Inorganic Chemistry
including bioinorganic chemistry
Inorg. Chem., 1996, 35(9), 2538-2545, DOI:10.1021/ic9509793

P-2545-M1

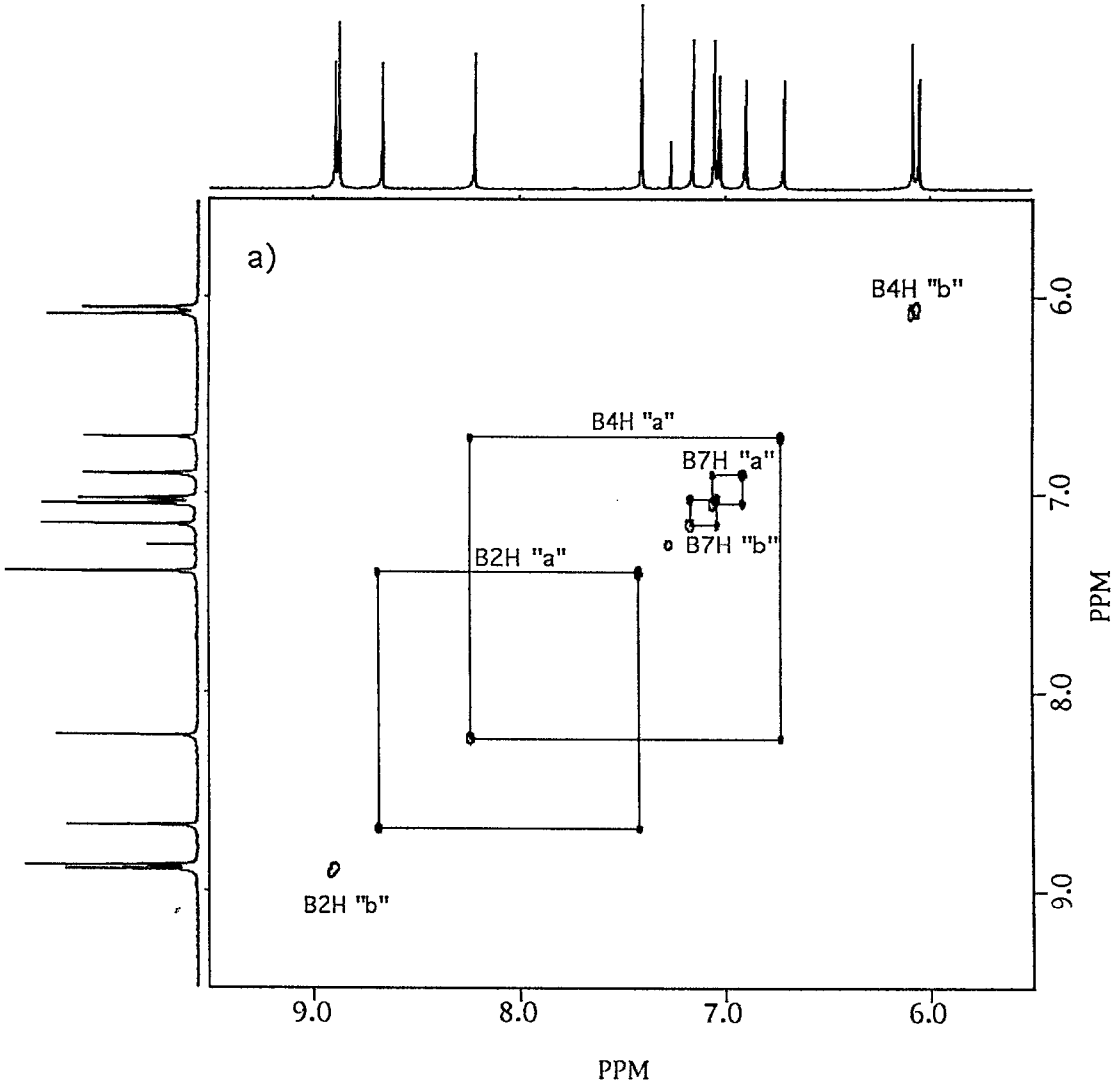
Assignment of the NMR Signals for *cis,cis,cis*-$RuCl_2(Me_2SO)_2(Me_3Bzm)_2$. The $B12H_3$ I signal at 3.95 ppm (assigned to ligand "b") has intense NOE cross-peaks to two signals, "b" B2H I and "b" B7H I. "b" B7H I was assigned from its second NOE to a methyl signal at 2.16 ppm, "b" $B11H_3$ I. "b" $B11H_3$ I has an intense NOE to a methyl signal at 1.70 ppm assigned to "b" $B10H_3$ I. The unassigned signal at 6.07 ppm is "b" B4H I, which possesses an expected NOE to "b" $B10H_3$ I. This same pattern was seen for the second set of I signals. "a" $B12H_3$ I at 3.90 ppm has an NOE to two signals, "a" B2H I and "a" B7H I. The methyl signal with an NOE to "a" B7H I was assigned to "a" $B11H_3$ I at 2.11 ppm This signal in turn had an NOE to another methyl signal (1.71 ppm) assigned to "a" $B10H_3$ I. "a" $B10H_3$ I had an NOE to the final unassigned I signal (6.73 ppm), assigning it to "a" B4H I. $B12H_3$ II resonating at 3.92 ppm (assigned to ligand "b") has NOE's to signals at 8.90 ppm and 7.17 ppm, "b" B2H II and "b" B7H II, respectively. The more upfield signal has an additional NOE to a methyl signal at 2.31 ppm distinguishing it as "b" B7H II. The 2.31 ppm signal is "b" $B11H_3$ II. "b" $B11H_3$ II has an NOE to "b" $B10H_3$ II (1.85 ppm). This signal in turn has an NOE to the 6.11 ppm signal, "b" B4H II. The final B12H signal ("a" II) at 3.36 ppm has NOE's to "a" B2H II and "a" B7H II. As per the above arguments, "a" B7H II was assigned to the more upfield signal at 7.06 ppm and "a" B2H II to the more downfield signal at 7.43 ppm. The more upfield signal has an NOE to a methyl signal at 2.41 ppm, assigning this signal to "a" $B11H_3$ II. The final unassigned methyl signal at 2.48 ppm is "a" $B10H_3$ II. The expected NOE between "a" $B10H_3$ II and "a" $B11H_3$ II cannot be seen due to the close proximity of the NOE to the diagonal. As expected, "a" $B10H_3$ II has an NOE to a more downfield signal at 8.25 ppm, "a" B4H II. Of note, slightly less intense exchange-NOE's were also seen between rotamers. For example, an exchange-NOE is present between "a" B2H I and "a" $B12H_3$ II. These NOE's result from dipole-dipole interactions which remain after exchange between I and II. These exchange NOE's were distinguished by the lower intensity compared to direct NOE's.

Supplementary Figure Captions

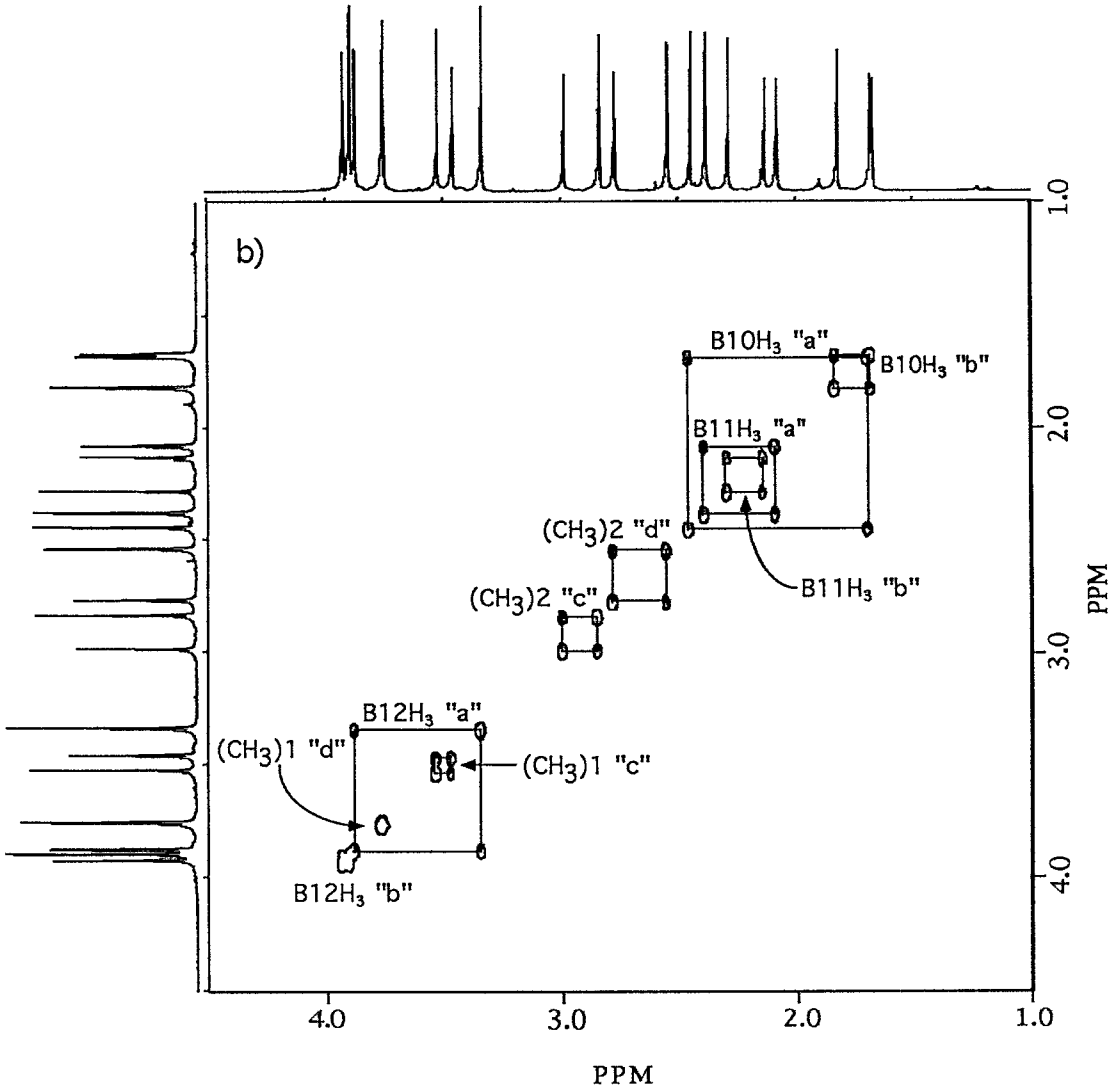
Figure S1. 2D ^{1}H Homonuclear EXSY spectrum of *cis,cis,cis*-$RuCl_2(DMSO)_2(Me_3Bzm)_2$ (**2**): a) downfield correlations, and b) upfield correlations. See Schemes 1 and 2 for Me_3Bzm notation and ligand positions, respectively.

Figure S2. 2D ^{1}H Homonuclear ROESY spectrum of *cis,cis,cis*-$RuCl_2(Me_2SO)_2(Me_3Bzm)_2$ (**2**). Assignment paths for II ligands "a" (——), "b" (- - - -), "c" (·····), and "d" (·····) are depicted. See Scheme 1 for Me_3Bzm notation and NOE connectivity path. Scheme 2 shows ligand positions.

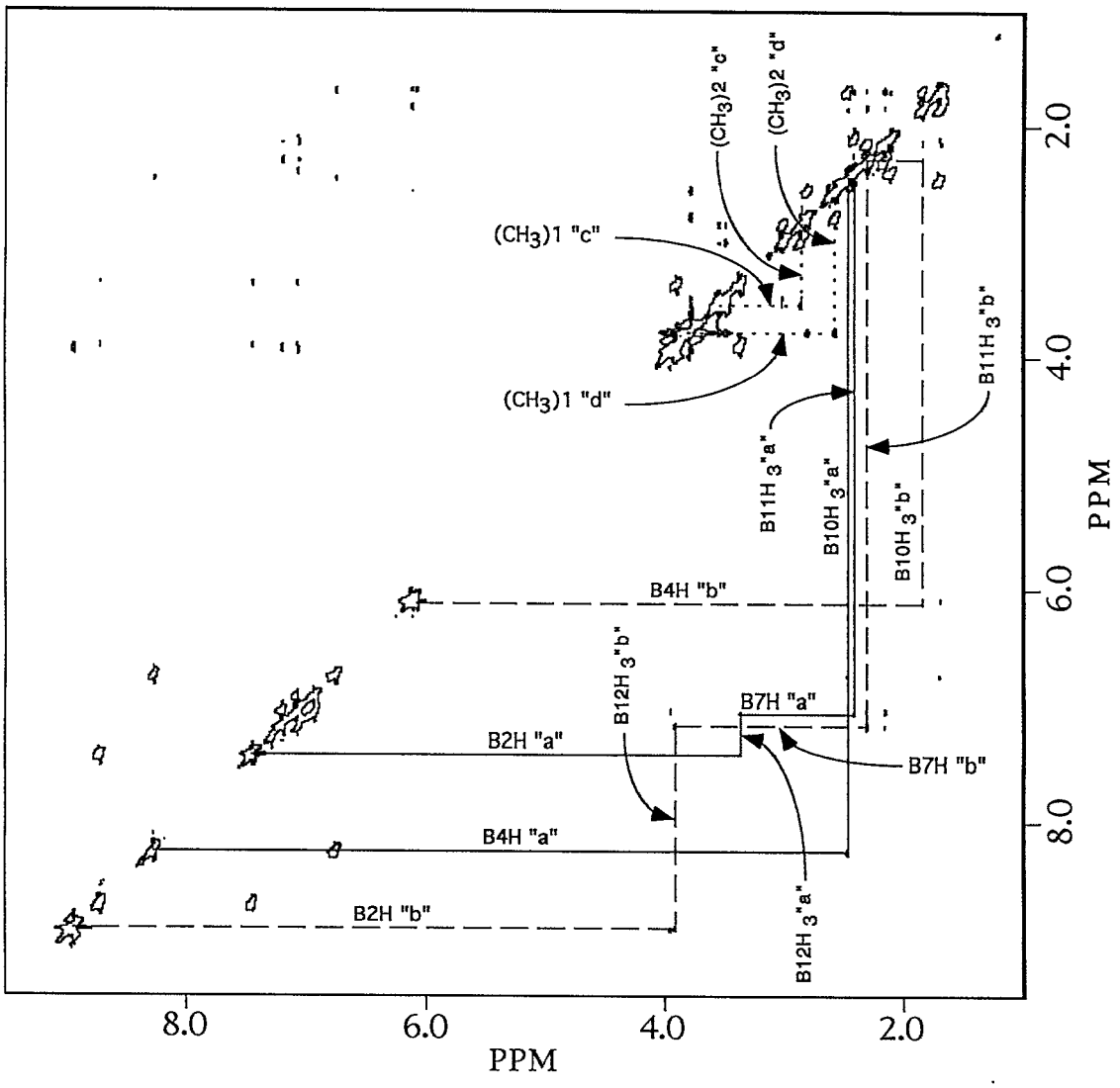
P-2545-M3



P-2545-my



P-2545-M5



P-2545-M6

Table of Bond Distances of 1 in Angstroms

Atom 1	Atom 2	Distance	Atom 1	Atom 2	Distance
Ru	Cl1	2.4148(7)	N3	C10	1.343(3)
Ru	Cl2	2.4233(6)	N3	C11	1.454(3)
Ru	S1	2.2576(7)	N3	C12	1.380(3)
Ru	S2	2.2541(6)	C5	C6	1.381(4)
Ru	N1	2.110(2)	C6	C7	1.368(4)
Ru	N2	2.124(2)	C7	C8	1.361(4)
S1	O1	1.479(2)	C8	C9	1.378(4)
S1	C1	1.781(3)	C12	C13	1.397(3)
S1	C2	1.783(3)	C12	C19	1.390(3)
S2	O2	1.478(2)	C13	C14	1.379(3)
S2	C3	1.782(3)	C14	C15	1.507(3)
S2	C4	1.781(4)	C14	C17	1.416(3)
N1	C5	1.349(3)	C16	C17	1.506(3)
N1	C9	1.344(3)	C17	C18	1.384(3)
N2	C10	1.325(3)	C18	C19	1.398(3)
N2	C19	1.399(3)			

Numbers in parentheses are estimated standard deviations.

P-2545-M7

Table of Bond Angles of 1 in Degrees

Atom 1	Atom 2	Atom 3	Angle	Atom 1	Atom 2	Atom 3	Angle
Cl1	Ru	Cl2	88.89(2)	Ru	N1	C9	122.6(2)
Cl1	Ru	S1	93.27(2)	C5	N1	C9	116.7(2)
Cl1	Ru	S2	87.70(2)	Ru	N2	C10	123.2(1)
Cl1	Ru	N1	176.20(6)	Ru	N2	C19	131.4(1)
Cl1	Ru	N2	87.29(5)	C10	N2	C19	105.0(2)
Cl2	Ru	S1	89.95(2)	C10	N3	C11	126.4(2)
Cl2	Ru	S2	174.96(3)	C10	N3	C12	107.0(2)
Cl2	Ru	N1	88.07(5)	C11	N3	C12	126.6(2)
Cl2	Ru	N2	86.32(5)	N1	C5	C6	122.4(2)
S1	Ru	S2	93.95(2)	C5	C6	C7	119.7(3)
S1	Ru	N1	89.01(5)	C6	C7	C8	118.5(3)
S1	Ru	N2	176.21(5)	C7	C8	C9	119.6(3)
S2	Ru	N1	95.18(5)	N1	C9	C8	123.0(2)
S2	Ru	N2	89.81(5)	N2	C10	N3	113.0(2)
N1	Ru	N2	90.23(7)	N3	C12	C13	131.9(2)
Ru	S1	O1	117.28(9)	N3	C12	C19	106.3(2)
Ru	S1	C1	113.8(1)	C13	C12	C19	121.8(2)
Ru	S1	C2	113.7(1)	C12	C13	C14	118.2(2)
O1	S1	C1	105.3(1)	C13	C14	C15	119.6(2)
O1	S1	C2	106.4(1)	C13	C14	C17	120.4(2)
C1	S1	C2	98.4(2)	C15	C14	C17	120.0(2)
Ru	S2	O2	120.15(8)	C14	C17	C16	120.2(2)
Ru	S2	C3	113.7(1)	C14	C17	C18	120.8(2)
Ru	S2	C4	111.1(1)	C16	C17	C18	118.9(2)
O2	S2	C3	105.3(1)	C17	C18	C19	118.7(2)
O2	S2	C4	105.3(1)	N2	C19	C12	108.7(2)
C3	S2	C4	98.9(2)	N2	C19	C18	131.3(2)
Ru	N1	C5	120.6(2)	C12	C19	C18	120.0(2)

P-2545-M8

TABLE : General displacement parameter expressions, B(Å).

	B(1,1)	B(2,2)	B(3,3)	B(1,2)	B(1,3)	B(2,3)	B_{eq}
Ru	2.009(5)	3.322(6)	2.217(5)	-0.244(6)	-0.108(4)	0.288(6)	2.537(3)
Cl1	2.42(2)	6.13(3)	5.24(3)	0.71(2)	-0.31(2)	1.42(3)	4.64(1)
Cl2	5.25(3)	4.96(3)	2.47(2)	-1.35(2)	-0.32(2)	-0.01(2)	4.27(1)
S1	2.70(2)	4.42(3)	2.84(2)	0.24(2)	0.12(2)	-0.42(2)	3.33(1)
S2	3.16(2)	4.46(3)	2.83(2)	-1.11(2)	0.08(2)	0.54(2)	3.50(1)
O1	3.68(7)	6.0(1)	2.97(6)	0.05(7)	-0.75(6)	-0.60(7)	4.27(4)
O2	5.85(9)	7.4(1)	3.30(7)	-2.64(8)	-0.17(7)	1.76(7)	5.55(5)
N1	2.70(6)	3.03(7)	2.99(7)	-0.08(6)	0.08(6)	0.12(6)	2.92(3)
N2	2.35(6)	3.13(7)	3.06(7)	-0.28(6)	-0.13(6)	0.25(6)	2.87(3)
N3	3.09(7)	2.86(7)	3.19(7)	0.01(6)	0.06(6)	0.39(6)	3.06(4)
C1	7.4(2)	4.8(1)	5.5(1)	1.2(1)	0.9(1)	-1.2(1)	5.88(8)
C2	3.39(9)	9.8(2)	4.1(1)	0.5(1)	1.38(8)	-1.0(1)	5.71(8)
C3	3.00(9)	6.1(1)	5.5(1)	-1.5(1)	-0.05(9)	-0.7(1)	4.87(6)
C4	5.9(1)	3.9(1)	6.0(1)	-0.8(1)	1.1(1)	0.6(1)	5.21(7)
C5	3.41(9)	3.18(9)	3.61(9)	-0.08(8)	0.80(7)	-0.01(8)	3.37(5)
C6	3.7(1)	4.0(1)	5.6(1)	0.50(9)	1.49(9)	-0.2(1)	4.36(6)
C7	3.6(1)	4.6(1)	6.6(2)	1.3(1)	0.3(1)	0.5(1)	4.93(6)
C8	4.1(1)	4.8(1)	4.9(1)	1.2(1)	-0.1(1)	1.2(1)	4.64(6)
C9	3.19(9)	4.5(1)	3.58(9)	0.37(9)	0.28(8)	0.89(9)	3.76(5)
C10	2.72(8)	3.30(9)	3.12(8)	-0.01(7)	-0.19(7)	0.19(8)	3.08(4)
C11	4.4(1)	3.3(1)	3.64(9)	0.06(9)	0.20(8)	0.65(9)	3.79(5)
C12	2.81(8)	2.83(8)	2.94(8)	-0.04(7)	0.23(7)	-0.17(7)	2.86(4)
C13	3.27(8)	2.91(9)	3.84(9)	-0.53(8)	0.53(7)	-0.16(8)	3.33(4)
C14	2.80(8)	3.20(9)	3.82(9)	-0.37(7)	0.32(7)	-0.81(8)	3.27(4)

P-2545-mq 45

TABLE : General displacement parameter expressions, B(Å).

	B(1,1)	B(2,2)	B(3,3)	B(1,2)	B(1,3)	B(2,3)	B_{eq}
C15	3.04(9)	4.7(1)	5.6(1)	-0.83(9)	0.25(9)	-0.6(1)	4.46(6)
C16	2.95(9)	6.0(1)	4.1(1)	0.2(1)	-0.68(8)	-0.3(1)	4.41(6)
C17	2.70(8)	3.6(1)	3.05(8)	0.10(8)	-0.07(7)	-0.69(8)	3.14(4)
C18	2.75(8)	3.22(9)	2.87(8)	-0.15(7)	-0.02(7)	-0.02(7)	2.97(4)
C19	2.39(7)	2.83(8)	2.85(8)	-0.22(7)	0.13(6)	-0.16(7)	2.70(4)

The form of the anisotropic displacement parameter is:

$\exp[-0.25\{h^2a^2\beta(1,1)+k^2b^2\beta(2,2)+l^2c^2\beta(3,3)+2hkab\beta(1,2)+2hlac\beta(1,3)+2klbc\beta(2,3)\}]$;

where a,b, and c are reciprocal lattice constants.

P-2545-M10

46

TABLE : Positional and thermal parameters of hydrogen atoms.

	x	y	z	B(A^2)
H1	0.2689	0.5403	0.1760	7*
H2	0.1265	0.5440	0.1703	7*
H3	0.1992	0.5474	0.2287	7*
H4	0.0389	0.1866	0.2301	7*
H5	0.0615	0.3357	0.2610	7*
H6	-0.0111	0.3323	0.2027	7*
H7	-0.1100	0.1115	0.1307	6*
H8	-0.0950	0.0430	0.0730	6*
H9	-0.1190	-0.0583	0.1224	6*
H10	0.1941	-0.1700	0.0948	6*
H11	0.0623	-0.2261	0.1011	6*
H12	0.0863	-0.1247	0.0517	6*
H13	0.4166	0.1317	0.0527	4*
H14	0.6034	0.0122	0.0708	5*
H15	0.6529	-0.1019	0.1563	6*
H16	0.5099	-0.0979	0.2211	6*
H17	0.3254	0.0235	0.2008	4*
H18	0.1724	0.4787	0.0222	4*
H19	0.2399	0.6930	-0.0314	4*
H20	0.3304	0.7945	0.0043	4*
H21	0.3792	0.6871	-0.0383	4*
H22	0.5746	0.7046	0.0466	4*
H23	0.7754	0.7134	0.0885	5*
H24	0.7896	0.6782	0.1515	5*

P-2545-M11

47

TABLE : Positional and thermal parameters of hydrogen atoms. (cont.)

	x	y	z	B(A^2)
	-	-	-	-----
H25	0.8287	0.5623	0.1095	5*
H26	0.6770	0.3207	0.2094	5*
H27	0.7772	0.3577	0.1706	5*
H28	0.7414	0.4736	0.2133	5*
H29	0.4791	0.3183	0.1663	3*

For starred atoms $B=1.3B_{eq}$ of the bonded atom.