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L11751

Table S1: Final Anisotropic Thermal Parameters β(i,j) with Estimated Standard Deviations in Parentheses (10^4 ; 10^5 Å^2 for Rh, P and Si) for **1**.

ATOM	BETA(1,1)	BETA(2,2)	BETA(3,3)	BETA(1,2)	BETA(1,3)	BETA(2,3)
Rh	703(4)	730(3)	264(2)	-181(3)	-28(2)	73(2)
P1	842(14)	851(13)	332(6)	-234(10)	-70(8)	56(7)
P2	848(14)	806(12)	317(6)	-294(10)	-58(7)	78(7)
P3	724(13)	836(13)	396(7)	-155(10)	-2(8)	95(7)
Si	886(15)	972(15)	320(7)	-199(12)	2(8)	32(8)
N1	83(5)	93(5)	34(2)	-12(4)	-3(3)	-7(3)
N2	116(6)	130(7)	42(3)	-1(5)	-10(3)	24(3)
C1	69(5)	85(5)	32(2)	-11(4)	-11(3)	4(3)
C2	142(9)	136(8)	34(3)	-32(7)	-9(4)	11(4)
C3	121(8)	116(7)	47(3)	-23(6)	5(4)	-7(4)
C4	108(7)	131(8)	55(4)	-32(6)	9(4)	-11(4)
C11	116(7)	115(7)	40(3)	-28(6)	-15(4)	-1(4)
C12	167(10)	103(7)	70(5)	-32(7)	-17(6)	-19(5)
C13	88(6)	110(6)	44(3)	-35(5)	-6(3)	9(3)
C14	141(9)	117(7)	43(3)	-42(6)	-2(4)	25(4)
C15	106(7)	101(6)	45(3)	-24(5)	-25(4)	3(3)
C16	175(11)	146(9)	40(3)	-42(8)	-29(5)	13(4)
C21	85(6)	104(6)	44(3)	-30(5)	-7(3)	9(3)
C22	116(8)	118(7)	66(4)	-52(6)	-7(5)	18(4)
C23	115(7)	90(6)	43(3)	-35(5)	-11(4)	3(3)
C24	139(9)	129(8)	44(3)	-39(7)	-13(4)	-15(4)
C25	103(6)	107(6)	33(3)	-41(5)	-6(3)	17(3)
C26	161(10)	151(9)	35(3)	-51(8)	-8(4)	3(4)
C31	95(7)	101(6)	55(4)	-25(5)	-17(4)	23(4)
C32	131(9)	99(7)	82(5)	-18(6)	-33(5)	29(5)
C33	111(7)	107(7)	54(4)	-26(6)	22(4)	-2(4)
C34	167(11)	142(9)	56(4)	-59(8)	33(5)	-22(5)
C35	98(7)	112(7)	52(3)	-26(5)	-20(4)	17(4)
C36	156(10)	162(10)	60(4)	-44(8)	-19(6)	2(5)

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L1175-2

Table S2 : Positional and Thermal Parameters of the Hydrogen Atom for **1.**

Atom	x	y	z	B_{iso}
H21	0.982	0.249	0.459	8.0
H22	0.889	0.152	0.449	8.0
H23	0.837	0.260	0.518	8.0
H31	0.864	0.566	0.417	8.0
H32	0.720	0.563	0.475	8.0
H33	0.717	0.619	0.383	8.0
H41	0.602	0.271	0.380	8.0
H42	0.548	0.430	0.359	8.0
H43	0.552	0.373	0.452	8.0
H111	1.049	-0.060	0.106	4.0
H112	1.061	-0.155	0.187	4.0
H121	0.939	-0.231	0.100	8.0
H122	0.824	-0.088	0.101	8.0
H123	0.835	-0.183	0.182	8.0
H131	0.758	0.082	0.342	4.0
H132	0.746	-0.031	0.287	4.0
H141	0.827	-0.134	0.411	8.0
H142	0.959	-0.073	0.390	8.0
H143	0.946	-0.186	0.335	8.0
H151	0.723	0.116	0.155	4.0
H152	0.727	0.248	0.194	4.0
H161	0.759	0.280	0.049	8.0
H162	0.894	0.152	0.042	8.0
H163	0.899	0.284	0.081	8.0
H211	1.418	0.349	0.234	4.0
H212	1.433	0.282	0.150	4.0
H221	1.533	0.468	0.127	8.0
H222	1.386	0.566	0.164	8.0
H223	1.401	0.499	0.080	8.0
H231	1.164	0.596	0.238	4.0
H232	1.020	0.557	0.269	4.0
H241	1.127	0.575	0.384	8.0
H242	1.265	0.462	0.349	8.0

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L1175-3

Table S2 (continued)

H243	1.121	0.423	0.380	8.0
H251	1.034	0.459	0.103	4.0
H252	1.161	0.527	0.085	4.0
H261	1.177	0.386	-0.024	8.0
H262	1.184	0.259	0.040	8.0
H263	1.311	0.326	0.022	8.0
H311	1.211	-0.135	0.337	4.0
H312	1.246	-0.190	0.248	4.0
H321	1.394	-0.338	0.333	8.0
H322	1.452	-0.218	0.352	8.0
H323	1.487	-0.273	0.263	8.0
H331	1.456	0.057	0.125	4.0
H332	1.511	-0.091	0.165	4.0
H341	1.455	-0.104	0.034	8.0
H342	1.298	-0.015	0.062	8.0
H343	1.353	-0.164	0.101	8.0
H351	1.488	-0.017	0.315	4.0
H352	1.433	0.140	0.292	4.0
H361	1.392	0.102	0.437	8.0
H362	1.299	0.005	0.428	8.0
H363	1.245	0.162	0.404	8.0

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L1175-4

Table S3 Bond Distances and Angles for the $SiMe_3$ and PEt_3 ligands in $Rh[C(N_2)SiMe_3)](PEt_3)_3$, **1**.

Si-C2	1.865(7)	Si-C3	1.865(7)
Si-C4	1.873(7)	P1-C11	1.840(6)
P1-C13	1.849(7)	P1-C15	1.846(7)
P2-C21	1.859(6)	P2-C23	1.835(6)
P2-C25	1.839(6)	P3-C31	1.847(6)
P3-C33	1.853(8)	P3-C35	1.848(7)
C11-C12	1.524(11)	C13-C14	1.519(9)
C15-C16	1.532(10)	C21-C22	1.539(9)
C23-C24	1.523(9)	C25-C26	1.530(9)
C31-C32	1.516(9)	C33-C34	1.510(11)
C35-C36	1.533(11)	C1-Si-C2	111.6(3)
C1-Si-C3	110.2(3)	C1-Si-C4	110.8(3)
C2-Si-C3	109.7(3)	C2-Si-C4	109.0(3)
C3-Si-C4	105.4(3)	Rh-P1-C11	119.7(2)
Rh-P1-C13	122.0(2)	Rh-P1-C15	111.2(2)
C11-P1-C13	101.8(3)	C11-P1-C15	100.0(3)
C13-P1-C15	98.1(3)	Rh-P2-C21	127.6(2)
Rh-P2-C23	117.3(2)	Rh-P2-C25	105.4(2)
C21-P2-C23	99.4(3)	C21-P2-C25	101.1(3)
C23-P2-C25	102.7(3)	Rh-P3-C31	117.8(2)
Rh-P3-C33	122.7(2)	Rh-P3-C35	112.0(2)
C31-P3-C33	101.2(3)	C31-P3-C35	98.9(3)
C33-P3-C35	100.4(3)	P1-C11-C12	118.6(5)
P1-C13-C14	114.2(5)	P1-C15-C16	114.0(5)
P2-C21-C22	117.2(5)	P2-C23-C24	111.9(5)
P2-C25-C26	113.4(5)	P3-C31-C32	119.2(5)
P3-C33-C34	114.8(5)	P3-C35-C36	114.5(5)

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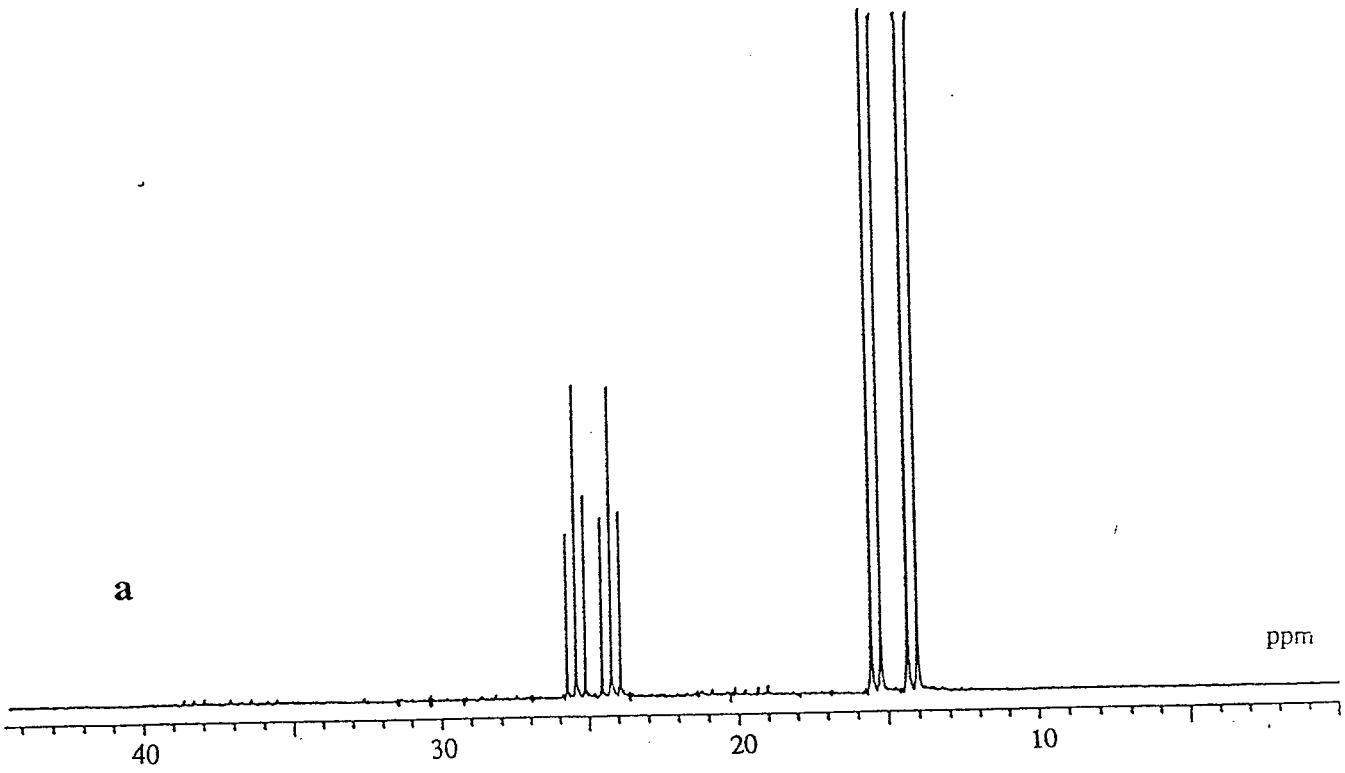
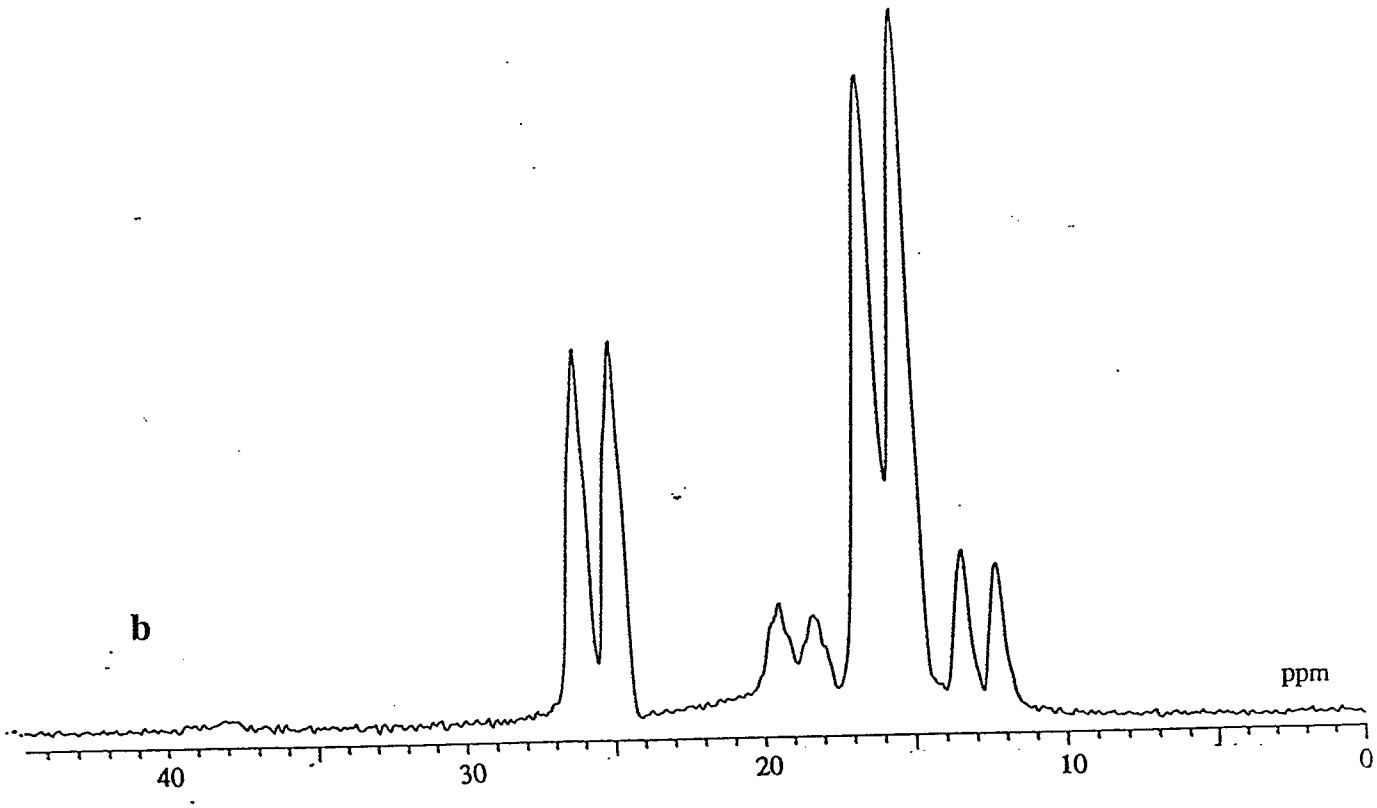
L1175-5





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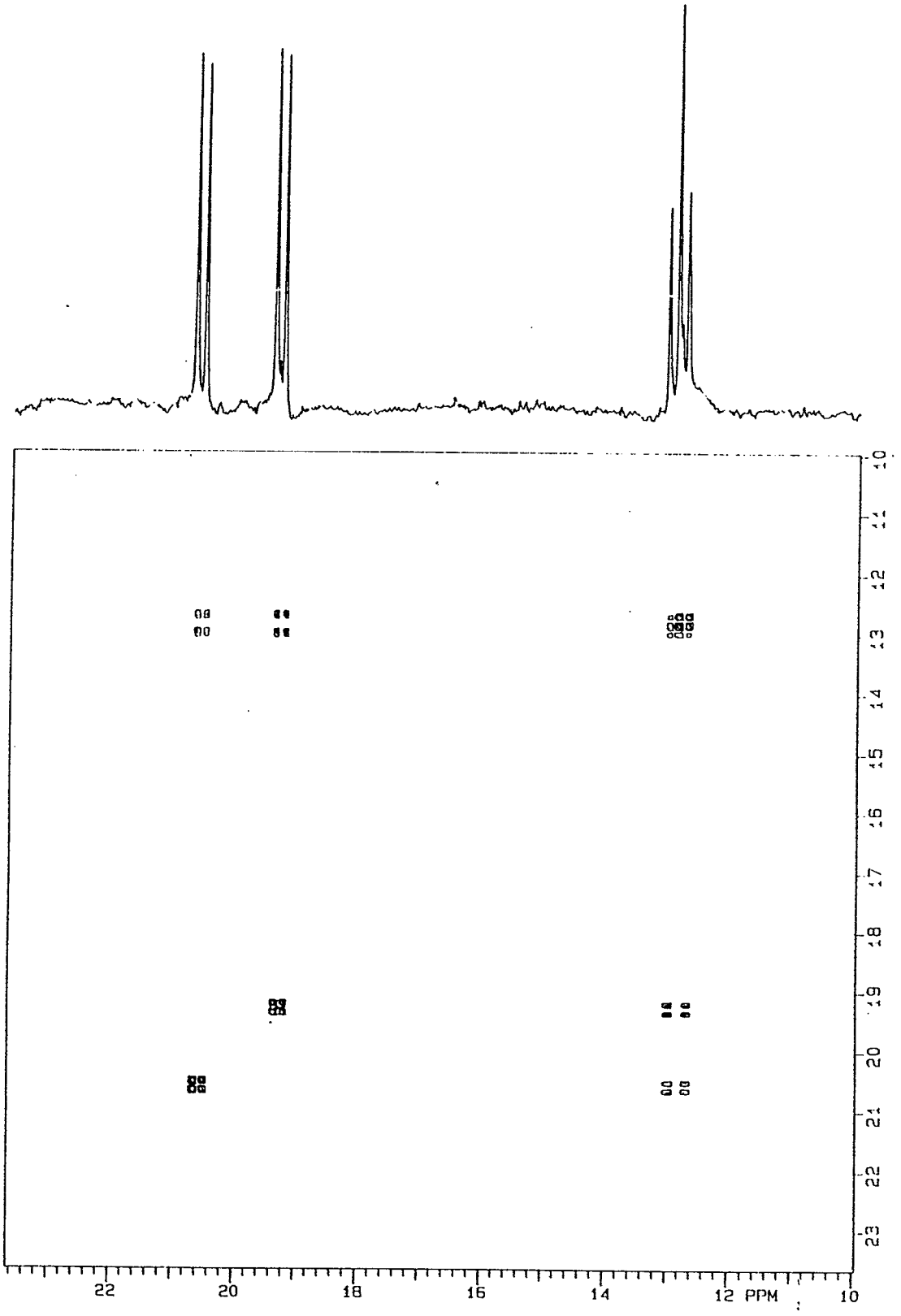
L1175-6



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L1175-7

Table S4. Final Anitropic Thermal Parameters (x 10^2) for **5.**

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Rh	4.68(3)	6.16(4)	4.64(3)	1.05(3)	1.16(2)	0.88(3)
P	4.37(10)	7.08(14)	7.38(14)	0.71(10)	1.15(10)	0.70(11)
Si	8.08(17)	6.59(15)	7.83(18)	0.00(13)	0.05(14)	-0.86(13)
N3	6.6(4)	8.1(4)	4.9(4)	2.8(4)	1.3(3)	0.2(3)
N4	5.5(3)	8.4(5)	5.7(4)	2.4(4)	0.9(3)	0.1(4)
N5	4.8(3)	8.1(4)	4.8(3)	1.6(3)	0.7(3)	1.0(3)
N32	7.5(4)	10.1(5)	5.2(4)	2.5(4)	2.1(3)	2.4(4)
N42	7.4(4)	6.9(4)	5.0(4)	0.5(3)	1.5(3)	1.5(3)
C1	4.7(4)	5.9(4)	4.5(4)	0.1(3)	2.4(3)	0.9(3)
C2	5.7(4)	6.9(5)	4.0(4)	1.3(4)	1.1(3)	0.7(4)
C6	2.3(2)	2.4(3)	3.4(3)	-0.3(2)	0.3(2)	-0.1(2)
C7	7.1(5)	7.9(5)	11.2(7)	-0.3(5)	0.0(5)	-2.1(5)
C8	10.4(6)	7.9(6)	9.2(6)	-2.0(5)	1.3(5)	0.7(5)
C9	7.4(5)	6.8(5)	6.8(5)	-0.5(4)	-0.9(4)	-0.3(4)
C10	7.7(6)	6.5(5)	15.9(8)	-0.2(5)	0.1(6)	-4.1(6)
C11	8.9(6)	6.0(5)	12.5(7)	-0.5(5)	0.9(6)	-1.0(5)
C12	15.3(8)	11.4(7)	8.2(6)	-2.4(7)	-2.2(6)	-1.1(6)
C21	8.5(9)	8.5(9)	10.9(10)	1.8(8)	-2.7(8)	3.0(9)
C22	9.1(9)	9.3(9)	12.6(10)	4.2(8)	0.8(9)	3.7(8)
C23	9.1(7)	9.3(7)	9.6(8)	2.8(7)	3.8(7)	0.9(7)
C24	12.4(8)	14.3(8)	11.5(8)	0.7(7)	1.5(7)	-4.6(7)
C25	6.1(7)	8.7(8)	16.5(10)	0.9(7)	-0.4(8)	2.4(9)
C26	9.4(7)	12.0(8)	33.6(11)	-1.8('7)	9.0(8)	-4.3(9)
C21'	8.8(10)	14.0(11)	12.3(11)	5.5(10)	3.0(10)	3.6(11)
C22'	23.5(12)	27.2(12)	14.9(11)	16.8(11)	11.6(11)	9.6(11)
C23'	8.8(11)	8.4(11)	7.9(11)	-2.4(11)	3.7(11)	-3.2(11)
C25'	10.5(11)	8.7(11)	23.5(12)	0.2(11)	7.2(11)	4.5(11)
C31	5.3(4)	7.4(5)	5.8(5)	1.6(4)	0.7(4)	0.9(4)
C33	10.2(10)	3.6(8)	4.2(7)	0.3(8)	2.7(7)	2.4(7)
C34	15.0(8)	9.2(6)	9.8(7)	0.4(6)	7.0(6)	0.3(5)
C35	19.1(12)	10.0(10)	7.9(9)	-5.5(11)	5.7(10)	-1.7(9)
C36	9.2(10)	16.5(11)	9.6(10)	0.1(10)	0.6(9)	6.3(10)
C33'	10.1(11)	6.3(11)	7.8(10)	0.3(10)	3.9(9)	4.3(10)
C35'	18.2(12)	11.1(11)	8.2(10)	0.9(11)	6.7(11)	3.5(10)
C36'	13.8(11)	22.9(12)	8.6(10)	-5.0(11)	-3.7(10)	8.5(11)

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L1175-8

Table S4 (continued).

C41	4.6(4)	6.1(4)	6.4(5)	0.3(4)	1.3(3)	0.2(4)
C43	12.6(9)	2.6(8)	5.5(8)	0.5(8)	0.5(7)	0.0(7)
C44	14.1(10)	6.5(8)	9.4(8)	1.0(8)	-0.5(8)	1.5(7)
C45	8.9(11)	10.7(11)	13.9(12)	-3.9(10)	-5.1(10)	7.2(10)
C46	23.8(12)	15.3(12)	5.6(10)	3.7(12)	7.6(11)	2.9(10)
C44'	10.9(12)	10.1(12)	9.9(12)	-3.0(12)	-1.2(12)	1.4(11)
C45'	22.4(13)	20.5(13)	8.0(12)	-4.1(12)	1.2(12)	1.1(12)
C46'	11.1(12)	18.1(12)	11.2(12)	2.7(12)	0.1(12)	7.5(12)
C43"	12.7(12)	1.2(11)	2.8(10)	2.8(10)	0.9(10)	0.6(10)
C44"	27.2(13)	11.0(12)	13.4(12)	0.0(12)	5.9(12)	2.5(12)
C45"	6.3(11)	34.4(13)	15.6(12)	4.2(12)	1.8(11)	17.2(12)
C46"	16.2(12)	17.5(12)	6.1(11)	3.7(12)	2.8(12)	1.4(12)

T.F. = $\exp[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{12}hka^*b^* + 2U_{13}hla^*c^* + 2U_{23}klb^*c^*)]$

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L1175-9

Table S5. Positional and Thermal Parameters of the Hydrogen Atoms for **5**.

Atom	x	y	z	B_{iso}
H7A	-0.175	0.542	0.047	8.6
H7B	-0.125	0.673	0.029	8.6
H7C	-0.200	0.678	0.071	8.6
H8A	-0.098	0.451	0.130	8.6
H8B	-0.122	0.589	0.152	8.6
H8C	0.000	0.527	0.160	8.6
H9A	0.021	0.440	0.059	8.6
H9B	0.121	0.513	0.089	8.6
H9C	0.071	0.570	0.040	8.6
H10A	0.345	1.031	0.107	9.1
H10B	0.379	0.938	0.150	9.1
H10C	0.399	1.092	0.154	9.1
H11A	0.208	1.276	0.154	9.1
H11B	0.083	1.223	0.153	9.1
H11C	0.147	1.216	0.107	9.1
H12A	0.151	1.041	0.234	9.1
H12B	0.274	1.099	0.237	9.1
H12C	0.254	0.945	0.233	9.1
H21A [a]	0.406	0.362	0.158	11.7
H21B [a]	0.457	0.458	0.198	11.7
H22A [a]	0.575	0.279	0.193	20.8
H22B [a]	0.637	0.409	0.180	20.8
H22C [a]	0.587	0.313	0.140	20.8
H23A [a]	0.539	0.441	0.074	11.7
H23B [a]	0.468	0.553	0.047	11.7
H24A [a]	0.397	0.347	0.023	20.8
H24B [a]	0.305	0.425	0.047	20.8
H24C [a]	0.377	0.313	0.075	20.8
H25A [a]	0.641	0.619	0.143	11.7
H25B [a]	0.570	0.721	0.169	11.7
H26A	0.652	0.826	0.110	20.8
H26B	0.520	0.830	0.099	20.8
H26C	0.591	0.728	0.072	20.8
H21A' [b]	0.526	0.560	0.204	11.7
H21B' [b]	0.580	0.449	0.175	11.7
H22A' [b]	0.520	0.375	0.235	20.8

L1175-10

Table S5 (continued).

H22B' [b]	0.451	0.317	0.189	20.8
H22C' [b]	0.398	0.428	0.219	20.8
H23A' [b]	0.389	0.328	0.114	11.7
H23B' [b]	0.504	0.352	0.093	11.7
H24A' [b]	0.377	0.291	0.042	20.8
H24B' [b]	0.417	0.436	0.032	20.8
H24C' [b]	0.302	0.413	0.053	20.8
H25A' [b]	0.539	0.608	0.064	11.7
H25B' [b]	0.624	0.579	0.109	11.7
H26A' [b]	0.648	0.795	0.084	20.8
H26B' [b]	0.606	0.798	0.134	20.8
H26C' [b]	0.521	0.827	0.089	20.8
H34A [a]	0.088	0.645	0.318	9.2
H34B [a]	0.065	0.691	0.265	9.2
H34C [a]	0.177	0.733	0.295	9.2
H35A [a]	0.031	0.460	0.240	9.2
H35B [a]	0.063	0.396	0.290	9.2
H35C [a]	0.127	0.352	0.247	9.2
H36A [a]	0.248	0.457	0.334	9.2
H36B [a]	0.326	0.563	0.313	9.2
H36C [a]	0.314	0.421	0.291	9.2
H34A' [b]	0.112	0.641	0.321	9.2
H34B' [b]	0.048	0.648	0.270	9.2
H34C' [b]	0.142	0.754	0.287	9.2
H35A' [b]	0.136	0.404	0.299	9.2
H35B' [b]	0.218	0.370	0.262	9.2
H35C' [b]	0.095	0.422	0.246	9.2
H36A' [b]	0.294	0.564	0.341	9.2
H36B' [b]	0.360	0.651	0.307	9.2
H36C' [b]	0.360	0.495	0.303	9.2
H44A [d]	0.212	1.070	-0.032	10.6
H44B [d]	0.314	1.046	0.007	10.6
H44C [d]	0.190	1.036	0.020	10.6

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L1175-11

Table S5 (continued).

H45A [d]	0.099	0.877	-0.062	10.6
H45B [d]	0.075	0.853	-0.010	10.6
H45C [d]	0.119	0.736	-0.039	10.6
H46A [d]	0.296	0.888	-0.084	10.6
H46B [d]	0.329	0.750	-0.061	10.6
H46C [d]	0.396	0.878	-0.043	10.6
H44A' [e]	0.106	0.771	-0.045	10.6
H44B' [e]	0.223	0.723	-0.060	10.6
H44C' [e]	0.167	0.855	-0.081	10.6
H45A' [e]	0.395	0.957	-0.015	10.6
H45B' [e]	0.331	0.958	-0.066	10.6
H45C' [e]	0.386	0.826	-0.045	10.6
H46A' [e]	0.171	1.021	0.025	10.6
H46B' [e]	0.077	0.916	0.008	10.6
H46C' [e]	0.112	1.023	-0.027	10.6
H44A" [e]	0.319	1.057	0.000	10.6
H44B" [e]	0.290	1.028	-0.054	10.6
H44C" [e]	0.381	0.942	-0.024	10.6
H45A" [e]	0.052	0.920	0.011	10.6
H45B" [e]	0.070	0.777	-0.009	10.6
H45C" [e]	0.042	0.896	-0.044	10.6
H46A" [e]	0.181	0.729	-0.058	10.6
H46B" [e]	0.310	0.762	-0.058	10.6
H46C" [e]	0.219	0.848	-0.088	10.6

Occupancy factors : a = 0.55, b = 0.45, c = 0.70, d = 0.40, e = 0.30

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L1175-12

Table S6. Geometry of the Disordered Phosphine and Isocyanide Ligands in **5.**

Phosphine (Occupancy 0.55)

P-C21	1.797(20)	C21-P-C23	101.0(8)
P-C23	1.826(14)	C21-P-C25	105.3(8)
P-C25	1.901(15)	C23-P-C25	103.1(8)
C21-C22	1.50(3)	P-C21-C22	119.3(15)
C23-C24	1.496(18)	P-C23-C24	115.4(10)
C25-C26	1.48(3)	P-C25-C26	112.0(11)

Phosphine (Occupancy 0.45)

P-C21'	1.87(3)	C21'-P-C23'	91.5(14)
P-C23'	1.99(4)	C21'-P-C25'	106.4(16)
P-C25'	1.76(4)	C23'-P-C25'	97.6(17)
C21'-C22'	1.28(5)	P-C21'-C22'	127.5(23)
C23'-C24	1.28(4)	P-C23'-C24	117.0(24)
C25'-C26	1.50(3)	P-C25'-C26	118(3)

Isocyanide # 1 (Occupancy 0.55)

C31-N32	1.142(11)	C31-N32-C33	167.9(10)
N32-C33	1.56(2)	N32-C33-C34	100.1(11)
C33-C34	1.54(2)	N32-C33-C35	112.0(16)
C33-C35	1.53(3)	N32-C33-C36	108.4(18)
C33-C36	1.42(3)	C34-C33-C35	109.4(18)
		C34-C33-C36	114.4(17)
		C35-C33-C36	112.0(18)

Isocyanide # 1 (Occupancy 0.45)

C31-N32	1.142(11)	C31-N32-C33'	169.7(14)
N32-C33'	1.35(4)	N32-C33'-C34	120.6(23)
C33'-C34	1.39(3)	N32-C33'-C35'	97.4(22)
C33'-C35'	1.60(4)	N32-C33'-C36'	111.8(25)
C33'-C36'	1.58(4)	C34-C33'-C35'	109.7(25)
		C34-C33'-C36'	111.4(25)
		C35'-C33'-C36'	103.7(23)

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L1175-13

Table S6 (continued).

Isocyanide # 2 (Occupancy 0.40)

C41-N42	1.146(10)	C41-N42-C43	164.9(13)
N42-C43	1.40(2)	N42-C43-C44	106(2)
C43-C44	1.59(2)	N42-C43-C45	111(2)
C43-C45	1.42(4)	N42-C43-C46	111(2)
C43-C46	1.55(5)	C44-C43-C45	107(2)
		C44-C43-C46	108(2)
		C45-C43-C46	114(3)

Isocyanide #2 (Occupancy 0.30)

C41-N42	1.146(10)	C41-N42-C43	164.9(13)
N42-C43	1.40(2)	N42-C43-C44'	121(2)
C43-C44'	1.34(5)	N42-C43-C45'	104(3)
C43-C45'	1.53(6)	N42-C43-C46'	103(2)
C43-C46'	1.65(5)	C44'-C43-C46'	103(3)
		C44'-C43-C46'	100(3)
		C45'-C43-C46'	125(3)

Isocyanide # 2 (Occupancy 0.30)

C41-N42	1.146(10)	C41-N42-C43"	153.9(19)
N42-C43"	1.57(4)	N42-C43"-C44"	109(4)
C43"-C""	1.35(8)	N42-C43"-C45"	99(3)
C43"-C45"	1.75(7)	N42-C43"-C45"	94(3)
C43"-C46"	1.67(7)	C44"-C43"-C45"	148(4)
		C44"-C43"-C46"	97(4)
		C45"-C43"-C46"	91(3)

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L1175-14

Table S7 : Bond Distances and Angles for the SiMe3, t-BuNC and PEt3 ligands for $Rh[CC(SiMe_3)N_2N(^t\text{-}Bu)](^t\text{-}BuNC)_2(PEt_3]$, **5**

P-C21[a]	1.797(20)	P-C21'[a]	1.87(3)
P-C23[a]	1.826(14)	P-C23'[a]	1.99(4)
P-C25[a]	1.901(15)	P-C25'[a]	1.76(4)
Si-C2	1.784(8)	Si-C10	1.792(10)
Si-C11	1.817(9)	Si-C12	1.815(10)
C6-C7	1.562(10)	C6-C8	1.554(11)
C6-C9	1.566(10)	N42-C41	1.146(10)
N32-C31	1.142(11)	N42-C43[a]	1.40(2)
N32-C33[a]	1.56(2)	N42-C43"[a]	1.57(4)
N32-C33'[a]	1.35(4)		
Rh-P-C21	116.8(7)	Rh-P-C21'[a]	119.6(9)
Rh-P-C23	114.9(4)	Rh-P-C23'[a]	114.0(11)
Rh-P-C25	113.9(5)	Rh-P-C25'[a]	121.7(11)
C2-Si-C10	113.1(4)	C10-Si-C11	109.3(5)
C2-Si-C11	108.1(4)	C10-Si-C12	107.6(5)
C2-Si-C12	109.3(4)	C11-Si-C12	109.6(5)
N5-C6-C7	109.6(5)	N5-C6-C8	108.8(5)
N5-C6-C9	110.9(5)	C7-C6-C8	109.9(6)
C8-C6-C9	110.1(6)	C31-N32-C33'[a]	169.7(14)
Rh-C31-N32	177.2(7)	C41-N42-C43"[a]	153.9(19)
C31-N32-C33	167.9(10)	C41-N42-C43	164.9(13)

[a] Bonds and angles involving disordered atoms