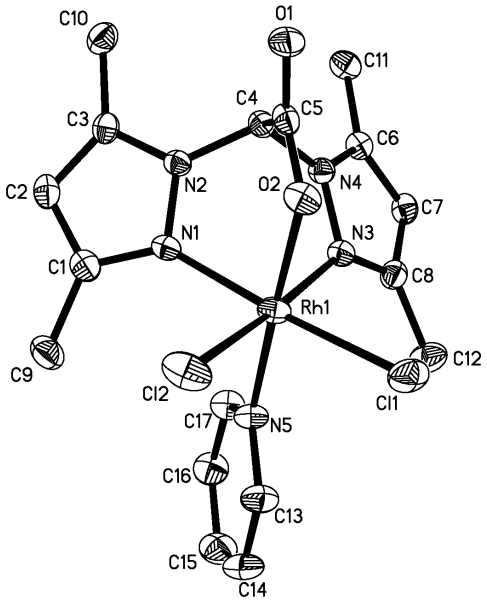
$RhCl_2$(bdmpza)(py)

3a

Report prepared for:

J. Miller, Prof. W. Jones

May 30, 2008



William W. Brennessel

X-ray Crystallographic Facility

Department of Chemistry

University of Rochester

120 Trustee Road

Rochester, NY 14627

Data collection

A crystal (0.34 x 0.10 x 0.06 mm^3) was placed onto the tip of a 0.1 mm diameter glass capillary tube or fiber and mounted on a Bruker SMART APEX II CCD Platform diffractometer for a data collection at 100.0(1) K.[1] A preliminary set of cell constants and an orientation matrix were calculated from 115 reflections harvested from three sets of 20 frames. These initial sets of frames were oriented such that orthogonal wedges of reciprocal space were surveyed. The data collection was carried out using MoKα radiation (graphite monochromator) with a frame time of 45 seconds and a detector distance of 3.95 cm. A randomly oriented region of reciprocal space was surveyed: four major sections of frames were collected with 0.50º steps in ω at four different ϕ settings and a detector position of -38º in 2θ. The intensity data were corrected for absorption.[2] Final cell constants were calculated from the xyz centroids of 4092 strong reflections from the actual data collection after integration.[3] See Table 1 for additional crystal and refinement information.

Structure solution and refinement

The structure was solved using SIR97[4] and refined using SHELXL-97.[5] The space group $P2_1/c$ was determined based on systematic absences and intensity statistics. A direct-methods solution was calculated which provided most non-hydrogen atoms from the E-map. Full-matrix least squares / difference Fourier cycles were performed which located the remaining non-hydrogen atoms. All non-hydrogen atoms were refined with anisotropic displacement parameters. All hydrogen atoms were placed in ideal positions and refined as riding atoms with relative isotropic displacement parameters. The final full matrix least squares refinement converged to $R1 = 0.0438$ (F^2, $I > 2\sigma(I)$) and $wR2 = 0.1114$ (F^2, all data).

Structure description

The structure is the one suggested with all atoms in general positions. The entire molecule is modeled as disordered over two positions (89:11).

Data collection, structure solution, and structure refinement were conducted at the X-ray Crystallographic Facility, B51 Hutchison Hall, Department of Chemistry, University of Rochester.

[1] *APEX2*, version 2.2-0; Bruker AXS: Madison, WI, 2007.

[2] Sheldrick, G. M. *SADABS*, version 2007/4; University of Göttingen: Göttingen, Germany, 2007.

[3] *SAINT*, version 7.46A; Bruker AXS: Madison, WI, 2007.

[4] Altomare, A.; Burla, M. C.; Camalli, M.; Cascarano, G. L.; Giacovazzo, C.; Guagliardi, A.; Moliterni, A. G. G.; Polidori, G.; Spagna, R. *SIR97: A new program for solving and refining crystal structures*; Istituto di Cristallografia, CNR: Bari, Italy, 1999.

[5] Sheldrick, G. M. *Acta. Cryst.* **2008**, *A64*, 112-122.

Some equations of interest:

$$R_{int} = \Sigma|F_o^2 - \langle F_o^2 \rangle| \,/\, \Sigma|F_o^2|$$

$$R1 = \Sigma||F_o| - |F_c|| \,/\, \Sigma|F_o|$$

$$wR2 = [\Sigma[w(F_o^2 - F_c^2)^2] \,/\, \Sigma[w(F_o^2)^2]]^{1/2}$$

where $w = 1 / [\sigma^2(F_o^2) + (aP)^2 + bP]$ and

$$P = 1/3 \max(0, F_o^2) + 2/3\, F_c^2$$

$$\text{GOF} = S = [\Sigma[w(F_o^2 - F_c^2)^2] / (m-n)]^{1/2}$$

where m = number of reflections and n = number of parameters

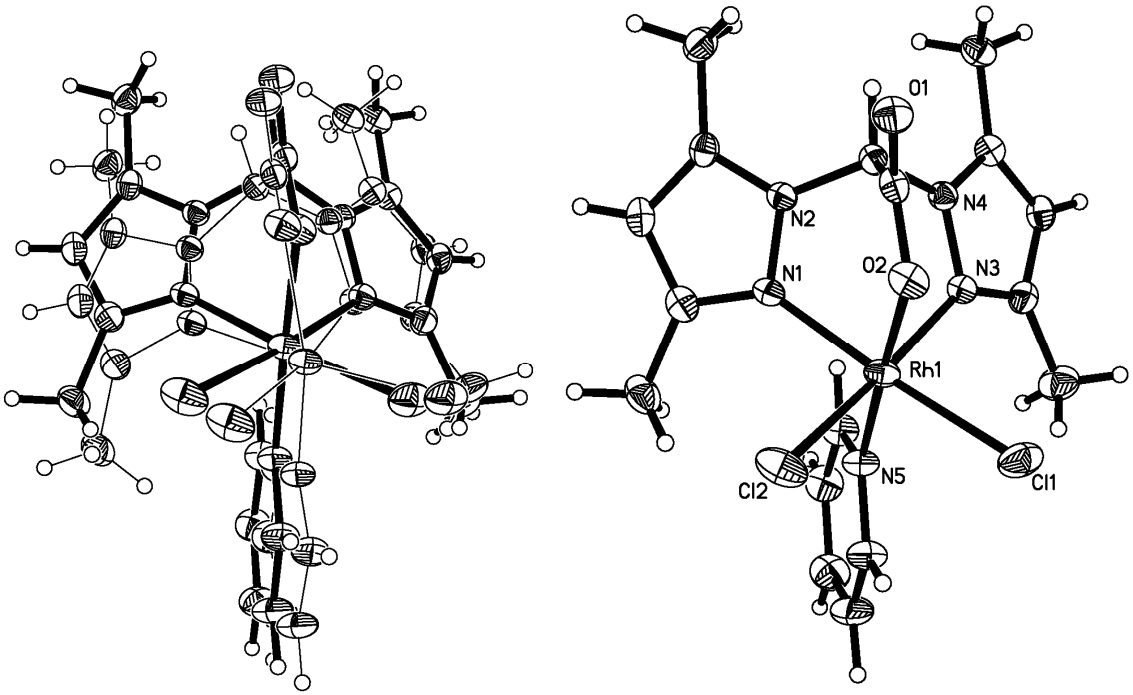


Table 1. Crystal data and structure refinement for **3a**.

Identification code	jonjlm06	
Empirical formula	C17 H20 Cl2 N5 O2 Rh	
Formula weight	500.19	
Temperature	100.0(1) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 11.8837(13) Å	α = 90°
	b = 13.5490(15) Å	β = 103.821(2)°
	c = 12.5024(14) Å	γ = 90°
Volume	1954.8(4) Å^3	
Z	4	
Density (calculated)	1.700 Mg/m^3	
Absorption coefficient	1.170 mm^{-1}	
F(000)	1008	
Crystal color, morphology	yellow, needle	
Crystal size	0.34 x 0.10 x 0.06 mm^3	
Theta range for data collection	1.76 to 37.03°	
Index ranges	$-20 \le h \le 20, -22 \le k \le 22, -21 \le l \le 19$	
Reflections collected	40055	
Independent reflections	9900 [R(int) = 0.0523]	
Observed reflections	6730	
Completeness to theta = 37.03°	99.4%	
Absorption correction	Multi-scan	
Max. and min. transmission	0.9331 and 0.6918	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	9900 / 33 / 330	
Goodness-of-fit on F^2	1.022	
Final R indices [I>2sigma(I)]	R1 = 0.0438, wR2 = 0.0981	
R indices (all data)	R1 = 0.0769, wR2 = 0.1114	
Largest diff. peak and hole	1.330 and -0.588 e.Å^{-3}	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **3a**. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Rh1	7537(1)	3738(1)	2907(1)	20(1)
Cl1	7954(1)	5348(1)	3521(1)	33(1)
Cl2	7935(1)	3151(1)	4699(1)	34(1)
O1	4019(2)	3596(2)	2315(2)	24(1)
O2	5889(5)	3988(4)	3020(4)	23(1)
N1	7058(2)	2342(1)	2305(2)	19(1)
N2	6005(2)	2281(1)	1576(2)	18(1)
C1	7459(2)	1418(2)	2451(2)	23(1)
C2	6631(2)	771(2)	1822(2)	24(1)
C3	5715(2)	1339(2)	1278(2)	21(1)
C5	5038(2)	3631(2)	2267(2)	19(1)
C4	5360(2)	3183(2)	1246(2)	18(1)
N4	6027(3)	3841(2)	723(3)	18(1)
N3	7050(4)	4231(5)	1322(4)	20(1)
C8	7461(5)	4798(4)	630(4)	22(1)
C11	4795(2)	3766(2)	-1198(2)	25(1)
C7	6705(3)	4751(2)	-414(3)	22(1)
C6	5817(3)	4128(2)	-343(2)	19(1)
C12	8572(3)	5344(4)	951(5)	34(1)
C9	8610(2)	1158(2)	3140(3)	31(1)
C10	4578(2)	1085(2)	518(2)	27(1)
N5	9208(2)	3532(2)	2836(2)	22(1)
C13	10087(2)	3829(2)	3668(2)	28(1)
C14	11229(2)	3682(2)	3641(3)	34(1)
C15	11489(3)	3200(2)	2748(3)	35(1)
C16	10581(3)	2898(2)	1899(3)	30(1)
C17	9455(2)	3081(2)	1960(3)	26(1)
Rh1'	7694(2)	4021(2)	3038(2)	20(1)
Cl1'	7916(6)	5715(5)	3372(7)	33(1)
Cl2'	8155(5)	3741(5)	4920(4)	34(1)
O1'	4313(16)	3487(18)	2480(20)	24(1)

O2'	5830(40)	3850(40)	3130(40)	23(1)
N1'	7374(14)	2555(13)	2700(15)	19(1)
N2'	6455(17)	2322(12)	1980(15)	18(1)
C1'	8034(17)	1661(15)	2969(17)	23(1)
C2'	7310(20)	986(16)	2276(19)	24(1)
C3'	6381(17)	1231(15)	1648(16)	21(1)
C5'	5260(30)	3550(20)	2450(20)	19(1)
C4'	5584(19)	3015(15)	1468(18)	18(1)
N4'	6090(30)	3700(30)	840(20)	18(1)
N3'	7120(40)	4130(50)	1370(30)	20(1)
C8'	7420(40)	4670(50)	590(30)	22(1)
C11'	4790(20)	3454(17)	-1028(19)	25(1)
C7'	6580(30)	4560(20)	-400(20)	22(1)
C6'	5740(30)	3960(30)	-221(19)	19(1)
C12'	8370(30)	5400(40)	840(50)	34(1)
C9'	9093(17)	1687(17)	3780(20)	31(1)
C10'	5513(18)	901(15)	861(19)	27(1)
N5'	9361(15)	3849(14)	2960(16)	22(1)
C13'	10218(15)	4208(18)	3750(17)	28(1)
C14'	11362(17)	4010(20)	3750(20)	34(1)
C15'	11590(20)	3500(20)	2860(20)	35(1)
C16'	10690(20)	3160(20)	2040(30)	30(1)
C17'	9550(20)	3340(20)	2110(20)	26(1)

Table 3. Bond lengths [Å] and angles [°] for **3a**.

Rh(1)-O(2)	2.025(5)	C(9)-H(9A)	0.9800
Rh(1)-N(5)	2.029(2)	C(9)-H(9B)	0.9800
Rh(1)-N(3)	2.039(4)	C(9)-H(9C)	0.9800
Rh(1)-N(1)	2.064(2)	C(10)-H(10A)	0.9800
Rh(1)-Cl(2)	2.3170(7)	C(10)-H(10B)	0.9800
Rh(1)-Cl(1)	2.3259(7)	C(10)-H(10C)	0.9800
O(1)-C(5)	1.228(3)	N(5)-C(17)	1.346(3)
O(2)-C(5)	1.299(7)	N(5)-C(13)	1.348(3)
N(1)-C(1)	1.337(3)	C(13)-C(14)	1.380(4)
N(1)-N(2)	1.363(3)	C(13)-H(13)	0.9500
N(2)-C(3)	1.352(3)	C(14)-C(15)	1.390(5)
N(2)-C(4)	1.449(3)	C(14)-H(14)	0.9500
C(1)-C(2)	1.409(4)	C(15)-C(16)	1.382(4)
C(1)-C(9)	1.475(4)	C(15)-H(15)	0.9500
C(2)-C(3)	1.374(3)	C(16)-C(17)	1.382(3)
C(2)-H(2)	0.9500	C(16)-H(16)	0.9500
C(3)-C(10)	1.494(3)	C(17)-H(17)	0.9500
C(5)-C(4)	1.543(3)	Rh(1')-N(5')	2.020(18)
C(4)-N(4)	1.449(3)	Rh(1')-N(3')	2.04(3)
C(4)-H(4)	1.0000	Rh(1')-N(1')	2.046(17)
N(4)-C(6)	1.353(3)	Rh(1')-O(2')	2.26(5)
N(4)-N(3)	1.372(3)	Rh(1')-Cl(2')	2.317(6)
N(3)-C(8)	1.335(3)	Rh(1')-Cl(1')	2.338(7)
C(8)-C(7)	1.398(4)	O(1')-C(5')	1.14(3)
C(8)-C(12)	1.483(4)	O(2')-C(5')	1.03(7)
C(11)-C(6)	1.497(3)	N(1')-N(2')	1.28(2)
C(11)-H(11A)	0.9800	N(1')-C(1')	1.44(2)
C(11)-H(11B)	0.9800	N(2')-C(4')	1.43(3)
C(11)-H(11C)	0.9800	N(2')-C(3')	1.53(3)
C(7)-C(6)	1.370(3)	C(1')-C(2')	1.41(3)
C(7)-H(7)	0.9500	C(1')-C(9')	1.41(3)
C(12)-H(12A)	0.9800	C(2')-C(3')	1.24(3)
C(12)-H(12B)	0.9800	C(2')-H(2')	0.9500
C(12)-H(12C)	0.9800	C(3')-C(10')	1.32(3)

C(5')-C(4')	1.560(18)	N(5)-Rh(1)-N(3)	93.2(2)
C(4')-N(4')	1.440(16)	O(2)-Rh(1)-N(1)	89.52(17)
C(4')-H(4')	1.0000	N(5)-Rh(1)-N(1)	92.60(8)
N(4')-C(6')	1.333(16)	N(3)-Rh(1)-N(1)	87.47(18)
N(4')-N(3')	1.377(16)	O(2)-Rh(1)-Cl(2)	88.15(13)
N(3')-C(8')	1.333(17)	N(5)-Rh(1)-Cl(2)	91.12(7)
C(8')-C(7')	1.401(17)	N(3)-Rh(1)-Cl(2)	175.41(13)
C(8')-C(12')	1.477(17)	N(1)-Rh(1)-Cl(2)	90.74(6)
C(11')-C(6')	1.495(16)	O(2)-Rh(1)-Cl(1)	87.35(16)
C(11')-H(11D)	0.9800	N(5)-Rh(1)-Cl(1)	90.55(6)
C(11')-H(11E)	0.9800	N(3)-Rh(1)-Cl(1)	90.12(18)
C(11')-H(11F)	0.9800	N(1)-Rh(1)-Cl(1)	176.13(6)
C(7')-C(6')	1.338(16)	Cl(2)-Rh(1)-Cl(1)	91.43(3)
C(7')-H(7')	0.9500	C(5)-O(2)-Rh(1)	119.1(4)
C(12')-H(12D)	0.9800	C(1)-N(1)-N(2)	105.9(2)
C(12')-H(12E)	0.9800	C(1)-N(1)-Rh(1)	138.62(19)
C(12')-H(12F)	0.9800	N(2)-N(1)-Rh(1)	115.37(14)
C(9')-H(9D)	0.9800	C(3)-N(2)-N(1)	112.00(19)
C(9')-H(9E)	0.9800	C(3)-N(2)-C(4)	129.4(2)
C(9')-H(9F)	0.9800	N(1)-N(2)-C(4)	118.57(18)
C(10')-H(10D)	0.9800	N(1)-C(1)-C(2)	109.2(2)
C(10')-H(10E)	0.9800	N(1)-C(1)-C(9)	123.6(2)
C(10')-H(10F)	0.9800	C(2)-C(1)-C(9)	127.1(2)
N(5')-C(13')	1.330(16)	C(3)-C(2)-C(1)	107.0(2)
N(5')-C(17')	1.334(16)	C(3)-C(2)-H(2)	126.5
C(13')-C(14')	1.385(17)	C(1)-C(2)-H(2)	126.5
C(13')-H(13')	0.9500	N(2)-C(3)-C(2)	105.9(2)
C(14')-C(15')	1.392(17)	N(2)-C(3)-C(10)	121.7(2)
C(14')-H(14')	0.9500	C(2)-C(3)-C(10)	132.5(2)
C(15')-C(16')	1.377(17)	O(1)-C(5)-O(2)	125.9(3)
C(15')-H(15')	0.9500	O(1)-C(5)-C(4)	117.9(3)
C(16')-C(17')	1.389(17)	O(2)-C(5)-C(4)	116.2(3)
C(16')-H(16')	0.9500	N(2)-C(4)-N(4)	109.8(2)
C(17')-H(17')	0.9500	N(2)-C(4)-C(5)	108.38(19)
O(2)-Rh(1)-N(5)	177.76(19)	N(4)-C(4)-C(5)	113.7(2)
O(2)-Rh(1)-N(3)	87.6(2)	N(2)-C(4)-H(4)	108.3

N(4)-C(4)-H(4)	108.3	N(5')-Rh(1')-N(1')	90.8(7)
C(5)-C(4)-H(4)	108.3	N(3')-Rh(1')-N(1')	82.0(18)
C(6)-N(4)-N(3)	111.0(2)	N(5')-Rh(1')-O(2')	167.5(14)
C(6)-N(4)-C(4)	129.0(2)	N(3')-Rh(1')-O(2')	88.3(19)
N(3)-N(4)-C(4)	120.0(2)	N(1')-Rh(1')-O(2')	77.1(14)
C(8)-N(3)-N(4)	106.3(3)	N(5')-Rh(1')-Cl(2')	91.9(6)
C(8)-N(3)-Rh(1)	139.1(3)	N(3')-Rh(1')-Cl(2')	172.5(8)
N(4)-N(3)-Rh(1)	114.4(2)	N(1')-Rh(1')-Cl(2')	92.2(5)
N(3)-C(8)-C(7)	109.0(3)	O(2')-Rh(1')-Cl(2')	85.8(13)
N(3)-C(8)-C(12)	123.5(3)	N(5')-Rh(1')-Cl(1')	93.0(5)
C(7)-C(8)-C(12)	127.5(3)	N(3')-Rh(1')-Cl(1')	96.1(18)
C(6)-C(7)-C(8)	107.5(2)	N(1')-Rh(1')-Cl(1')	175.9(5)
C(6)-C(7)-H(7)	126.3	O(2')-Rh(1')-Cl(1')	99.3(13)
C(8)-C(7)-H(7)	126.3	Cl(2')-Rh(1')-Cl(1')	89.4(3)
N(4)-C(6)-C(7)	106.1(2)	C(5')-O(2')-Rh(1')	117(4)
N(4)-C(6)-C(11)	122.5(2)	N(2')-N(1')-C(1')	106.4(17)
C(7)-C(6)-C(11)	131.4(2)	N(2')-N(1')-Rh(1')	118.0(12)
C(17)-N(5)-C(13)	119.0(2)	C(1')-N(1')-Rh(1')	134.8(14)
C(17)-N(5)-Rh(1)	120.27(17)	N(1')-N(2')-C(4')	124.0(17)
C(13)-N(5)-Rh(1)	120.76(18)	N(1')-N(2')-C(3')	114.4(16)
N(5)-C(13)-C(14)	121.5(3)	C(4')-N(2')-C(3')	121.5(19)
N(5)-C(13)-H(13)	119.2	C(2')-C(1')-C(9')	140.1(19)
C(14)-C(13)-H(13)	119.2	C(2')-C(1')-N(1')	100.6(17)
C(13)-C(14)-C(15)	119.7(3)	C(9')-C(1')-N(1')	119.2(19)
C(13)-C(14)-H(14)	120.1	C(3')-C(2')-C(1')	123(2)
C(15)-C(14)-H(14)	120.1	C(3')-C(2')-H(2')	118.7
C(16)-C(15)-C(14)	118.3(3)	C(1')-C(2')-H(2')	118.7
C(16)-C(15)-H(15)	120.8	C(2')-C(3')-C(10')	143(2)
C(14)-C(15)-H(15)	120.8	C(2')-C(3')-N(2')	95.8(18)
C(17)-C(16)-C(15)	119.5(3)	C(10')-C(3')-N(2')	121(2)
C(17)-C(16)-H(16)	120.2	O(2')-C(5')-O(1')	118(4)
C(15)-C(16)-H(16)	120.2	O(2')-C(5')-C(4')	126(4)
N(5)-C(17)-C(16)	121.9(3)	O(1')-C(5')-C(4')	115(3)
N(5)-C(17)-H(17)	119.0	N(2')-C(4')-N(4')	109(2)
C(16)-C(17)-H(17)	119.0	N(2')-C(4')-C(5')	104.1(18)
N(5')-Rh(1')-N(3')	92.9(18)	N(4')-C(4')-C(5')	110(2)

N(2')-C(4')-H(4')	111.2	C(1')-C(9')-H(9D)	109.5
N(4')-C(4')-H(4')	111.2	C(1')-C(9')-H(9E)	109.5
C(5')-C(4')-H(4')	111.2	H(9D)-C(9')-H(9E)	109.5
C(6')-N(4')-N(3')	113.1(15)	C(1')-C(9')-H(9F)	109.5
C(6')-N(4')-C(4')	130.1(19)	H(9D)-C(9')-H(9F)	109.5
N(3')-N(4')-C(4')	116.7(18)	H(9E)-C(9')-H(9F)	109.5
C(8')-N(3')-N(4')	104.1(16)	C(3')-C(10')-H(10D)	109.5
C(8')-N(3')-Rh(1')	135(2)	C(3')-C(10')-H(10E)	109.5
N(4')-N(3')-Rh(1')	120(2)	H(10D)-C(10')-H(10E)	109.5
N(3')-C(8')-C(7')	108.9(16)	C(3')-C(10')-H(10F)	109.5
N(3')-C(8')-C(12')	122(3)	H(10D)-C(10')-H(10F)	109.5
C(7')-C(8')-C(12')	128(3)	H(10E)-C(10')-H(10F)	109.5
C(6')-C(11')-H(11D)	109.5	C(13')-N(5')-C(17')	122.3(17)
C(6')-C(11')-H(11E)	109.5	C(13')-N(5')-Rh(1')	120.6(13)
H(11D)-C(11')-H(11E)	109.5	C(17')-N(5')-Rh(1')	117.1(13)
C(6')-C(11')-H(11F)	109.5	N(5')-C(13')-C(14')	120.4(17)
H(11D)-C(11')-H(11F)	109.5	N(5')-C(13')-H(13')	119.8
H(11E)-C(11')-H(11F)	109.5	C(14')-C(13')-H(13')	119.8
C(6')-C(7')-C(8')	108.7(16)	C(13')-C(14')-C(15')	118.6(18)
C(6')-C(7')-H(7')	125.6	C(13')-C(14')-H(14')	120.7
C(8')-C(7')-H(7')	125.6	C(15')-C(14')-H(14')	120.7
N(4')-C(6')-C(7')	105.2(15)	C(16')-C(15')-C(14')	119.5(18)
N(4')-C(6')-C(11')	123.5(18)	C(16')-C(15')-H(15')	120.3
C(7')-C(6')-C(11')	129.6(19)	C(14')-C(15')-H(15')	120.3
C(8')-C(12')-H(12D)	109.5	C(15')-C(16')-C(17')	119.5(19)
C(8')-C(12')-H(12E)	109.5	C(15')-C(16')-H(16')	120.3
H(12D)-C(12')-H(12E)	109.5	C(17')-C(16')-H(16')	120.3
C(8')-C(12')-H(12F)	109.5	N(5')-C(17')-C(16')	119.7(18)
H(12D)-C(12')-H(12F)	109.5	N(5')-C(17')-H(17')	120.2
H(12E)-C(12')-H(12F)	109.5	C(16')-C(17')-H(17')	120.2

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **3a**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U_{11} + ... + 2\ h\ k\ a^*\ b^*\ U_{12}\ ]$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Rh1	17(1)	27(1)	14(1)	-3(1)	3(1)	1(1)
Cl1	28(1)	34(1)	35(1)	-16(1)	3(1)	-1(1)
Cl2	31(1)	56(1)	15(1)	2(1)	5(1)	7(1)
O1	17(1)	29(1)	30(1)	1(1)	11(1)	1(1)
O2	21(1)	32(2)	19(2)	0(1)	9(1)	4(1)
N1	16(1)	23(1)	17(1)	1(1)	2(1)	1(1)
N2	17(1)	20(1)	17(1)	1(1)	3(1)	1(1)
C1	22(1)	27(1)	24(1)	5(1)	10(1)	3(1)
C2	29(1)	19(1)	27(1)	4(1)	11(1)	3(1)
C3	25(1)	19(1)	20(1)	1(1)	8(1)	-1(1)
C5	20(1)	18(1)	20(1)	4(1)	7(1)	4(1)
C4	17(1)	19(1)	20(1)	3(1)	5(1)	2(1)
N4	18(1)	19(1)	17(1)	1(1)	4(1)	0(1)
N3	19(1)	20(2)	19(1)	0(1)	3(1)	1(1)
C8	23(1)	20(2)	25(1)	0(1)	7(1)	-1(1)
C11	25(1)	30(1)	18(1)	-2(1)	3(1)	3(1)
C7	26(1)	21(2)	22(1)	4(1)	11(1)	1(1)
C6	21(1)	21(2)	17(1)	-1(1)	7(1)	2(1)
C12	25(2)	39(1)	37(2)	1(1)	9(2)	-9(2)
C9	23(1)	34(1)	37(2)	10(1)	7(1)	7(1)
C10	29(1)	24(1)	26(1)	-1(1)	4(1)	-5(1)
N5	17(1)	30(1)	19(1)	-3(1)	2(1)	-2(1)
C13	22(1)	35(1)	23(1)	-5(1)	1(1)	-1(1)
C14	19(1)	44(2)	35(2)	-2(1)	-1(1)	-5(1)
C15	20(1)	44(2)	40(2)	6(2)	10(1)	-1(1)
C16	26(1)	37(2)	31(2)	0(1)	13(1)	1(1)
C17	24(1)	33(2)	23(1)	-2(1)	7(1)	0(1)
Rh1'	17(1)	27(1)	14(1)	-3(1)	3(1)	1(1)
Cl1'	28(1)	34(1)	35(1)	-16(1)	3(1)	-1(1)
Cl2'	31(1)	56(1)	15(1)	2(1)	5(1)	7(1)
O1'	17(1)	29(1)	30(1)	1(1)	11(1)	1(1)

O2'	21(1)	32(2)	19(2)	0(1)	9(1)	4(1)
N1'	16(1)	23(1)	17(1)	1(1)	2(1)	1(1)
N2'	17(1)	20(1)	17(1)	1(1)	3(1)	1(1)
C1'	22(1)	27(1)	24(1)	5(1)	10(1)	3(1)
C2'	29(1)	19(1)	27(1)	4(1)	11(1)	3(1)
C3'	25(1)	19(1)	20(1)	1(1)	8(1)	-1(1)
C5'	20(1)	18(1)	20(1)	4(1)	7(1)	4(1)
C4'	17(1)	19(1)	20(1)	3(1)	5(1)	2(1)
N4'	18(1)	19(1)	17(1)	1(1)	4(1)	0(1)
N3'	19(1)	20(2)	19(1)	0(1)	3(1)	1(1)
C8'	23(1)	20(2)	25(1)	0(1)	7(1)	-1(1)
C11'	25(1)	30(1)	18(1)	-2(1)	3(1)	3(1)
C7'	26(1)	21(2)	22(1)	4(1)	11(1)	1(1)
C6'	21(1)	21(2)	17(1)	-1(1)	7(1)	2(1)
C12'	25(2)	39(1)	37(2)	1(1)	9(2)	-9(2)
C9'	23(1)	34(1)	37(2)	10(1)	7(1)	7(1)
C10'	29(1)	24(1)	26(1)	-1(1)	4(1)	-5(1)
N5'	17(1)	30(1)	19(1)	-3(1)	2(1)	-2(1)
C13'	22(1)	35(1)	23(1)	-5(1)	1(1)	-1(1)
C14'	19(1)	44(2)	35(2)	-2(1)	-1(1)	-5(1)
C15'	20(1)	44(2)	40(2)	6(2)	10(1)	-1(1)
C16'	26(1)	37(2)	31(2)	0(1)	13(1)	1(1)
C17'	24(1)	33(2)	23(1)	-2(1)	7(1)	0(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for **3a**.

	x	y	z	U(eq)
H2	6695	73	1781	29
H4	4627	3010	696	22
H11A	4090	3846	-930	37
H11B	4902	3067	-1348	37
H11C	4723	4148	-1877	37
H7	6790	5089	-1056	26
H12A	8747	5488	1742	50
H12B	8510	5963	537	50
H12C	9195	4940	788	50
H9A	8812	1611	3768	47
H9B	9190	1210	2701	47
H9C	8595	480	3409	47
H10A	3963	1480	708	41
H10B	4415	382	591	41
H10C	4611	1226	-243	41
H13	9917	4148	4288	33
H14	11836	3908	4230	41
H15	12269	3081	2722	41
H16	10731	2567	1278	36
H17	8836	2882	1367	31
H2'	7538	314	2291	29
H4'	4897	2674	994	22
H11D	4370	3015	-632	37
H11E	5113	3066	-1543	37
H11F	4250	3948	-1436	37
H7'	6592	4856	-1085	26
H12D	8820	5307	1601	50
H12E	8033	6070	769	50
H12F	8870	5326	333	50
H9D	9434	1024	3870	47
H9E	8952	1911	4479	47

H9F	9629	2143	3545	47
H10D	5602	190	768	41
H10E	5513	1241	169	41
H10F	4779	1029	1063	41
H13'	10046	4601	4320	33
H14'	11976	4229	4334	41
H15'	12370	3381	2820	41
H16'	10832	2802	1432	36
H17'	8922	3099	1555	31

Table 6. Torsion angles [°] for **3a**.

N5-Rh1-O2-C5	161(3)	N1-N2-C4-N4	60.1(3)
N3-Rh1-O2-C5	50.7(3)	C3-N2-C4-C5	114.4(3)
N1-Rh1-O2-C5	-36.8(3)	N1-N2-C4-C5	-64.6(3)
Cl2-Rh1-O2-C5	-127.5(3)	O1-C5-C4-N2	-108.6(3)
Cl1-Rh1-O2-C5	140.9(3)	O2-C5-C4-N2	69.4(3)
O2-Rh1-N1-C1	-135.6(3)	O1-C5-C4-N4	129.1(3)
N5-Rh1-N1-C1	43.7(3)	O2-C5-C4-N4	-53.0(4)
N3-Rh1-N1-C1	136.8(3)	N2-C4-N4-C6	111.8(4)
Cl2-Rh1-N1-C1	-47.4(2)	C5-C4-N4-C6	-126.7(4)
Cl1-Rh1-N1-C1	-171.7(7)	N2-C4-N4-N3	-65.4(5)
O2-Rh1-N1-N2	40.34(19)	C5-C4-N4-N3	56.2(5)
N5-Rh1-N1-N2	-140.36(16)	C6-N4-N3-C8	2.3(8)
N3-Rh1-N1-N2	-47.3(2)	C4-N4-N3-C8	179.9(5)
Cl2-Rh1-N1-N2	128.49(14)	C6-N4-N3-Rh1	-174.2(4)
Cl1-Rh1-N1-N2	4.3(10)	C4-N4-N3-Rh1	3.4(7)
C1-N1-N2-C3	1.6(2)	O2-Rh1-N3-C8	138.6(10)
Rh1-N1-N2-C3	-175.62(15)	N5-Rh1-N3-C8	-39.4(10)
C1-N1-N2-C4	-179.3(2)	N1-Rh1-N3-C8	-131.8(10)
Rh1-N1-N2-C4	3.5(2)	Cl2-Rh1-N3-C8	161(2)
N2-N1-C1-C2	-1.3(3)	Cl1-Rh1-N3-C8	51.2(10)
Rh1-N1-C1-C2	174.90(18)	O2-Rh1-N3-N4	-46.5(5)
N2-N1-C1-C9	176.8(2)	N5-Rh1-N3-N4	135.6(5)
Rh1-N1-C1-C9	-7.1(4)	N1-Rh1-N3-N4	43.1(5)
N1-C1-C2-C3	0.6(3)	Cl2-Rh1-N3-N4	-24(3)
C9-C1-C2-C3	-177.4(2)	Cl1-Rh1-N3-N4	-133.9(5)
N1-N2-C3-C2	-1.2(3)	N4-N3-C8-C7	-1.1(8)
C4-N2-C3-C2	179.8(2)	Rh1-N3-C8-C7	174.1(7)
N1-N2-C3-C10	177.7(2)	N4-N3-C8-C12	-178.9(6)
C4-N2-C3-C10	-1.3(4)	Rh1-N3-C8-C12	-3.7(14)
C1-C2-C3-N2	0.4(3)	N3-C8-C7-C6	-0.5(7)
C1-C2-C3-C10	-178.4(2)	C12-C8-C7-C6	177.2(6)
Rh1-O2-C5-O1	168.4(2)	N3-N4-C6-C7	-2.6(6)
Rh1-O2-C5-C4	-9.4(4)	C4-N4-C6-C7	-179.9(4)
C3-N2-C4-N4	-120.9(3)	N3-N4-C6-C11	176.4(5)

C4-N4-C6-C11	-0.9(7)	C1'-N1'-N2'-C4'	-177.7(17)
C8-C7-C6-N4	1.8(5)	Rh1'-N1'-N2'-C4'	-6(2)
C8-C7-C6-C11	-177.0(5)	C1'-N1'-N2'-C3'	-1(2)
O2-Rh1-N5-C17	-156(3)	Rh1'-N1'-N2'-C3'	170.8(11)
N3-Rh1-N5-C17	-45.6(3)	N2'-N1'-C1'-C2'	0(2)
N1-Rh1-N5-C17	42.0(2)	Rh1'-N1'-C1'-C2'	-169.4(15)
Cl2-Rh1-N5-C17	132.8(2)	N2'-N1'-C1'-C9'	-178.2(18)
Cl1-Rh1-N5-C17	-135.8(2)	Rh1'-N1'-C1'-C9'	12(3)
O2-Rh1-N5-C13	25(3)	C9'-C1'-C2'-C3'	179(3)
N3-Rh1-N5-C13	135.5(3)	N1'-C1'-C2'-C3'	1(3)
N1-Rh1-N5-C13	-136.9(2)	C1'-C2'-C3'-C10'	175(3)
Cl2-Rh1-N5-C13	-46.1(2)	C1'-C2'-C3'-N2'	-1(2)
Cl1-Rh1-N5-C13	45.3(2)	N1'-N2'-C3'-C2'	1(2)
C17-N5-C13-C14	0.0(4)	C4'-N2'-C3'-C2'	178.0(18)
Rh1-N5-C13-C14	179.0(2)	N1'-N2'-C3'-C10'	-175.7(19)
N5-C13-C14-C15	-1.2(4)	C4'-N2'-C3'-C10'	1(3)
C13-C14-C15-C16	1.2(5)	Rh1'-O2'-C5'-O1'	-178(3)
C14-C15-C16-C17	0.0(5)	Rh1'-O2'-C5'-C4'	13(6)
C13-N5-C17-C16	1.2(4)	N1'-N2'-C4'-N4'	64(3)
Rh1-N5-C17-C16	-177.8(2)	C3'-N2'-C4'-N4'	-113(2)
C15-C16-C17-N5	-1.2(5)	N1'-N2'-C4'-C5'	-54(3)
N5'-Rh1'-O2'-C5'	-69(8)	C3'-N2'-C4'-C5'	130(2)
N3'-Rh1'-O2'-C5'	27(4)	O2'-C5'-C4'-N2'	48(5)
N1'-Rh1'-O2'-C5'	-55(4)	O1'-C5'-C4'-N2'	-120(3)
Cl2'-Rh1'-O2'-C5'	-148(4)	O2'-C5'-C4'-N4'	-68(5)
Cl1'-Rh1'-O2'-C5'	123(4)	O1'-C5'-C4'-N4'	123(3)
N5'-Rh1'-N1'-N2'	-134.1(15)	N2'-C4'-N4'-C6'	126(4)
N3'-Rh1'-N1'-N2'	-41(2)	C5'-C4'-N4'-C6'	-121(4)
O2'-Rh1'-N1'-N2'	48.8(18)	N2'-C4'-N4'-N3'	-49(5)
Cl2'-Rh1'-N1'-N2'	134.0(14)	C5'-C4'-N4'-N3'	64(5)
Cl1'-Rh1'-N1'-N2'	22(8)	C6'-N4'-N3'-C8'	0(8)
N5'-Rh1'-N1'-C1'	34.9(19)	C4'-N4'-N3'-C8'	176(5)
N3'-Rh1'-N1'-C1'	128(3)	C6'-N4'-N3'-Rh1'	171(4)
O2'-Rh1'-N1'-C1'	-142(2)	C4'-N4'-N3'-Rh1'	-13(7)
Cl2'-Rh1'-N1'-C1'	-57.0(18)	N5'-Rh1'-N3'-C8'	-51(9)
Cl1'-Rh1'-N1'-C1'	-169(6)	N1'-Rh1'-N3'-C8'	-141(9)

O2'-Rh1'-N3'-C8'	142(9)	C8'-C7'-C6'-C11'	-166(5)
Cl2'-Rh1'-N3'-C8'	180(11)	N3'-Rh1'-N5'-C13'	146(3)
Cl1'-Rh1'-N3'-C8'	43(9)	N1'-Rh1'-N5'-C13'	-132(2)
N5'-Rh1'-N3'-N4'	142(6)	O2'-Rh1'-N5'-C13'	-119(7)
N1'-Rh1'-N3'-N4'	51(5)	Cl2'-Rh1'-N5'-C13'	-39(2)
O2'-Rh1'-N3'-N4'	-26(6)	Cl1'-Rh1'-N5'-C13'	50(2)
Cl2'-Rh1'-N3'-N4'	12(23)	N3'-Rh1'-N5'-C17'	-36(3)
Cl1'-Rh1'-N3'-N4'	-125(5)	N1'-Rh1'-N5'-C17'	46(2)
N4'-N3'-C8'-C7'	0(8)	O2'-Rh1'-N5'-C17'	59(7)
Rh1'-N3'-C8'-C7'	-169(6)	Cl2'-Rh1'-N5'-C17'	138(2)
N4'-N3'-C8'-C12'	168(6)	Cl1'-Rh1'-N5'-C17'	-132(2)
Rh1'-N3'-C8'-C12'	-1(13)	C17'-N5'-C13'-C14'	-4(4)
N3'-C8'-C7'-C6'	1(8)	Rh1'-N5'-C13'-C14'	174(2)
C12'-C8'-C7'-C6'	-166(6)	N5'-C13'-C14'-C15'	5(4)
N3'-N4'-C6'-C7'	1(6)	C13'-C14'-C15'-C16'	-3(5)
C4'-N4'-C6'-C7'	-174(4)	C14'-C15'-C16'-C17'	0(5)
N3'-N4'-C6'-C11'	167(5)	C13'-N5'-C17'-C16'	1(5)
C4'-N4'-C6'-C11'	-8(7)	Rh1'-N5'-C17'-C16'	-177(3)
C8'-C7'-C6'-N4'	-1(6)	C15'-C16'-C17'-N5'	1(5)