Electronic Supplementary Information for Dalton Transactions


# A Novel Heteroleptic Paddlewheel Diruthenium Bicyclic Guanidinate Complex: Synthesis, Structure, and Scope

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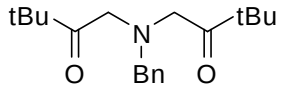
**Contents**

**Procedure for the synthesis of TBO**

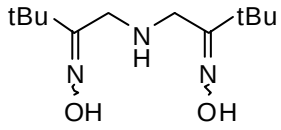
**(1,1'-(benzylazanediyl)bis(3,3-dimethylbutan-2-one)), 1.** [1]




Benzylamine (545 ml, 5 mmol, 1 equiv.) and 1-bromo-3,3-dimethylbutan-2-one (990mg, 5.5 mmol, 1.1 equiv.) were dissolved in 20 ml acetonitrile. Potassium carbonate (1.4 g, 10 mmol, 2.0 equiv.) was added and then brought to reflux for 6 hours. Then the mixture was filtered and the washed twice with MeCN. Removal of the solvent gave crude product that was further purified by column chromatography with gradient elution (hexane/EA; 50/1 to 20/1). After removing the solvent *in vacuo*, product **(**1.23 g) was obtained as colorless oil.

(**1**) colorless oil; 81% yield; $^{1}$H NMR (300 MHz, $CDCl_3$, ppm): δ = 7.35-7.19(m, 5H), 3.83 (s, 2H), 3.76 (s, 4H), 1.04 (s, 18H); $^{13}$C NMR (75 MHz, $CDCl_3$, ppm): δ = 214.5, 138.8, 128.9, 128.2, 127.2, 77.4, 77.0, 76.6, 57.7, 56.1, 43.3, 26.3; IR (film): 3022, 1217, 760 $cm^{-1}$; LRMS (ESI) m/z 326.2 ($M+Na^+$); HRMS (ESI) m/z 326.2102 ($M+Na^+$), calc. for $C_{19}H_{29}O_2N_1Na_1$ 326.2091.

**(1-(2-(hydroxyimino)-3,3-dimethylbutylamino)-3,3-dimethylbutan-2-one oxime), 2.** [2]




A flame dried round bottom flask under nitrogen, was charged with Pd/C (424mg, 0.4mmol, 0.1equiv.), MeOH, and **1** (1.21g, 4mmol, 1equiv.). $H_2$ was bubbled into the reaction mixture for 10 minutes, sealed and stirred to react overnight. After the complete consumption of starting material, the suspension was filtered and the solvent removed to afford the oxime as colorless oil. The crude oil was used for the next step without further purification.

Hydroxylamine hydrochloride (1.4g, 20mmol, 5 equiv.), potassium hydroxide (1.12g, 20mmol, 5 equiv.) and debenzylated **2** (850mg, 4mmol, 1equiv.) were dissolved in EtOH. The solution was brought to reflux for 2 hours. Water was added after the subsequent removal of solvent and extracted with EA thrice. The combined organic layer was dried over sodium sulfate and solvent

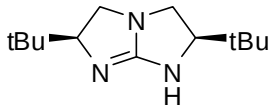
[1] H. Orsolya, C. Marco, Q. Silvio, S. Ian, P. Gianluca, *Adv. Synth. Catal.*, 2005, **347**, 677 –688.

[2] Y. K. Joon, L. Tom, *Org. Lett.*, 2005, **7**, 4391-4393.

was removed. The crude was purified with column chromatography with gradient elution (hexane/EA; 8/1 to 2/1)to yield 855mg of product as white solid.

(**2**) white solid; 88% yield; $^{1}$H NMR (300 MHz, $CDCl_3$, ppm): δ = 3.65(s, 2H), 3.51 (s, 1H), 1.17 (s, 9H), 1.15 (s, 9H); $^{13}$C NMR (75 MHz, $CDCl_3$, ppm): δ = 162.9, 44.5, 37.1, 27.4; IR (film): 3022, 1217, 760 cm$^{-1}$; LRMS (ESI) m/z 244.1 ($M+H^+$); HRMS (ESI) m/z 244.2025 ($M+H^+$), calc. for $C_{12}H_{26}O_2N_3$ 244.2020.

**(*cis*-3,7-di-*tert*-butyl)-1,4,6-triazabicyclo[3.3.0]oct-4-ene, 3.** [3]

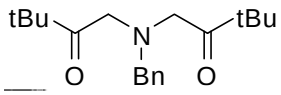


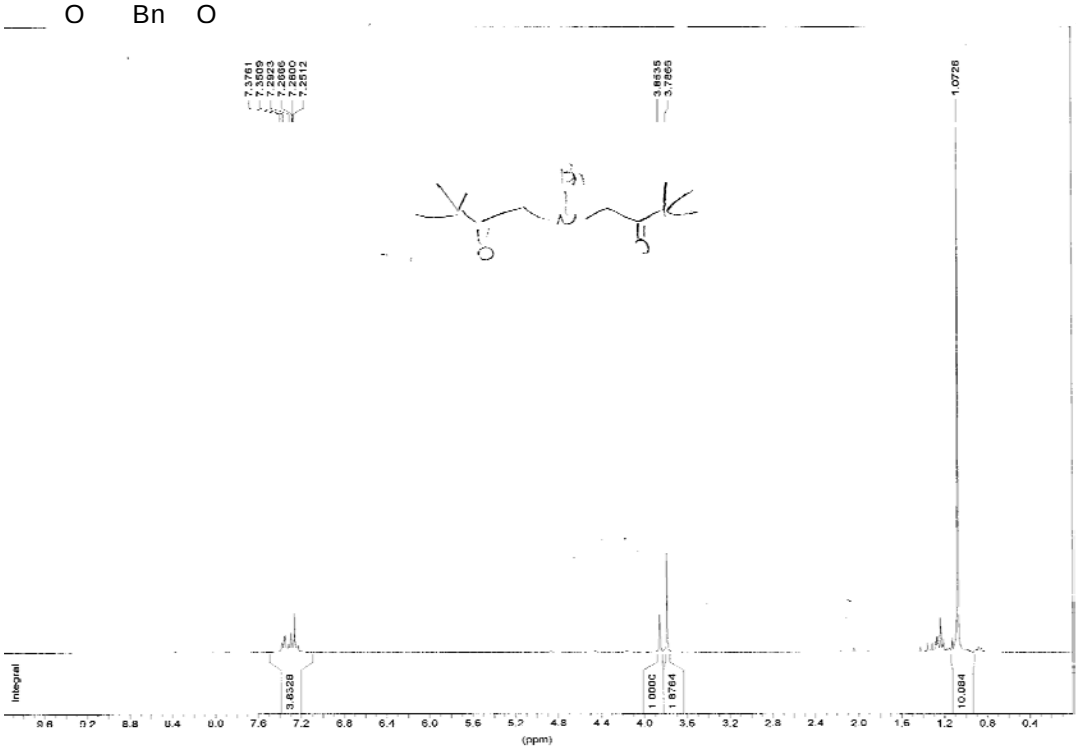

**2** (972mg, 4 mmol, 1 equiv.) was added slowly at -10$^{o}$C to a suspension of $LiAlH_4$ (605mg, 16 mmol, 4 equiv.) in 30 ml THF. The reaction mixture was heated to reflux overnight, then cooled to 0$^{o}$C and quenched by sequential addition of water (3 ml) and 10% aqueous NaOH (3 ml). The mixture was then stirred at ambient temperature for 30 min. Filtration of the mixture and drying of the filtrate over over $Na_2SO_4$ and removal of solvent to yield triamine as yellow oil. The triamine was used in the next step without further purification.

The crude free triamine (500 mg, 2.6 mmol) was dissolved in nitromethane (10 ml). Dimethyl trithiocarbonate (360 µl, 3.3 mmol, 1.25 equiv.) in nitromethane (1 ml) was added to the mixture slowly, followed by refluxing at 110 $^{o}$C for 2 h. The mixture was then cooled to room temperature. Acetic acid (610 µl, 10.6 mmol, 4 equiv.) and MeI (490 µl, 5.3 mmol, 2 equiv.) were added. The mixture was refluxed again at 110 $^{o}$C for 3 h, then left stirring at room temperature overnight. $CH_2Cl_2$ (10 ml) was added to dilute the reaction mixture and the solvent was removed under reduced pressure. $CH_2Cl_2$ (10 ml) was added and the solution was loaded onto a plug of silica gel. It was eluted with copious amount of $CH_2Cl_2$ to flush out a dark colored portion of impurities and then $CH_2Cl_2$/MeOH (19:1) was used to recover the product, which was assumed to be a HI salt. Acid-base workup gave the free guanidine as 1:1 *cis-trans* mixture (a pale yellow solid, 310 mg, 61 % yield calculated from guanidine). **3** (88mg) was obtained as pure *cis* isomer from recrystallization wth diethyl ether.

[3] W.-P. Ye, D.-S. Leow, S.L.M. Goh, C.-T. Tan, C.-H. Chian, C.-H. Tan, *Tetrahedron Letters,* 2006,**47**,1007–1010.

(**3**) off-white solid; $^{1}$H NMR (300 MHz, $CDCl_3$, ppm): δ = 4.09(s, 2H), 3. 94-3. 88 (t, 2H), 3.25-3.19 (s, 2H), 2.84-2.79 (t, 2H); $^{13}$C NMR (75 MHz, $CDCl_3$, ppm): δ = 167.6, 73.5, 49.3, 33.4, 25.8; IR (film): 3022, 1217, 760 $cm^{-1}$; LRMS (ESI) m/z 224.3 ($M+H^+$); HRMS (ESI) m/z 224.2115 ($M+H^+$), calc. for $C_{13}H_{26}N_3$ 224.2121.

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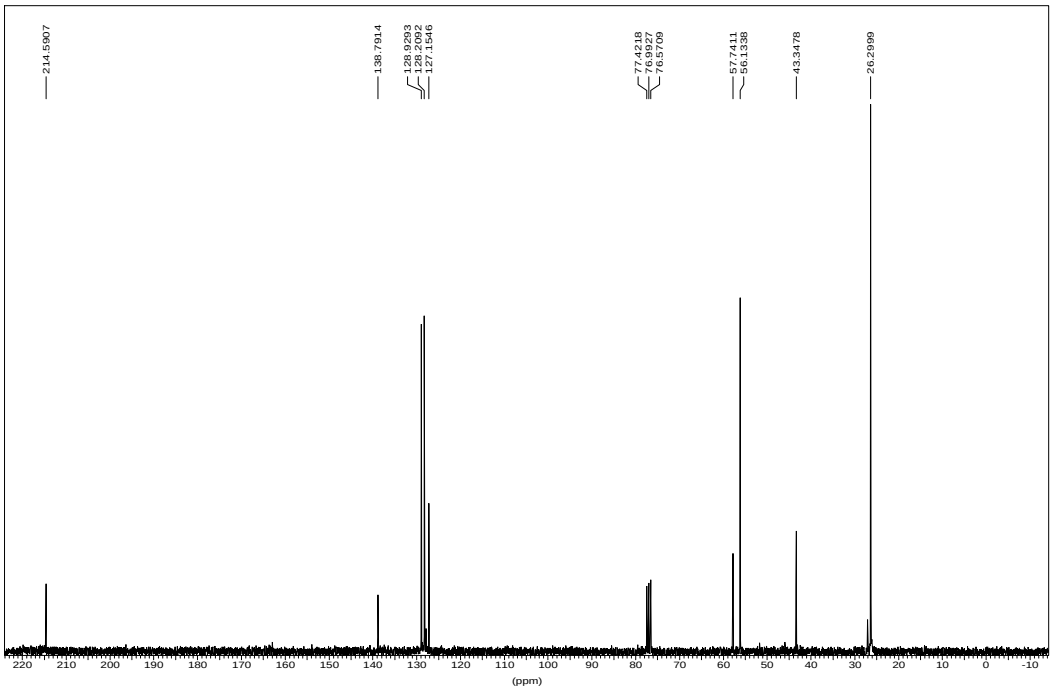

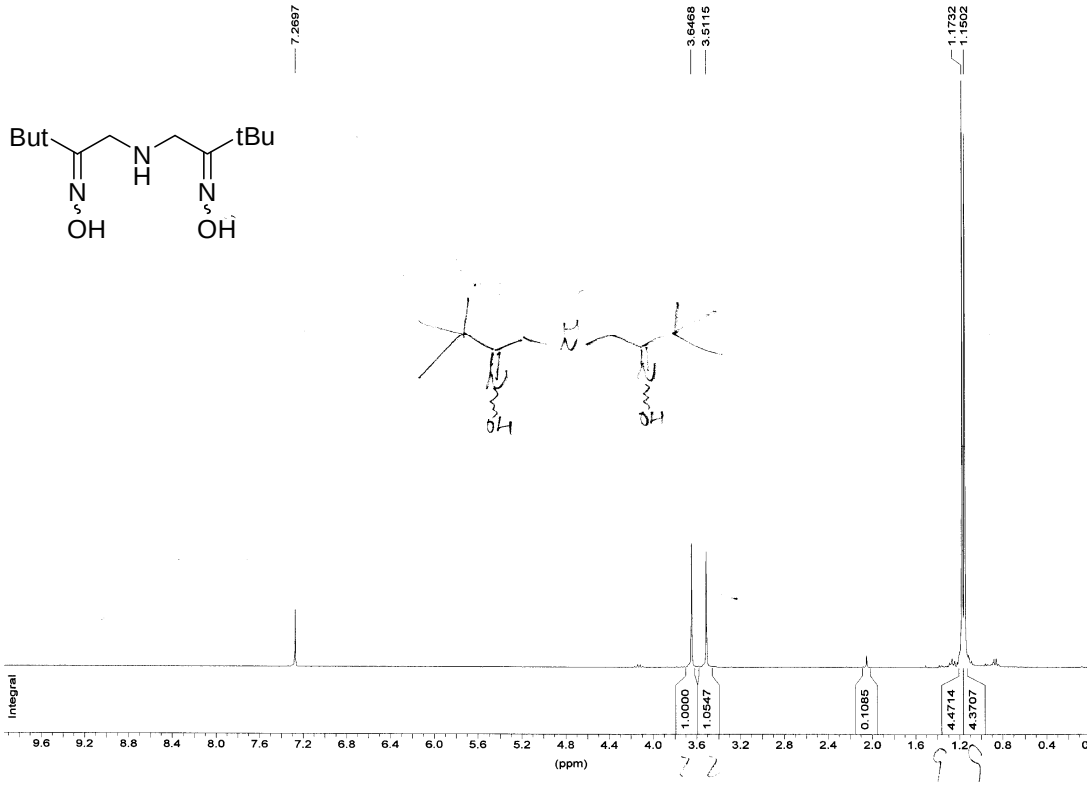


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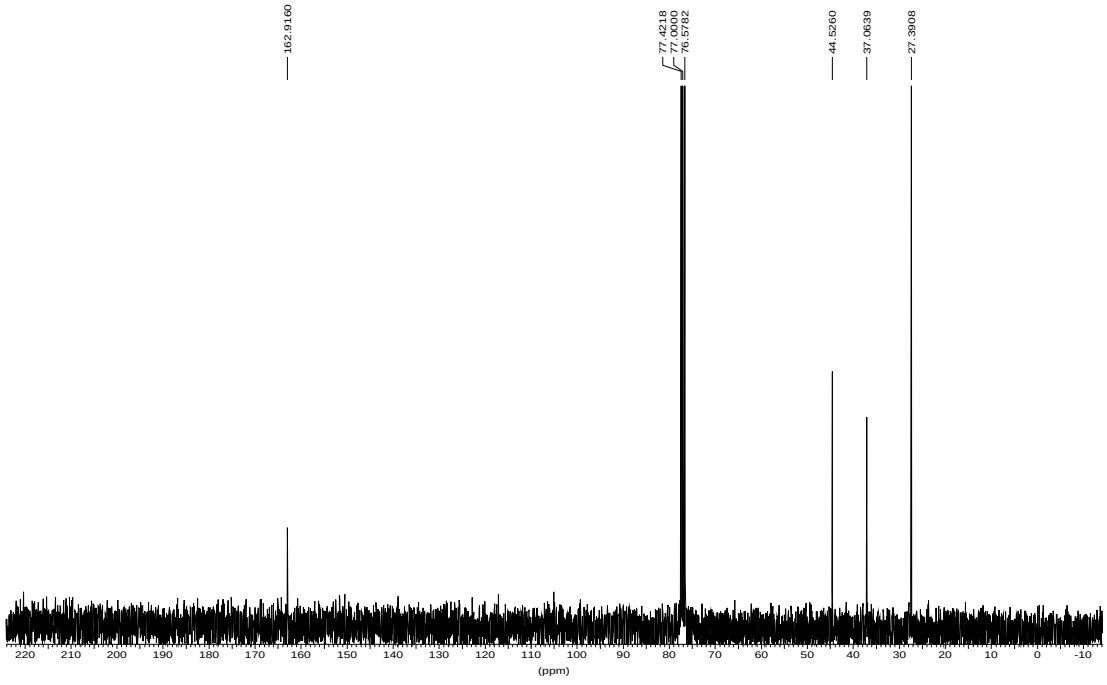

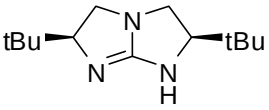


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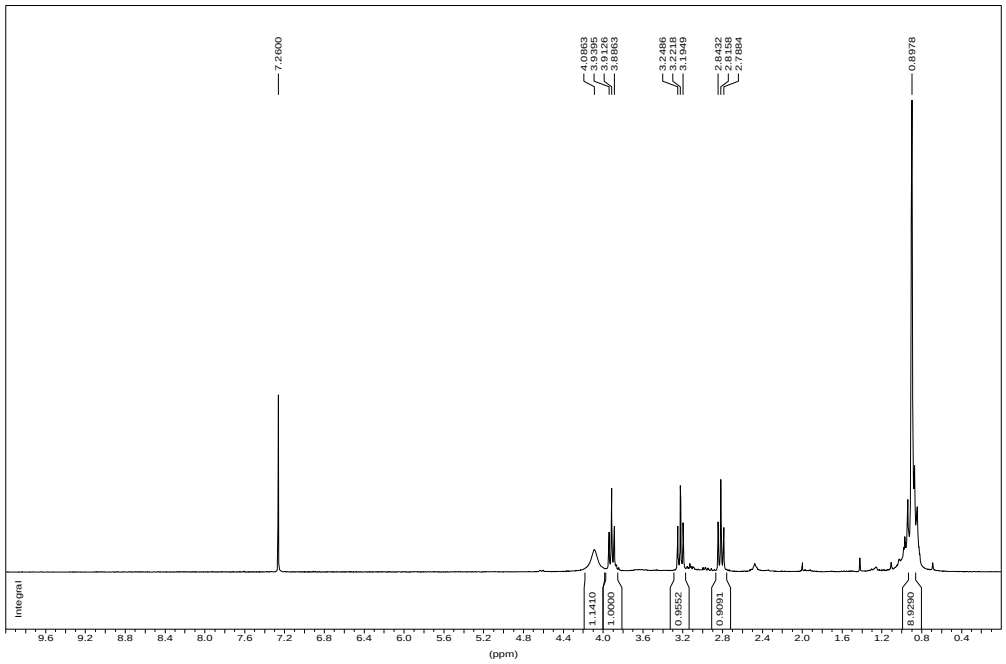


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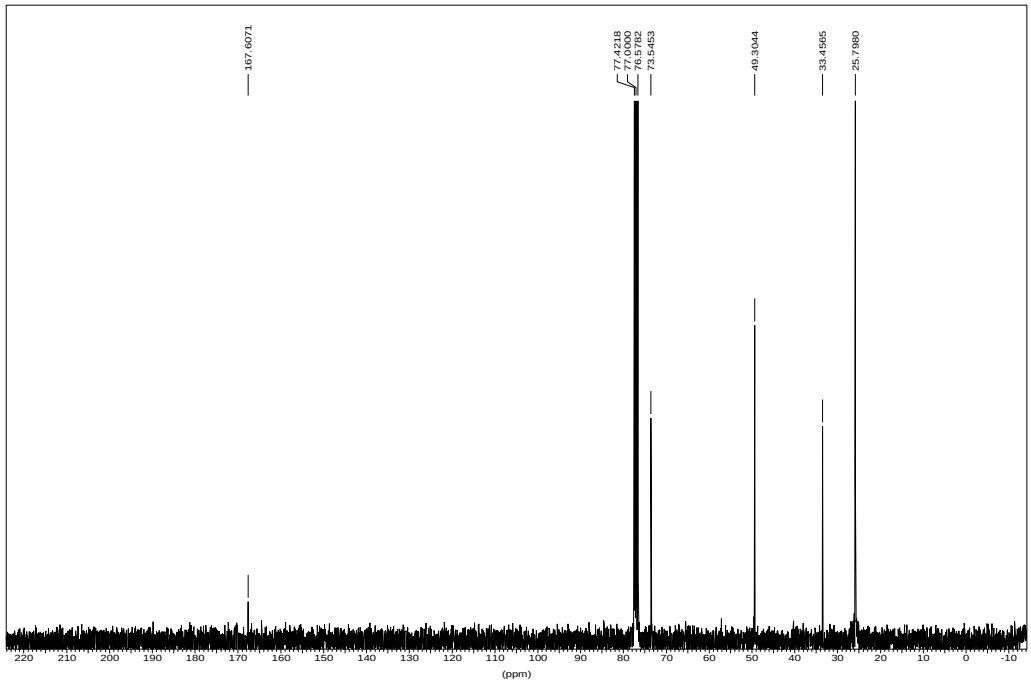

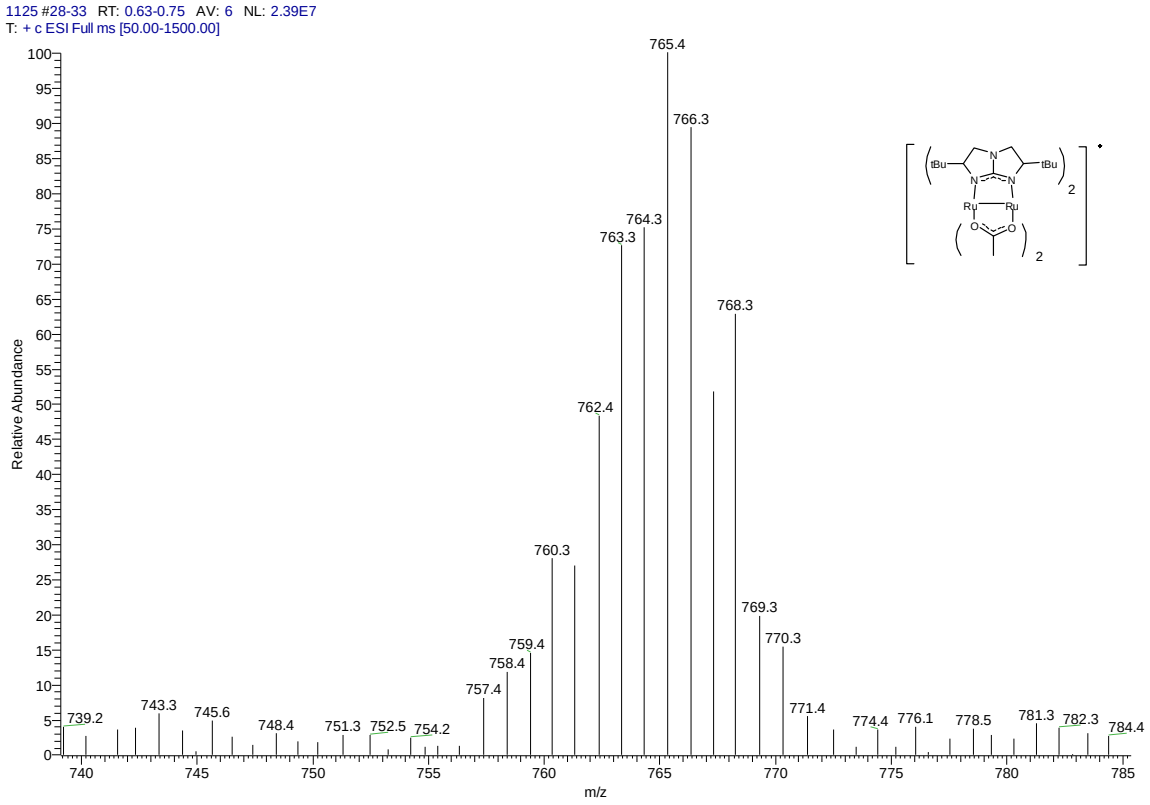
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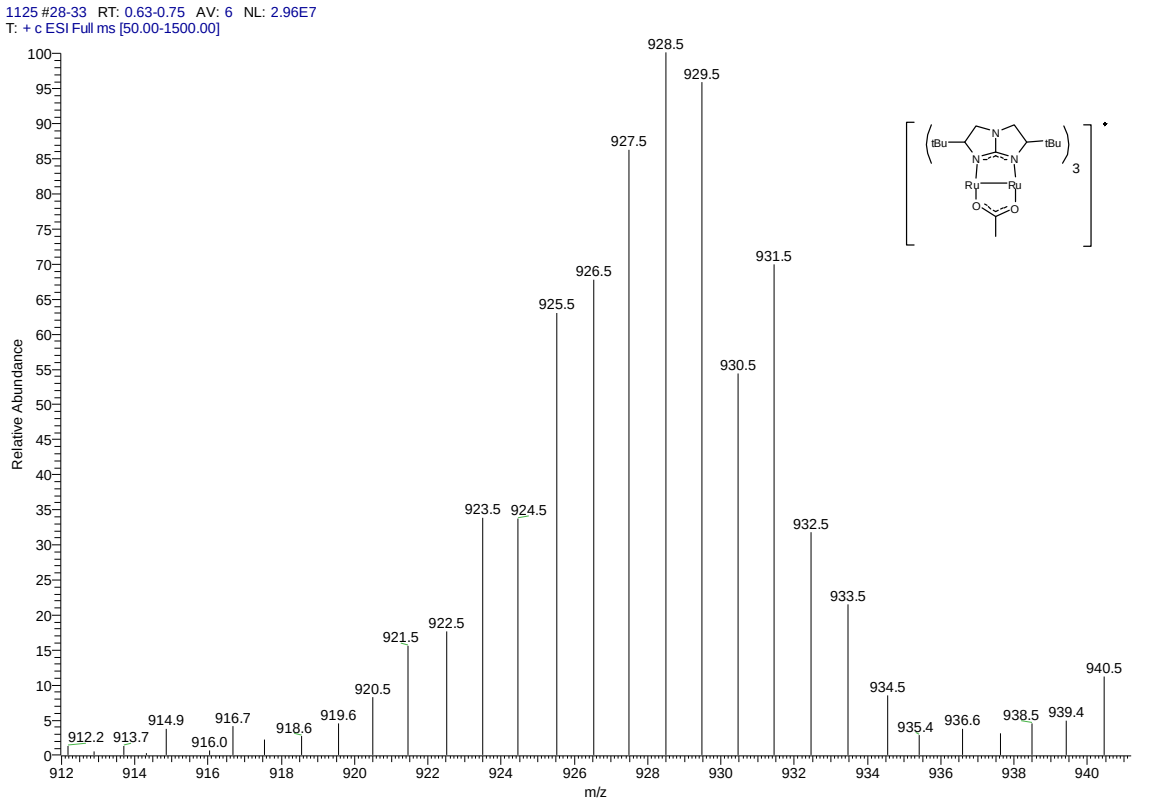
766.3
764.3
763.3
768.3
762.4
760.3
769.3
770.3
759.4
758.4
757.4
771.4
743.3
739.2
745.6
748.4
751.3
752.5
754.2
774.4
776.1
778.5
781.3
782.3
784.4
tBu
N
Ru
O
2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916.0
912.2
913.7
935.4
936.6
938.5
939.4
tBu
N
Ru
O
3

Table 1. Crystal data and structure refinement for $Ru_2(TBO)_2(OAc)_2Cl$•3MeCN (**1•3MeCN**).

| | | |
|---|---|---|
| Empirical formula | C36 H63 Cl N9 O4 Ru2 | |
| Formula weight | 923.54 | |
| Temperature | 223(2) K | |
| Wavelength | 0.71073 Å | |
| Crystal system | Monoclinic | |
| Space group | P2(1)/n | |
| Unit cell dimensions | a = 11.3725(13) Å | α= 90°. |
| | b = 34.635(4) Å | β= 117.046(2)°. |
| | c = 12.3568(13) Å | γ = 90°. |
| Volume | 4334.9(8) $Å^3$ | |
| Z | 4 | |
| Density (calculated) | 1.415 $Mg/m^3$ | |
| Absorption coefficient | 0.804 $mm^{-1}$ | |
| F(000) | 1916 | |
| Crystal size | 0.60 x 0.20 x 0.10 $mm^3$ | |
| Theta range for data collection | 1.94 to 27.50°. | |
| Index ranges | -14<=h<=14, -45<=k<=40, -10<=l<=16 | |
| Reflections collected | 30059 | |
| Independent reflections | 9946 [R(int) = 0.0688] | |
| Completeness to theta = 27.50° | 99.9 % | |
| Absorption correction | Semi-empirical from equivalents | |
| Max. and min. transmission | 0.9239 and 0.6439 | |
| Refinement method | Full-matrix least-squares on $F^2$ | |
| Data / restraints / parameters | 9946 / 0 / 472 | |
| Goodness-of-fit on $F^2$ | 1.236 | |
| Final R indices [I>2sigma(I)] | R1 = 0.0861, wR2 = 0.1767 | |
| R indices (all data) | R1 = 0.1056, wR2 = 0.1849 | |
| Largest diff. peak and hole | 2.182 and -1.668 $e.Å^{-3}$ | |

Table 2. Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters ($Å^2$x $10^3$) for 8610. U(eq) is defined as one third of the trace of the orthogonalized $U^{ij}$ tensor.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| Ru(1) | 1372(1) | 1042(1) | 4419(1) | 18(1) |
| Ru(2) | 3090(1) | 1110(1) | 3876(1) | 18(1) |
| Cl(1) | -474(2) | 895(1) | 4938(2) | 30(1) |
| O(1) | 1901(4) | 469(1) | 4803(4) | 26(1) |
| O(2) | 3414(4) | 520(1) | 4128(4) | 26(1) |
| O(3) | 145(4) | 891(1) | 2644(4) | 29(1) |
| O(4) | 1732(5) | 1005(1) | 2098(4) | 31(1) |
| N(1) | 2697(5) | 1153(1) | 6198(4) | 21(1) |
| N(2) | 4456(5) | 1166(1) | 5647(4) | 21(1) |
| N(3) | 4770(5) | 931(2) | 7481(5) | 28(1) |
| N(4) | 871(5) | 1602(1) | 3980(5) | 25(1) |
| N(5) | 2742(5) | 1684(1) | 3634(5) | 24(1) |
| N(6) | 1664(7) | 2200(2) | 3925(6) | 37(1) |
| N(7) | 4719(7) | 1006(2) | 3159(6) | 46(2) |
| C(1) | 3945(6) | 1103(2) | 6411(6) | 25(1) |
| C(2) | 2653(7) | 992(2) | 7315(6) | 27(1) |
| C(3) | 3885(7) | 735(2) | 7891(7) | 38(2) |
| C(4) | 5779(7) | 736(2) | 7264(6) | 35(2) |
| C(5) | 5833(7) | 991(2) | 6255(5) | 27(1) |
| C(6) | 2588(8) | 1305(2) | 8166(6) | 39(2) |
| C(7) | 3872(9) | 1543(3) | 8759(8) | 57(2) |
| C(8) | 1468(9) | 1583(2) | 7438(8) | 54(2) |
| C(9) | 2340(10) | 1111(3) | 9125(7) | 60(3) |
| C(10) | 6921(7) | 1300(2) | 6749(6) | 33(2) |
| C(11) | 6831(10) | 1553(3) | 7715(9) | 65(3) |
| C(12) | 6842(9) | 1547(3) | 5701(8) | 55(2) |
| C(13) | 8255(8) | 1097(3) | 7293(8) | 52(2) |
| C(14) | 1755(7) | 1807(2) | 3812(6) | 30(2) |
| C(15) | 15(9) | 1886(2) | 4208(8) | 48(2) |
| C(16) | 912(9) | 2248(2) | 4609(9) | 51(2) |

| | | | | |
|---|---|---|---|---|
| C(17) | 3014(8) | 2335(2) | 4283(7) | 42(2) |
| C(18) | 3518(7) | 2039(2) | 3672(6) | 30(2) |
| C(19) | -1320(8) | 1953(3) | 3119(9) | 58(3) |
| C(20) | -2104(9) | 1578(3) | 2739(9) | 63(3) |
| C(21) | -2131(11) | 2233(3) | 3454(13) | 98(5) |
| C(22) | -1181(10) | 2101(4) | 2019(11) | 99(5) |
| C(23) | 3368(8) | 2164(2) | 2419(7) | 35(2) |
| C(24) | 3842(11) | 1837(2) | 1891(8) | 58(3) |
| C(25) | 4263(9) | 2512(2) | 2601(9) | 52(2) |
| C(26) | 1962(9) | 2264(3) | 1544(8) | 59(2) |
| C(27) | 2764(7) | 327(2) | 4543(6) | 26(1) |
| C(28) | 3052(8) | -95(2) | 4750(7) | 38(2) |
| C(29) | 559(7) | 918(2) | 1858(5) | 30(2) |
| C(30) | -409(9) | 841(3) | 564(7) | 51(2) |
| C(31) | 5366(7) | 881(2) | 2785(7) | 36(2) |
| C(32) | 6177(9) | 712(3) | 2268(9) | 62(3) |
| N(1S) | 3498(10) | -171(3) | -122(9) | 96(3) |
| C(1S) | 3119(10) | -3(3) | 429(8) | 53(2) |
| C(2S) | 2598(11) | 198(3) | 1134(9) | 64(3) |
| N(2S) | -208(10) | 157(3) | 8208(8) | 76(3) |
| C(3S) | 285(8) | 89(2) | 7616(8) | 45(2) |
| C(4S) | 912(8) | 2(2) | 6855(8) | 44(2) |

Table 3. Bond lengths [Å] and angles [°] for 8610.

| | |
|---|---|
| Ru(1)-N(4) | 2.027(5) |
| Ru(1)-N(1) | 2.059(5) |
| Ru(1)-O(3) | 2.061(4) |
| Ru(1)-O(1) | 2.066(4) |
| Ru(1)-Ru(2) | 2.3493(7) |
| Ru(1)-Cl(1) | 2.5097(17) |
| Ru(2)-N(5) | 2.022(5) |
| Ru(2)-N(2) | 2.037(5) |
| Ru(2)-O(4) | 2.063(4) |
| Ru(2)-O(2) | 2.074(4) |
| Ru(2)-N(7) | 2.416(8) |
| O(1)-C(27) | 1.262(7) |
| O(2)-C(27) | 1.264(8) |
| O(3)-C(29) | 1.260(8) |
| O(4)-C(29) | 1.265(8) |
| N(1)-C(1) | 1.332(8) |
| N(1)-C(2) | 1.509(8) |
| N(2)-C(1) | 1.331(8) |
| N(2)-C(5) | 1.520(8) |
| N(3)-C(1) | 1.362(8) |
| N(3)-C(4) | 1.459(9) |
| N(3)-C(3) | 1.483(8) |
| N(4)-C(14) | 1.322(8) |
| N(4)-C(15) | 1.496(8) |
| N(5)-C(14) | 1.310(8) |
| N(5)-C(18) | 1.503(8) |
| N(6)-C(14) | 1.376(8) |
| N(6)-C(16) | 1.459(9) |
| N(6)-C(17) | 1.467(10) |
| N(7)-C(31) | 1.118(10) |
| C(2)-C(3) | 1.535(10) |
| C(2)-C(6) | 1.536(9) |
| C(4)-C(5) | 1.552(9) |

| | |
|---|---|
| C(5)-C(10) | 1.537(9) |
| C(6)-C(9) | 1.497(10) |
| C(6)-C(8) | 1.522(12) |
| C(6)-C(7) | 1.542(11) |
| C(10)-C(12) | 1.520(10) |
| C(10)-C(11) | 1.521(11) |
| C(10)-C(13) | 1.524(10) |
| C(15)-C(19) | 1.520(12) |
| C(15)-C(16) | 1.551(12) |
| C(17)-C(18) | 1.531(10) |
| C(18)-C(23) | 1.541(9) |
| C(19)-C(21) | 1.518(13) |
| C(19)-C(20) | 1.524(13) |
| C(19)-C(22) | 1.526(13) |
| C(23)-C(26) | 1.508(11) |
| C(23)-C(24) | 1.523(10) |
| C(23)-C(25) | 1.525(10) |
| C(27)-C(28) | 1.494(9) |
| C(29)-C(30) | 1.496(9) |
| C(31)-C(32) | 1.462(11) |
| N(1S)-C(1S) | 1.120(12) |
| C(1S)-C(2S) | 1.438(13) |
| N(2S)-C(3S) | 1.131(11) |
| C(3S)-C(4S) | 1.447(11) |

| | |
|---|---|
| N(4)-Ru(1)-N(1) | 95.0(2) |
| N(4)-Ru(1)-O(3) | 89.2(2) |
| N(1)-Ru(1)-O(3) | 175.07(19) |
| N(4)-Ru(1)-O(1) | 177.46(19) |
| N(1)-Ru(1)-O(1) | 86.50(18) |
| O(3)-Ru(1)-O(1) | 89.20(18) |
| N(4)-Ru(1)-Ru(2) | 89.81(15) |
| N(1)-Ru(1)-Ru(2) | 89.23(14) |
| O(3)-Ru(1)-Ru(2) | 88.23(13) |
| O(1)-Ru(1)-Ru(2) | 88.18(12) |

| | |
|---|---|
| N(4)-Ru(1)-Cl(1) | 95.53(15) |
| N(1)-Ru(1)-Cl(1) | 93.22(15) |
| O(3)-Ru(1)-Cl(1) | 88.90(14) |
| O(1)-Ru(1)-Cl(1) | 86.41(13) |
| Ru(2)-Ru(1)-Cl(1) | 173.90(4) |
| N(5)-Ru(2)-N(2) | 93.9(2) |
| N(5)-Ru(2)-O(4) | 90.8(2) |
| N(2)-Ru(2)-O(4) | 175.32(19) |
| N(5)-Ru(2)-O(2) | 178.86(19) |
| N(2)-Ru(2)-O(2) | 86.29(18) |
| O(4)-Ru(2)-O(2) | 89.03(18) |
| N(5)-Ru(2)-Ru(1) | 90.46(14) |
| N(2)-Ru(2)-Ru(1) | 91.67(14) |
| O(4)-Ru(2)-Ru(1) | 88.26(13) |
| O(2)-Ru(2)-Ru(1) | 88.41(12) |
| N(5)-Ru(2)-N(7) | 102.9(2) |
| N(2)-Ru(2)-N(7) | 94.1(2) |
| O(4)-Ru(2)-N(7) | 84.9(2) |
| O(2)-Ru(2)-N(7) | 78.18(19) |
| Ru(1)-Ru(2)-N(7) | 165.02(16) |
| C(27)-O(1)-Ru(1) | 119.6(4) |
| C(27)-O(2)-Ru(2) | 119.0(4) |
| C(29)-O(3)-Ru(1) | 119.5(4) |
| C(29)-O(4)-Ru(2) | 119.4(4) |
| C(1)-N(1)-C(2) | 104.4(5) |
| C(1)-N(1)-Ru(1) | 112.6(4) |
| C(2)-N(1)-Ru(1) | 126.5(4) |
| C(1)-N(2)-C(5) | 105.6(5) |
| C(1)-N(2)-Ru(2) | 112.4(4) |
| C(5)-N(2)-Ru(2) | 127.2(4) |
| C(1)-N(3)-C(4) | 106.1(5) |
| C(1)-N(3)-C(3) | 105.0(5) |
| C(4)-N(3)-C(3) | 124.3(6) |
| C(14)-N(4)-C(15) | 106.5(5) |
| C(14)-N(4)-Ru(1) | 114.5(4) |

| | |
|---|---|
| C(15)-N(4)-Ru(1) | 134.9(4) |
| C(14)-N(5)-C(18) | 105.2(5) |
| C(14)-N(5)-Ru(2) | 115.0(4) |
| C(18)-N(5)-Ru(2) | 136.7(4) |
| C(14)-N(6)-C(16) | 105.3(6) |
| C(14)-N(6)-C(17) | 103.2(6) |
| C(16)-N(6)-C(17) | 126.7(7) |
| C(31)-N(7)-Ru(2) | 165.2(6) |
| N(2)-C(1)-N(1) | 127.5(6) |
| N(2)-C(1)-N(3) | 115.4(6) |
| N(1)-C(1)-N(3) | 116.7(6) |
| N(1)-C(2)-C(3) | 103.6(5) |
| N(1)-C(2)-C(6) | 113.5(5) |
| C(3)-C(2)-C(6) | 114.2(6) |
| N(3)-C(3)-C(2) | 101.1(5) |
| N(3)-C(4)-C(5) | 101.8(5) |
| N(2)-C(5)-C(10) | 112.5(5) |
| N(2)-C(5)-C(4) | 102.4(5) |
| C(10)-C(5)-C(4) | 113.4(5) |
| C(9)-C(6)-C(8) | 110.2(7) |
| C(9)-C(6)-C(2) | 108.0(7) |
| C(8)-C(6)-C(2) | 109.2(6) |
| C(9)-C(6)-C(7) | 110.1(7) |
| C(8)-C(6)-C(7) | 107.5(7) |
| C(2)-C(6)-C(7) | 111.9(6) |
| C(12)-C(10)-C(11) | 110.2(7) |
| C(12)-C(10)-C(13) | 107.3(6) |
| C(11)-C(10)-C(13) | 109.2(7) |
| C(12)-C(10)-C(5) | 109.1(6) |
| C(11)-C(10)-C(5) | 112.5(6) |
| C(13)-C(10)-C(5) | 108.3(6) |
| N(5)-C(14)-N(4) | 128.4(6) |
| N(5)-C(14)-N(6) | 116.8(6) |
| N(4)-C(14)-N(6) | 114.6(6) |
| N(4)-C(15)-C(19) | 114.1(7) |

| | |
|---|---|
| N(4)-C(15)-C(16) | 101.6(6) |
| C(19)-C(15)-C(16) | 113.9(7) |
| N(6)-C(16)-C(15) | 101.3(6) |
| N(6)-C(17)-C(18) | 102.3(6) |
| N(5)-C(18)-C(17) | 102.6(5) |
| N(5)-C(18)-C(23) | 113.9(6) |
| C(17)-C(18)-C(23) | 114.3(6) |
| C(21)-C(19)-C(15) | 110.0(9) |
| C(21)-C(19)-C(20) | 106.6(8) |
| C(15)-C(19)-C(20) | 110.5(6) |
| C(21)-C(19)-C(22) | 110.5(8) |
| C(15)-C(19)-C(22) | 111.9(7) |
| C(20)-C(19)-C(22) | 107.1(10) |
| C(26)-C(23)-C(24) | 109.3(7) |
| C(26)-C(23)-C(25) | 110.0(6) |
| C(24)-C(23)-C(25) | 107.9(6) |
| C(26)-C(23)-C(18) | 112.4(6) |
| C(24)-C(23)-C(18) | 108.8(6) |
| C(25)-C(23)-C(18) | 108.2(6) |
| O(1)-C(27)-O(2) | 124.3(6) |
| O(1)-C(27)-C(28) | 118.1(6) |
| O(2)-C(27)-C(28) | 117.6(6) |
| O(3)-C(29)-O(4) | 124.1(6) |
| O(3)-C(29)-C(30) | 117.4(7) |
| O(4)-C(29)-C(30) | 118.5(6) |
| N(7)-C(31)-C(32) | 178.3(9) |
| N(1S)-C(1S)-C(2S) | 177.4(12) |
| N(2S)-C(3S)-C(4S) | 179.8(12) |

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Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for 8610. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{23}$ | $U^{13}$ | $U^{12}$ |
|---|---|---|---|---|---|---|
| Ru(1) | 21(1) | 17(1) | 19(1) | 1(1) | 12(1) | 2(1) |
| Ru(2) | 24(1) | 17(1) | 19(1) | 1(1) | 15(1) | 2(1) |
| Cl(1) | 26(1) | 35(1) | 33(1) | 3(1) | 18(1) | 0(1) |
| O(1) | 32(3) | 20(2) | 31(2) | 3(2) | 19(2) | 2(2) |
| O(2) | 30(3) | 18(2) | 36(3) | 1(2) | 20(2) | 3(2) |
| O(3) | 30(3) | 37(2) | 19(2) | -2(2) | 12(2) | -1(2) |
| O(4) | 35(3) | 42(3) | 22(2) | -3(2) | 18(2) | -2(2) |
| N(1) | 22(3) | 24(2) | 20(2) | -1(2) | 12(2) | -1(2) |
| N(2) | 24(3) | 23(2) | 18(2) | -2(2) | 13(2) | -2(2) |
| N(3) | 12(3) | 41(3) | 26(3) | 8(2) | 4(2) | 3(2) |
| N(4) | 29(3) | 22(2) | 33(3) | 8(2) | 21(3) | 8(2) |
| N(5) | 23(3) | 24(3) | 32(3) | 6(2) | 19(2) | 2(2) |
| N(6) | 54(4) | 24(3) | 53(4) | 5(3) | 41(3) | 6(3) |
| N(7) | 49(4) | 41(4) | 47(4) | 2(3) | 21(4) | 2(3) |
| C(1) | 27(3) | 23(3) | 27(3) | -9(2) | 13(3) | -6(2) |
| C(2) | 32(4) | 37(3) | 19(3) | -3(3) | 16(3) | -9(3) |
| C(3) | 33(4) | 50(4) | 35(4) | 12(3) | 20(3) | -8(3) |
| C(4) | 28(4) | 43(4) | 34(4) | 14(3) | 14(3) | 6(3) |
| C(5) | 34(4) | 34(3) | 13(3) | -1(2) | 11(3) | -3(3) |
| C(6) | 52(5) | 53(4) | 21(3) | -17(3) | 26(4) | -13(4) |
| C(7) | 68(6) | 70(6) | 48(5) | -29(4) | 39(5) | -29(5) |
| C(8) | 70(6) | 54(5) | 64(6) | -19(4) | 53(5) | -6(4) |
| C(9) | 75(7) | 90(7) | 38(5) | -6(4) | 46(5) | -14(5) |
| C(10) | 27(4) | 46(4) | 31(4) | 1(3) | 18(3) | -3(3) |
| C(11) | 70(7) | 63(6) | 81(7) | -37(5) | 50(6) | -31(5) |
| C(12) | 43(5) | 63(5) | 56(5) | 19(4) | 20(4) | -9(4) |
| C(13) | 30(4) | 82(6) | 45(5) | 13(4) | 18(4) | 0(4) |
| C(14) | 33(4) | 28(3) | 34(4) | 10(3) | 19(3) | 6(3) |
| C(15) | 64(6) | 35(4) | 74(6) | 19(4) | 56(5) | 21(4) |
| C(16) | 75(6) | 31(4) | 79(6) | 1(4) | 63(6) | 7(4) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C(17) | 60(5) | 32(4) | 49(5) | -5(3) | 37(4) | -10(3) |
| C(18) | 35(4) | 25(3) | 35(4) | 7(3) | 21(3) | 1(3) |
| C(19) | 41(5) | 54(5) | 95(7) | 47(5) | 44(5) | 26(4) |
| C(20) | 45(5) | 86(7) | 62(6) | 41(5) | 27(5) | 29(5) |
| C(21) | 71(8) | 71(7) | 194(14) | 45(8) | 97(9) | 39(6) |
| C(22) | 46(6) | 147(11) | 109(9) | 94(9) | 40(6) | 32(7) |
| C(23) | 45(5) | 32(4) | 43(4) | 7(3) | 33(4) | 0(3) |
| C(24) | 106(8) | 50(5) | 55(5) | 6(4) | 70(6) | -4(5) |
| C(25) | 60(6) | 38(4) | 80(6) | 18(4) | 52(5) | 6(4) |
| C(26) | 63(6) | 79(6) | 40(5) | 21(4) | 28(5) | -1(5) |
| C(27) | 28(4) | 26(3) | 27(3) | -1(3) | 15(3) | 3(3) |
| C(28) | 44(5) | 27(3) | 52(5) | 5(3) | 30(4) | 6(3) |
| C(29) | 35(4) | 36(4) | 14(3) | 2(3) | 9(3) | 3(3) |
| C(30) | 48(5) | 74(6) | 25(4) | -4(4) | 12(4) | -5(4) |
| C(31) | 31(4) | 45(4) | 37(4) | -2(3) | 20(3) | 1(3) |
| C(32) | 54(6) | 86(7) | 60(6) | -13(5) | 37(5) | 8(5) |
| N(1S) | 87(7) | 141(9) | 67(6) | -18(6) | 41(6) | 26(7) |
| C(1S) | 60(6) | 63(6) | 42(5) | 3(4) | 28(5) | 4(5) |
| C(2S) | 84(8) | 57(6) | 64(6) | -8(5) | 46(6) | -2(5) |
| N(2S) | 85(7) | 92(7) | 72(6) | 0(5) | 55(6) | 4(5) |
| C(3S) | 45(5) | 46(5) | 44(5) | 2(4) | 21(4) | -1(4) |
| C(4S) | 42(5) | 41(4) | 51(5) | -2(4) | 24(4) | -1(3) |

Table 5. Hydrogen coordinates ( x $10^4$) and isotropic displacement parameters ($Å^2$x $10^3$) for 8610.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| H(2A) | 1862 | 826 | 7051 | 33 |
| H(3A) | 4262 | 732 | 8780 | 45 |
| H(3B) | 3690 | 469 | 7585 | 45 |
| H(4A) | 5519 | 470 | 6981 | 42 |
| H(4B) | 6631 | 732 | 7996 | 42 |
| H(5A) | 5964 | 825 | 5667 | 32 |
| H(7A) | 4607 | 1374 | 9235 | 85 |
| H(7B) | 3792 | 1739 | 9283 | 85 |
| H(7C) | 4027 | 1667 | 8131 | 85 |
| H(8A) | 643 | 1441 | 7053 | 81 |
| H(8B) | 1637 | 1707 | 6819 | 81 |
| H(8C) | 1408 | 1777 | 7976 | 81 |
| H(9A) | 1531 | 962 | 8743 | 90 |
| H(9B) | 2257 | 1304 | 9654 | 90 |
| H(9C) | 3072 | 940 | 9598 | 90 |
| H(11A) | 7532 | 1744 | 7993 | 98 |
| H(11B) | 6922 | 1394 | 8395 | 98 |
| H(11C) | 5982 | 1683 | 7373 | 98 |
| H(12A) | 7521 | 1744 | 6009 | 83 |
| H(12B) | 5980 | 1669 | 5300 | 83 |
| H(12C) | 6974 | 1386 | 5123 | 83 |
| H(13A) | 8956 | 1288 | 7613 | 78 |
| H(13B) | 8336 | 946 | 6668 | 78 |
| H(13C) | 8323 | 927 | 7943 | 78 |
| H(15A) | -125 | 1794 | 4899 | 58 |
| H(16A) | 391 | 2487 | 4380 | 61 |
| H(16B) | 1489 | 2249 | 5487 | 61 |
| H(17A) | 3546 | 2332 | 5168 | 51 |
| H(17B) | 3016 | 2596 | 3979 | 51 |

| | | | | |
|---|---|---|---|---|
| H(18A) | 4464 | 1989 | 4214 | 36 |
| H(20A) | -2217 | 1476 | 3417 | 95 |
| H(20B) | -1630 | 1392 | 2498 | 95 |
| H(20C) | -2962 | 1627 | 2061 | 95 |
| H(21A) | -2209 | 2135 | 4154 | 147 |
| H(21B) | -3003 | 2259 | 2774 | 147 |
| H(21C) | -1698 | 2482 | 3648 | 147 |
| H(22A) | -671 | 1918 | 1811 | 149 |
| H(22B) | -733 | 2349 | 2212 | 149 |
| H(22C) | -2050 | 2131 | 1336 | 149 |
| H(24A) | 3750 | 1913 | 1101 | 86 |
| H(24B) | 3317 | 1608 | 1807 | 86 |
| H(24C) | 4762 | 1782 | 2430 | 86 |
| H(25A) | 4180 | 2595 | 1820 | 77 |
| H(25B) | 5172 | 2440 | 3125 | 77 |
| H(25C) | 4006 | 2720 | 2973 | 77 |
| H(26A) | 1917 | 2341 | 771 | 88 |
| H(26B) | 1663 | 2476 | 1873 | 88 |
| H(26C) | 1403 | 2041 | 1426 | 88 |
| H(28A) | 2490 | -209 | 5064 | 57 |
| H(28B) | 3970 | -131 | 5332 | 57 |
| H(28C) | 2883 | -218 | 3988 | 57 |
| H(30A) | -1263 | 779 | 516 | 77 |
| H(30B) | -102 | 626 | 258 | 77 |
| H(30C) | -488 | 1069 | 78 | 77 |
| H(32A) | 5885 | 451 | 2002 | 93 |
| H(32B) | 7094 | 709 | 2879 | 93 |
| H(32C) | 6092 | 866 | 1580 | 93 |
| H(2S1) | 1766 | 82 | 998 | 96 |
| H(2S2) | 2457 | 467 | 891 | 96 |
| H(2S3) | 3220 | 181 | 1989 | 96 |
| H(4S1) | 665 | 196 | 6221 | 66 |
| H(4S2) | 629 | -250 | 6488 | 66 |
| H(4S3) | 1863 | 3 | 7344 | 66 |