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# THE SPECTROSCOPIC AND STRUCTURAL CHARACTERIZATION OF trans-HYDRIDO(1,1-DIMETHYLALLENE)BIS-(TRICYCLOHEXYLPHOSPHINE)PLATINUM(II) HEXAFLUOROPHOSPHATE

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#### Summary

The platinum hydride cation, trans- $[PtH(CH_3OH)(P(C_6H_{11})_3)_2]^*$  reacts with 1,1-dimethylallene at room temperature in dichloromethane to lose methanol and form an allene complex which has been characterised by <sup>1</sup>H NMR spectroscopy and X-ray diffraction. Crystals grown from a mixture of hexane and o-dichlorobenzene are monoclinic, space group  $P2_1/n$ , with cell dimensions a 14.807(2), b 29.404(7), c 11.621(2) Å and  $\beta$  90.75(1)°. There are four units of trans- $[PtH(C_5H_8)(P(C_6H_{11})_3)_2]PF_6$ ,  $C_6H_4Cl_2$  in the cell. Three dimensional X-ray data collected by diffractometer techniques have permitted full matrix least-squares refinement to a conventional agreement factor R = 0.052. The platinum atom has a square planar coordination geometry, with the planar allene ligand bonded at an angle of 89.2(6)° to the coordination plane, and Pt-C distances of 2.305(10) and 2.233(10) Å.

### Introduction

Recent studies [1-4] have shown that insertion of an unsaturated species such as an olefin or diene into a transition metal—hydride bond occurs by a prior coordination of the unsaturated species to the transition metal in a site *trans* to the hydride ligand. Although spectroscopic evidence for such hydridoolefin complexes with *trans* geometry is available, structural elucidations of the isolated species are few in number [5,6]. Our investigations [7] of the reaction between the solvated cation *trans*-[PtH(CH<sub>3</sub>OH)(PCy<sub>3</sub>)<sub>2</sub>]<sup>+</sup> (PCy<sub>3</sub> = tricyclohexylphosphine) and a number of methyl-substituted cumulated dienes (allenes) have

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led to the isolation of crystalline samples of some of the *trans*-[PtH(1,2-diene)- $(PCy_3)_2$ ]<sup>+</sup> cationic species. In this paper we describe the spectroscopic and structural characterization of the *trans*-[PtH(1,1-dimethylallene)(PCy\_3)\_2]<sup>+</sup> cation, suitable crystals of which were obtained as the hexafluorophosphate salt, with the inclusion of a molecule of *o*-dichlorobenzene solvent. The spectroscopic evidence for two additional allene products is also presented.

## Experimental

#### Preparation and spectroscopy

The complex trans-[PtH(1,1-dimethylallene)(PCy<sub>3</sub>)<sub>2</sub>]PF<sub>6</sub> (I) was formed from the reaction of equimolar amounts of [trans-PtH(CH<sub>3</sub>OH)(PCy<sub>3</sub>)<sub>2</sub>]PF<sub>6</sub> [8] and 1,1-dimethylallene in dichloromethane at 25°C. Treatment of the solution with n-pentane precipitated white crystals. Similar reactions with 1,2-pentadiene or 2,3-pentadiene in place of 1,1-dimethylallene gave the analogous products.

The <sup>1</sup>H NMR spectrum of I showed that bonding of the substituted allene to the platinum atom occurs through the less sterically-hindered end. This was evident from the resonance of the olefinic protons at  $\delta$  –4.54 ppm, a broad singlet shifted upfield from the free allene value ( $\Delta\delta$  0.52 ppm). Platinum satellites of one-fourth intensity flanked this peak, J(Pt-H) 40 Hz. The resonances of the methyl protons of the allene ligand were overlapped by the cyclohexyl protons at  $\delta$  –1.0 to –2.0 ppm and were not observed. The high field hydride resonance at  $\delta$  3.13 ppm was a 1/2/1 triplet, indicative of the trans arrangement of the phosphine ligands, J(P-H) 12 Hz [9]. This resonance is shifted downfield from the position in the complex trans-[PtH(CH<sub>3</sub>OH)(PCy<sub>3</sub>)<sub>2</sub>]PF<sub>6</sub>, where the ligand is trans to a weakly coordinated solvent molecule [8]. The downfield shift ( $\Delta\delta$  14.7 ppm) of the hydride resonance is indicative of the substantial trans influence of the allene ligand. Platinum complexes with coordinated 1,2-pentadiene and 2,3-pentadiene ligands were also characterized spectroscopically and the results are presented in Table 1.

The IR spectrum showed evidence of the hydride ligand from the Pt—H stretching mode  $\nu(\text{Pt}-\text{H}) 2175 \pm 5 \text{ cm}^{-1}$ . However the characteristic  $\nu_{asym}$ -(C=C=C) of the coordinated allene was not observed in the 1680–1760 cm<sup>-1</sup> region as expected for square-planar complexes [10].

## X-ray crystallography

Colourless crystals of trans-[PtH(1,1-dimethylallene)(PCy<sub>3</sub>)<sub>2</sub>]PF<sub>6</sub> were

## TABLE 1

<sup>I</sup> H NMR DATA	<sup>1</sup> FOR [ <i>trans</i> -PtH(allene)(PCy <sub>3</sub> ) <sub>2</sub> ]PF <sub>d</sub>	COMPLEXES
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Allene	Hydride reso	nance	Allene protons	
	δ (ppm)	J(P-H) (Hz)	δ (ppm)	J(Pt—H) (Hz)
1,1-Dimethylallene	+3.13	12	-4.59(m)	40
1,2-Pentadiene	+5.08	12	-4.26(m)	38
2,3-Pentadiene	+5.04	12	-4.01(m)	38

<sup>a</sup> Spectra run in CDCl<sub>3</sub> at 25°C with TMS reference.

TABLE 2

CRYSTAL DATA

C45H76Cl2F6P3Pt	F.w. 1115.05
Analysis <sup>a</sup> Found (Calc.)	С, 50.62 (50.63): Н, 7.23 (6.96)
a 14.807(2) Å	β 90.75(1)°
6 29.404(7) Å	V 5059.2 Å <sup>3</sup>
c 11.621(2) Å	Z = 4
Density (obsvd) 1.48 g cm $^{-3}$	(calcd.) 1.47 g cm $^{-3}$

<sup>a</sup> Chemalytics, Inc., Tempe, Arizona.

obtained by recrystallization from an o-dichlorobenzene/hexane mixture, and contained a solvent molecule of o-dichlorobenzene per formula unit. Preliminary Weissenberg and precession photographs showed monoclinic symmetry and systematic absences of k = 2n + 1 for 0k0 and h + l = 2n + 1 for h0l. This is only consistent with the monoclinic space group  $P2_1/n$ , a nonstandard setting of space group  $P2_1/c$  [11].

A suitable crystal (0.302 mm  $\times$  0.139 mm  $\times$  0.127 mm) was mounted so that its longest dimension [100] was slightly offset from colinearity with the diffractometer spindle axis. The 12 faces of the crystal were identified by optical goniometry as forms {100}, {010}, and {001}, and faces (110), (011), (110), (011), and (111).

Lattice constants were determined at 25°C from a least-squares refinement \* using the setting angles of 30 high-intensity reflections in the range  $60^{\circ} > 2\theta > 45^{\circ}$ . These reflections were carefully centered using prefiltered Cu radiation ( $\lambda$  1.54056 Å) on a Picker FACS-1 diffractometer. The density of the crystals was determined by flotation in a mixture of CCl<sub>4</sub> and n-pentane. Crystal data are given in Table 2.

Intensity data were measured in the  $\theta - 2\theta$  scan mode at a takeoff angle of 1.8°. The counter was positioned 34 cm from the crystal with an aperture size of 5 × 5 mm<sup>2</sup>. The reflections were scanned at 2° min<sup>-1</sup> from 0.75° below the  $K_{\alpha_1}$  peak to 0.75° above the  $K_{\alpha_2}$  peak. Backgrounds were counted for 10 s at each end of the scan. Copper foil attenuators were automatically inserted for intense reflections. Intensity data with  $h \leq 0, k \geq 0, \pm l$  were collected in three shells out to a 2 $\theta$  maximum of 130°. During data collection five standard reflections were measured after every 250 observations. Their intensities decreased by less than 6% throughout data collection. No corrections were made for this decrease.

The intensities of 7123 reflections were recorded in all. After correction for background, Lorentz and polarization effects, 5103 independent reflections were found with significant intensities  $I > 3\sigma(I)$ , where  $\sigma(I) = [Tc + 0.25(tc/tb)^2 - (B_1 + B_2) + (pI)^2]^{1/2}$ , and Tc = total counts, tc/2tb is the ratio of time spent counting peak intensities to that time spent counting backgrounds,  $B_1$  and  $B_2$ 

<sup>\*</sup> Computing was performed on the DEC PDP-10 and the CDC Cyber 73/14 at the University of Western Ontario. Local versions of the following programs were used: cell refinement and orientation matrix, PICKTT; Fourier syntheses, FORDAP, by A. Zalkin; full-matrix least-squares refinement, WOCLS, a version of J.A. Ibers' NUCLS; function and errors, ORFFE, by W.R. Busing, H.A. Levy and K.O. Martin; and ORTEP for molecular illustrations, by C.K. Johnson.

are the background counts, p is the ignorance factor [12] which was 0.04. An absorption correction was applied to the intensity data [13]. The transmission factors ranged from 0.328 to 0.536 ( $\mu$  70.25 cm<sup>-1</sup> for Cu- $K_{\alpha}$  radiation).

### Structure solution and refinement

The positional parameters for the platinum atom and the two phosphine phosphorus atoms were located from a three dimensional Patterson synthesis. A series of least-squares refinements and difference Fourier synthesis calculations located the remaining 56 non-H atoms. Refinement was carried out by full matrix least-squares techniques. The function minimized is  $\Sigma w(|F_0| - |F_c|)^2$ , where  $|F_0|$  and  $|F_c|$  are the observed and calculated structure amplitudes, and  $w = 4F_0^2/\sigma^2(F_0^2)$ . Scattering factors for neutral Pt, P, F, Cl and C atoms were those of Cromer and Waber [14]. The scattering factors for H were those of Stewart et al. [15]. Anomalous dispersion contributions were included in the calculations of  $F_c$ ; the values for  $\Delta f'$  and  $\Delta f''$  for Pt, P, and Cl were those of Cromer and Liberman [16].

At first the cyclohexyl rings (C—C 1.54 Å), the C atoms of the o-dichlorobenzene solvate (C—C 1.392 Å) and the F atoms of the hexaflorophosphate anion (P—F 1.58 Å) were refined as rigid groups with overall isotropic thermal parameters [17]. With all of the non-H atoms included with isotropic thermal parameters the structure refined to  $R_1 = \Sigma ||F_0| - |F_c|| / \Sigma |F_0| = 0.12$  and  $R_2 = (\Sigma w (|F_0| - |F_c|)^2 / \Sigma w F_0^2)^{1/2} = 0.15$ .

Some disorder was noted in the region of the  $PF_6$  anion. Attempts to refine a disorder model employing two rigid groups were not successful. The best agreement was obtained refining the seven atoms of the anion as individual atoms, with anisotropic thermal parameters.

In a subsequent difference Fourier synthesis, all the cyclohexyl, benzene and methyl H atoms were located at peak heights varying from 0.96(17) to  $0.47(17) e^{A^{-3}}$ . Idealized positions for these atoms were determined assuming  $sp^3$  and  $sp^2$  hybridisation for cyclohexyl and benzene C atoms respectively, and a C-H distance of 0.95 Å. Of the two olefinic H atoms attached to C(1), one was readily located,  $0.8(1) e^{A^{-3}}$  at (0.231, 0.050, -0.057), while the other was less evident at  $0.4(1) e^{A^{-3}}$  at (0.112, 0.038, -0.057). Since the calculation of ideal geometries for these two atoms required arbitrary assumptions, they were not included in the model. In order to define the coordination plane of the Pt atom, a careful search was made for the hydride atom. Two peaks were present in the expected region, each of  $0.7(1) e^{A^{-3}}$ , one 1.87 Å and the other 1.53 Å distant from the Pt atom. The closest non-bonding approach to the former was 5 ClHl, at 2.30 Å, whereas the latter was 2.29 Å from P(1), a distance less than the sum of the Van der Waals radii, 3.1 Å. We therefore tentatively assigned the peak at fractional coordinates (0.150, 0.182, 0.090) to the hydride ligand, though we do not place much confidence in these coordinates. This atom was not included in the model, nor were any attempts made to refine H atom parameters.

The final cycles employed 4871 unique observations and 238 variables, and resulted in values of  $R_1 = 0.052$  and  $R_2 = 0.057$ . In a total difference Fourier synthesis the highest peak of 0.9(1) e<sup>A<sup>-3</sup></sup> at (0.1655, 0.3333, -0.1988) was associated with the o-dichlorobenzene solvent molecule. Other peaks greater

RAMETERS <sup>a</sup>
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ATOMIC POSITIONAL

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**TABLE 3** 

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Atom	X	y	Ņ	$U_{11}^{b}$	$v_{22}$	$U_{33}$	$U_{12}$	$U_{13}$	$U_{23}$
Ъl	1628.7(3)	1289.9(1)	30,2(3)	385(2)	437(2)	438(2)	7 (3)	22(1)	7(3)
P(1)	3186(2)	1357(1)	299(2)	442(15)	412(15)	424(13)	-5(12)	15(10)	-27(11)
P(2)	70(2)	1326(1)	263(2)	408(13)	444(14)	443(13)	16(13)	21(10)	7(13)
P(3)	1640(2)	2791(1)	2973(3)	510(20)	917(26)	869(24)	13(18)	17(17)	-82(21)
CI(1)	1746(4)	4634(2)	-3331(5)	1578(46)	1495(45)	1765(50)	159(36)	209(37)	658(38)
CI(2)	1652(7)	4608(3)	-667(6)	4547(139)	2029(74)	1659(62)	54(78)	-13(71)	-872(56)
C(1)	1662(7)	578(3)	-800(8)	500(65)	508(62)	505(60)	24(50)	28(49)	-65(51)
C(2)	1634(7)	908(4)	-1630(10)	437(66)	623(71)	603(71)	-11(52)	38(52)	-111(60)
C(3)	1632(7)	1091(4)	-2639(11)	454(67)	781(80)	628(79)	-39(59)	25(56)	-40(68)
C(4)	1595(8)	1601(5)	-2910(11)	678(87)	937(103)	810(89)	98(75)	51(67)	196(76)
C(5)	1660(10)	792(5)	-3700(11)	1020(112)	1240(123)	622(83)	-180(92)	115(75)	-93(82)
F(1.)	2697(5)	2766(4)	2964(8)	543(48)	2074(103)	1237(70)	-47 (56)	-26(45)	8(68)
F(2)	1635(7)	2482(5)	4022(12)	1009(78)	2833(157)	2565(148)	140(84)	224(82)	1887(133)
F(3)	1707(8)	3203(4)	3818(10)	1757(105)	2041(123)	1600(100)	62(87)	-209(79)	-1046(94)
F(4)	1659(7)	3126(4)	1946(8)	1294(82)	2112(115)	1215(77)	105(75)	103(62)	650(78)
F(5)	1600(7)	2412(4)	2046(13)	1154(84)	1801(107)	3330(180)	236(74)	-274(95)	-1740(119)
F(6)	590(5)	2821(3)	2971(7)	534(47)	1968(96)	1187(67)	202(54)	100(43)	34(64)
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<sup>a</sup> Estimated standard deviations in this and other tables are given in parentheses and correspond to the least significant digits. The positional and thermal parameters have been multiplied by  $10^4$ . <sup>b</sup>  $U_{ij} = \beta_{ij}/(2\pi^2 a_i^* a_j^*)$  Å<sup>2</sup>. The thermal ellipsoid is given by  $\exp[-(\beta_{1j}h^2 + \beta_{22}h^2 + \beta_{33})^2 + 2\beta_{12}hh + 2\beta_{13}hl + 2\beta_{23}hl)]$ .

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TABLE 4	£ ·						
GROUP	PARAMETERS						
Group	xg <sup>a</sup>	Уg	zg	δ	e	η	
Cy 1	0.3375(3)	0.0843(2)	0.2763(4)	0.088(5)	-2.645(3)	1.234(5)	
Су 2	0.3998(3)	0.2387(2)	0.0298(4)	-1.949(4)	-2.524(4)	2.818(5)	
Су З	0.4571(3)	0.0950(2)	-0.1642(4)	0.859(6)	2.370(3)	1.796(6)	
Cy 4	0.1311(3)	0.0930(2)	0.1736(4)	2.394(6)	2.392(3)	-1.717(6)	
Су 5	0.0791(3)	0.2350(2)	0.0406(4)	-1.131(4)	2.506(4)	2.723(5)	
Су б	-0.0097(3)	0.0764(2)	0.2667(4)	-3.210(5)	2.595(3)	1.230(5)	
Ph	0.1606(5)	0.3723(3)	-0.2054(7)	-3.207(8)	-2.674(9)	1.532(8)	

 $a_{x_g}$ ,  $y_g$  and  $z_g$  are the fractional coordinates of the group origin;  $\delta$ ,  $\epsilon$  and  $\eta$  (radians) are the group orientation angles. See ref. 17.

than 0.5 e Å<sup>-3</sup> corresponded to the hydride ligand, and the two olefinic H atoms, or were of no chemical significance. A listing of atomic positional and thermal parameters is given in Table 3. Group parameters are listed in Table 4, and derived group atom parameters are presented in Table 5. H atom parameters are given in Table 6, and observed and calculated structure amplitudes, as  $10F_0$  vs.  $10F_c$  in electrons can be obtained \*. The error in an observation of unit weight was 1.81 electrons.

#### Description of the structure

The crystals contain discrete ions and molecules of solvent. Atom F(5) of the  $PF_6$  anion is in the vicinity of the probable location of the hydride H atom, as is shown in the stereoview (Fig. 1). The shortest cation—anion distance is 2.53 Å, between F(1) and 5C4H1 \*\*.

For the solvent molecule, the closest approach to the cation is 2.44 Å between 7C5Hl and 5C3Hl. The inner coordination sphere of the cation is shown in Fig. 2, and selected intraionic bond distances and angles are given in Table 7.

The cation has a distorted square planar geometry, with *trans*-phosphine ligands. The dimethylallene ligand is coordinated to the metal primarily through the H<sub>2</sub>C=C double bond, with the bulky  $C(CH_3)_2$  substituent bent back away from the metal atom. The solution <sup>1</sup>H NMR spectrum is consistent with this geometry. The Pt--C(1) and Pt--C(2) distances are 2.305(10) and 2.233(10) Å, significantly different by 5.1  $\sigma$ . These values are longer than those observed in another Pt complex in which the allene ligand is perpendicular to the coordination plane, for in the dimer  $[Cl_2Pt((CH_3)_2C=C=C(CH_3)_2)]_2$  distances of 2.25(2) and 2.07(2) Å were found [18,19]. Shorter distances of 2.107(8) and 2.049(7) Å were observed in  $(P(C_6H_5)_3)_2Pt(CH_2=C=C(CH_3)_2)$ ,

<sup>\*</sup> The table of observed and calculated structure factors has been deposited as NAPS document. Please contact ASIS/NAPS, c/o Microfiche Publications, P.O. Box 3513, Grand Central Station, New York, N.Y. 10017.

<sup>\*\*</sup> All non bonding distances have been calculated assuming C-H 1.05 Å for sp<sup>3</sup> hybridization, and 1.00 Å for sp<sup>2</sup> hybridization at C.



Fig. 1. Stereoview showing the cation and the anion.

in which the allene ligand lies in the plane of the Pt and P atoms [20]. Internuclear distances for allene complexes of Rh and Pd have been tabulated by other authors [10,21].

Two least-squares planes have been calculated, and the results are given in Table 8. The five C atoms of the allene ligand are coplanar within experimental error. The plane of the allene ligand is at an angle of  $89.2(6)^\circ$  to the Pt, P, P plane.

The distances C(1)-C(2) and C(2)-C(3) within the allene ligand are 1.367(14) and 1.290(15) Å, respectively, consistent with values found for other allene ligands [10,21]. The deviation from linearity upon coordination is 20(1)°, and is slightly less than the values ranging from 25 to 40° observed elsewhere [10,21]. The C(4) methyl group in the 1,2-diene ligand is bent away from the bulky phosphine ligands. The bond angles C(2)-C(3)-C(4) and C(4)-C(3)-C(5)are 127(1) and 114(1)°, respectively, and are significantly different from the value expected for  $sp^2$  hybridization at C(3). The C(2)-C(3)-C(5) angle however is 120(1)°. The shortest non-bonding distances between H atoms on C(4) and C(5) with the cyclohexyl H atoms are 2.17 Å with 4C2H2 and 2.33 Å with 3C6H2, respectively. The angles observed and the environments of the methyl groups are very similar to those found in the platinum(0) complex of the same ligand by Yasuoka et al. [20]. In that case the shortest approach was phenyl H atom H(36) to a methyl H atom at 2.50 Å, and steric repulsions were thought to be responsible for the distortions observed.



Fig. 2. Inner coordination sphere of the cation. Atoms are drawn with 50% probability thermal ellipsoids.

DERIVED GR	OUP ATOM PA	RAMETERS							
Atom	×	ĸ	~	Β (Å <sup>2</sup> )	Atom	ĸ	v	N	в (Ų)
Cyclohexyl (1)					Cyclohexyl	(5)			
10(1)	3509(5)	1085(2)	1667(4)	3.5(2)	5C(1)	-317(4)	1910(2)	511(6)	3,9(2)
1C(2)	3248(5)	578(2)	1695(5)	4.5(2)	50(2)	-1320(4)	1960(2)	823(6)	5.0(2)
10(3)	3632(5)	358(2)	2802(6)	5.7(3)	5C(3)	-1498(4)	2447(2)	1253(6)	5.3(3)
AC(4)	3240(6)	601(2)	3859(5)	6.2(3)	5C(4)	-1265(5)	2789(2)	300(7)	5.9(3)
1C(5)	3501(5)	1108(2)	3830(4)	5.9(3)	5C(5)	-262(5)	2740(2)	-11(7)	5,6(3)
1C(6)	3117(5)	1327(2)	2724(6)	4.9(2)	5C(6)	-84(4)	2253(2)	-442(5)	4,2(2)
Cyclohexyl (2)					Cyclohexyl	(6)			
20(1)	3535(4)	1950(2)	487(6)	3.6(2)	6C(1)	-234(5)	1026(2)	1597(4)	3.5(2)
2C(2)	3261(4)	2268(2)	-512(5)	5,0(2)	6C(2)	143(5)	1251(2)	2700(6)	4,9(2)
2C(3)	3447(5)	2765(2)	-165(7)	5.5(2)	6C(3)	238(6)	1007(2)	3761(4)	6.4(3)
2C(4)	4460(5)	2825(2)	109(7)	6.5(3)	6C(4)	41(6)	503(2)	3737(5)	6,4(3)
2C(5)	4734(5)	2507(2)	1108(6)	6.1(3)	6C(5)	337(5)	278(2)	2635(6)	6,0(3)
2C(6)	4548(4)	2010(2)	761(6)	4.8(2)	6C(6)	45(5)	522(2)	1574(5)	4,2(2)
Cyclohexyl (3)					a-Dichlorob	ouzoo			
3C(1)	3881(4)	1060(2)		3.7(2)	7C(1)	1661(7)	4146(3)	-2593(11)	7.3(3)
3C(2)	4840(5)	910(2)	-420(4)	4.9(2)	7C(2)	1626(8)	4119(4)	-1398(10)	10.7(5)
3C(3)	5224(4)	589(2)	-1340(6)	5,2(2)	7C(3)	1571(9)	3698(5)		12.7(6)
3C(4)	5260(4)	839(3)	-2503(5)	6.2(3)	7C(4)	1551(8)	3301(4)	-1515(13)	11.0(5)
3C(5)	4301(5)	990(3)	-2864(4)	5,8(3)	7C(5)	1586(8)	3327(3)	-2710(12)	10,8(5)
3C(6)	3918(4)	1311(2)	1944(5)	4.7(2)	7C(6)	1641(7)	3748(5)	-3249(7)	9,3(4)
Cyclohexyl (4)									
4C(1)	618(4)	1041(2)		3.6(2)					
4C(2)	-706(4)	1315(2)	-1984(5)	4.5(2)					
4C(3)	-1072(5)	1004(3)	-2949(4)	5.3(2)					
4C(4)	-2005(4)	820(3)	-2614(5)	5.8(3)					
4C(5)	-1917(5)	546(2)	-1488(6)	5.5(3)					
4C(6)		856(2)	-523(4)	4,8(2)					

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TABLE 5

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METERS		Atom
ND THERMAL PARA		B (Å <sup>2</sup> )
IONAL (X10 <sup>4</sup> ) A		N
IN ATOM POSIT	مراجع والمراجع	у
6. HYDROGE		×
TABLE		Atom

z B (Å <sup>2</sup> )	-2173 5.16	-1057 3.87		-2190 4.94	-3643 5.77	-3056 5.77	-2505 6.54	-3207 6.54	1278 6 0R			-1592 6,08 412 5.46	1592 6,08 412, 5,46 175 5,46		-1592 6,08 -412, 5,46 175 5,46 1171 4,80 170 5,29	-1592 6,08 -412, 5,46 175 5,46 1171 4,30 170 5,29 1417 5,29	-1592 6,08 -412, 5,46 175 5,46 1171 4,30 170 5,29 1417 5,29 1417 5,29	$\begin{array}{c} -1592 \\ -1592 \\ -412 \\ 175 \\ 5.46 \\ 1171 \\ 4.30 \\ 170 \\ 5.29 \\ 1417 \\ 5.29 \\ 1417 \\ 5.29 \\ 1417 \\ 5.29 \\ 1417 \\ 5.29 \\ 1417 \\ 5.77 \\ 1920 \\ 5.77 \end{array}$	$\begin{array}{c} -1592 \\ -1592 \\ -412 \\ 175 \\ 5.46 \\ 1171 \\ 1.71 \\ 4.30 \\ 5.29 \\ 1417 \\ 5.29 \\ 1417 \\ 5.29 \\ 1417 \\ 5.29 \\ 1417 \\ 5.29 \\ 1416 \\ 5.77 \\ -355 \\ 6.56 \end{array}$	$\begin{array}{c} -1592 \\ -1592 \\ -412 \\ 175 \\ 5.46 \\ 1171 \\ 1.71 \\ 4.30 \\ 5.29 \\ 1417 \\ 5.29 \\ 1417 \\ 5.29 \\ 1417 \\ 5.29 \\ 1417 \\ 5.29 \\ 1416 \\ 5.77 \\ 1920 \\ 5.77 \\ -355 \\ 6.56 \\ 6.56 \\ 6.56 \end{array}$	$\begin{array}{c} -1592 \\ -1592 \\ -412 \\ 175 \\ 5.46 \\ 1171 \\ 171 \\ 1.70 \\ 5.29 \\ 1417 \\ 5.29 \\ 1417 \\ 5.29 \\ 1417 \\ 5.29 \\ 1417 \\ 5.29 \\ 1417 \\ 5.29 \\ 1417 \\ 5.29 \\ 1417 \\ 5.29 \\ 6.66 \\ 5.77 \\ -501 \\ 6.66 \\ 6.$	$\begin{array}{c} -1592 \\ -1592 \\ -412 \\ 175 \\ 5.46 \\ 1171 \\ 177 \\ 170 \\ 5.29 \\ 1417 \\ 5.29 \\ 1417 \\ 5.29 \\ 1417 \\ 5.29 \\ 1456 \\ 5.77 \\ 1920 \\ 5.77 \\ 1920 \\ 5.77 \\ -355 \\ 5.69 \\ 6.56 \\ 5.76 \\ 6.5$	$\begin{array}{c} -1592 \\ -1592 \\ -412 \\ 175 \\ 1171 \\ 175 \\ 5.46 \\ 5.46 \\ 1171 \\ 170 \\ 5.29 \\ 1417 \\ 5.29 \\ 1417 \\ 5.29 \\ 1456 \\ 5.77 \\ 1920 \\ 5.77 \\ 1920 \\ 5.77 \\ 1920 \\ 5.77 \\ -355 \\ 6.66 \\ 6.66 \\ 6.66 \\ 6.106 \\ 6.106 \\ -1104 \\ 4.56 \end{array}$	$\begin{array}{c} -1592 \\ -1592 \\ -412 \\ 175 \\ 175 \\ 5.46 \\ 1171 \\ 171 \\ 170 \\ 5.29 \\ 1417 \\ 5.29 \\ 1456 \\ 5.77 \\ 1920 \\ 5.77 \\ 1920 \\ 5.77 \\ 1920 \\ 5.69 \\ 6.66 \\ 6.56 \\ 6.66 \\ 6.56 \\ -501 \\ 6.20 \\ 6.46 \\ 6.20 \\ 6.46 \\ 6.20 \\ 6.46 \\ 6.20 \\ -640 \\ 4.56 \end{array}$	$\begin{array}{c} -1592 \\ -1592 \\ -412 \\ 175 \\ 5.46 \\ 1171 \\ 171 \\ 170 \\ 5.29 \\ 1417 \\ 5.29 \\ 1456 \\ 5.77 \\ 1920 \\ 5.77 \\ 1920 \\ 5.77 \\ 1920 \\ 5.69 \\ 6.66 \\ 5.77 \\ -501 \\ 6.66 \\ 6.56 \\ -501 \\ 6.66 \\ 6.56 \\ -1104 \\ 4.56 \\ -640 \\ 6.20 \\ 1651 \\ 3.63 \\ 1651 \end{array}$	$\begin{array}{c} -1592 \\ -1592 \\ -412 \\ 175 \\ 1171 \\ 175 \\ 5.46 \\ 5.46 \\ 1417 \\ 1417 \\ 5.29 \\ 1417 \\ 5.29 \\ 1456 \\ 5.77 \\ 1920 \\ 5.77 \\ 1920 \\ 5.77 \\ 1920 \\ 5.66 \\ 6.66 \\ 6.56 \\ 6.66 \\ 6.56 \\ 6.66 \\ 6.56 \\ -1104 \\ 4.56 \\ -640 \\ -640 \\ -640 \\ -610 \\ 6.20 \\ 6.20 \\ 6.56 \\ -640 \\ -512 \\ 5.12 \\ 5.12 \end{array}$	$\begin{array}{c} -1592 \\ -1592 \\ -412 \\ 175 \\ 1171 \\ 176 \\ 5.46 \\ 5.46 \\ 1417 \\ 5.29 \\ 1417 \\ 5.29 \\ 1456 \\ 5.77 \\ 1920 \\ 5.77 \\ 1920 \\ 5.77 \\ 1920 \\ 5.69 \\ 6.66 \\ 6.56 \\ 6.56 \\ 6.66 \\ 6.56 \\ 6.66 \\ 6.50 \\ 6.66 \\ 6.50 \\ 6.66 \\ 6.50 \\ 1651 \\ 6.20 \\ 6.20 \\ 6.77 \\ 777 \\ 777 \\ 777 \\ 777 \\ 780 \\ 780 \\ 712 \\ 512 \\ 5.12 \end{array}$	$\begin{array}{c} -1592 \\ -1592 \\ -412 \\ 175 \\ 1171 \\ 176 \\ 5.46 \\ 5.46 \\ 1417 \\ 5.29 \\ 1417 \\ 5.29 \\ 1456 \\ 5.77 \\ 1920 \\ 5.77 \\ 1920 \\ 5.77 \\ -355 \\ 6.66 \\ 6.56 \\ 6.56 \\ 6.56 \\ 6.56 \\ 6.56 \\ 6.56 \\ 6.56 \\ 6.56 \\ 6.56 \\ 6.56 \\ 1004 \\ 4.56 \\ -1104 \\ 4.56 \\ -640 \\ 6.20 \\ 6.20 \\ 6.20 \\ 6.77 \\ 7.22 \\ 4447 \\ 7.22 \\ 4447 \\ 7.22 \\ 7$	$\begin{array}{c} -1592 \\ -1592 \\ -412 \\ 175 \\ 175 \\ 5.46 \\ 1171 \\ 1417 \\ 1417 \\ 5.29 \\ 1417 \\ 5.29 \\ 1456 \\ 5.77 \\ 1920 \\ 5.77 \\ 1920 \\ 5.77 \\ -355 \\ 6.66 \\ 6.56 \\ 6.56 \\ 6.56 \\ 6.66 \\ 6.56 \\ 6.56 \\ -640 \\ -1104 \\ 4.56 \\ -640 \\ -640 \\ 6.20 \\ 6.72 \\ 1651 \\ 3.63 \\ 2715 \\ 5.12 \\ 2721 \\ 5.12 \\ 3.63 \\ 3$	$\begin{array}{c} -1592 \\ -1592 \\ -412 \\ 175 \\ 175 \\ 5.46 \\ 1171 \\ 1417 \\ 1417 \\ 5.29 \\ 1417 \\ 5.29 \\ 1417 \\ 5.29 \\ 1456 \\ 5.77 \\ -355 \\ 5.69 \\ 6.66 \\ 5.77 \\ -501 \\ 6.66 \\ 5.77 \\ -501 \\ 6.66 \\ 6.56 \\ 6.66 \\ 6.56 \\ -640 \\ -640 \\ -640 \\ -640 \\ -640 \\ -640 \\ -640 \\ -640 \\ -640 \\ -712 \\ 5.12 \\ 2715 \\ 5.12 \\ 2715 \\ 5.12 \\ 2715 \\ 5.12 \\ 2753 \\ 6.74 \\ 6.74 \\ 6.74 \end{array}$	$\begin{array}{c} -1592 \\ -1592 \\ -412 \\ 175 \\ 175 \\ 5.46 \\ 1171 \\ 1417 \\ 1417 \\ 5.29 \\ 1417 \\ 5.29 \\ 1417 \\ 5.29 \\ 1456 \\ 5.77 \\ -355 \\ 5.69 \\ 6.66 \\ 5.77 \\ -501 \\ 6.66 \\ 6.56 \\ 6.66 \\ 6.56 \\ -640 \\ -1104 \\ 4.56 \\ -640 \\ -640 \\ 6.74 \\ 4.56 \\ -640 \\ -640 \\ -722 \\ 3.63 \\ 3.63 \\ 5.12 \\ 2712 \\ 5.12 \\ 2712 \\ 5.12 \\ 2715 \\ 5.12 \\ 2715 \\ 5.12 \\ 2718 \\ 5.12 \\ 5$	$\begin{array}{c} -1592 \\ -1592 \\ -412 \\ 175 \\ 175 \\ 1171 \\ 171 \\ 171 \\ 170 \\ 5.46 \\ 5.46 \\ 5.46 \\ 5.46 \\ 5.29 \\ 1417 \\ 5.29 \\ 1456 \\ -5.77 \\ -5.9 \\ 5.77 \\ -5.9 \\ 6.77 \\ -5.9 \\ 6.76 \\ 6.56 \\ 6.20 \\ 6.66 \\ 6.20 \\ 6.74 \\ 4.56 \\ -640 \\ -640 \\ 6.20 \\ 6.74 \\ 4.56 \\ 5.12 \\ 2715 \\ 5.12 \\ 271 \\ 272$	$\begin{array}{c} -1592 \\ -1592 \\ -412 \\ 175 \\ 175 \\ 1171 \\ 171 \\ 171 \\ 170 \\ 5.46 \\ 5.46 \\ 5.46 \\ 5.29 \\ 1417 \\ 5.29 \\ 1456 \\ 5.77 \\ -5.29 \\ 1651 \\ 5.77 \\ -5.29 \\ 6.77 \\ -5.29 \\ 6.77 \\ -5.29 \\ 6.77 \\ -5.29 \\ 6.74 \\ 4.56 \\ -6.40 \\ -6.40 \\ -6.40 \\ -6.40 \\ -6.40 \\ -6.41 \\ -7.22 \\ 3.63 \\ -6.74 \\ -7.22 \\ 3.63 \\ -7.22 \\ 3.63 \\ -6.74 \\ -6.30 \\ 6.74 \\ 6.30 \\ 6.$	$\begin{array}{c} -1592 \\ -1592 \\ 175 \\ 175 \\ 175 \\ 1171 \\ 175 \\ 5.46 \\ 1171 \\ 1417 \\ 1417 \\ 1417 \\ 5.29 \\ 1417 \\ 5.29 \\ 5.46 \\ 5.77 \\ -355 \\ 6.66 \\ 5.77 \\ -356 \\ 6.66 \\ 6.20 \\ 6.66 \\ 6.20 \\ 6.66 \\ 6.20 \\ 6.12 \\ 2715 \\ 2715 \\ 6.74 \\ 4.56 \\ -6.12 \\ 2715 \\ 3.63 \\ 2715 \\ 3.63 \\ 6.74 \\ 4.56 \\ 6.30 \\ 6.74 \\ 4.56 \\ 6.30 \\ 6.74 \\ 4.56 \\ 6.30 \\ 6.74 \\ 4.56 \\ 6.30 \\ 6.30 \\ 158 \\ 6.74 \\ 4.62 \\ 4.56 \\ 6.30 \\ 6.30 \\ 4.62 \\ 6.80$	$\begin{array}{c} -1592 \\ -1592 \\ 175 \\ 175 \\ 1171 \\ 175 \\ 1171 \\ 176 \\ 1171 \\ 1417 \\ 1417 \\ 1417 \\ 1417 \\ 5.29 \\ 5.46 \\ 5.77 \\ 1920 \\ 5.77 \\ 1651 \\ 5.77 \\ 1920 \\ 6.56 \\ 6.77 \\ 6.56 \\ 6.77 \\ 1920 \\ 6.74 \\ 4.56 \\ -6.12 \\ 2715 \\ 2715 \\ 5.12 \\ 2715 \\ 6.74 \\ 4.56 \\ 6.30 \\ 6.74 \\ 4.56 \\ 6.30 \\ 2620 \\ 6.30 \\ 1588 \\ 6.74 \\ 4.56 \\ 6.30 \\ 2620 \\ 6.30 \\ 2632 \\ 6.30 \\ 2632 \\ 6.30 \\ 4.62 \\ 4.62 \\ 4.62 \\ 4.62 \\ 4.62 \\ 6.30 \\ 4.62 \\ 4.6$	$\begin{array}{c} -1592 \\ -1592 \\ 175 \\ 175 \\ 175 \\ 1171 \\ 175 \\ 5.46 \\ 1171 \\ 1417 \\ 1417 \\ 1417 \\ 5.29 \\ 5.46 \\ 5.77 \\ -569 \\ 5.77 \\ -569 \\ 5.77 \\ -569 \\ 6.56 \\ 5.77 \\ -501 \\ 6.66 \\ 6.20 \\ 6.56 \\ 6.20 \\ 6.66 \\ 6.20 \\ 6.74 \\ 4.56 \\ -6.12 \\ 2715 \\ 5.12 \\ 2715 \\ 3.63 \\ 6.74 \\ 4.56 \\ 6.30 \\ 2632 \\ 6.74 \\ 4.56 \\ 6.30 \\ 2632 \\ 6.30 \\ 1588 \\ 6.74 \\ 4.62 \\ 2632 \\ 6.30 \\ 13.75 \\ -46 \\ 13.75 \\ 13.75 \\ -15 \\ 13.75 \\ -16 \\ 13.75 \\ -16 \\ 13.75 \\ -17 \\ -10 $	$\begin{array}{c} -1592 \\ -1592 \\ 171 \\ 175 \\ 171 \\ 175 \\ 1171 \\ 1171 \\ 1417 \\ 1417 \\ 1417 \\ 1417 \\ 1417 \\ 1417 \\ 1417 \\ 1417 \\ 5.29 \\ 5.46 \\ 5.77 \\ 1920 \\ 5.77 \\ 5.29 \\ 5.77 \\ 1920 \\ 6.66 \\ 6.66 \\ 6.66 \\ 6.74 \\ 4.56 \\ 6.63 \\ 6.74 \\ 4.56 \\ 6.30 \\ 12.30 \\ 13.75 \\ -1176 \\ 12.30 \\ 13.75 \\ -1176 \\ 12.30 \\ -1176 \\ 12.30 \\ -1176 \\ 12.30 \\ -1176 \\ -1176 \\ -1176 \\ -1176 \\ -12.30$	$\begin{array}{c} -1592 \\ -1592 \\ 175 \\ 175 \\ 1171 \\ 1171 \\ 1171 \\ 1171 \\ 1171 \\ 1171 \\ 1171 \\ 1175 \\ 5.46 \\ 5.46 \\ 5.77 \\ -355 \\ 5.77 \\ -356 \\ 5.77 \\ -5.19 \\ 6.56 \\ 5.77 \\ -5.19 \\ 6.50 \\ 6.56 \\ 6.50 \\ 6.56 \\ 6.50 \\ 6.512 \\ 2.72 \\ 2.72 \\ 1.651 \\ 3.63 \\ 6.74 \\ 4.56 \\ 6.30 \\ 2.63 \\ 3.63 \\ 6.74 \\ 4.56 \\ 6.30 \\ 2.63 \\ 6.74 \\ 4.56 \\ 6.30 \\ 2.63 \\ 6.74 \\ 4.56 \\ 6.30 \\ 1.22 \\ 3.63 \\ 6.74 \\ 4.56 \\ 6.30 \\ 1.22 \\ 3.63 \\ 6.74 \\ 4.56 \\ 6.30 \\ 1.23 \\ 5.12 $
y	1402	179	1562	1429	1172	767	1065	627	431	298	1103	688		2010	2010 1900	2010 1900 1748	2010 1900 2477	2010 1900 1748 2477 2504	2010 1900 1748 2477 2504 2730	2010 1900 1748 2477 2504 2730 3089	2010 1900 2477 2504 2504 3089 2949	2010 1900 2477 2477 2504 3089 2949 2949	2010 1900 2477 2477 2504 2604 2949 2949 2192	2010 1900 2477 2477 2504 3089 2796 2192 2192 2219	2010 1900 2477 2477 2504 2604 2949 2949 2192 2192 2192 2193	2010 1900 2477 2477 2504 2604 2949 2949 2192 2192 1033	2010 1900 2477 2477 2504 2604 2796 2192 2192 1033 1229 1561	2010 1900 2477 2477 2504 2604 2949 2949 2192 2192 1033 11229 11229	2010 1900 2477 2477 2504 2604 2796 2192 2192 1033 1144 1144 11229	2010 1900 2477 2477 2604 2604 2796 2192 2192 1033 1144 11229 11229 11229 11229	2010 1748 2477 2504 2504 2730 2796 2730 2796 2219 2219 2219 2219 2219 2219 2219 22	2010 1748 2477 2504 2504 2730 2796 2219 2219 2219 2219 2219 2219 2219 22	2010 1748 2477 2504 2504 2730 2796 2192 2192 2192 2192 11244 11229 1223 1229 1229 297 297	2010 1748 2477 2504 2504 2504 2730 2504 2192 2192 2192 2192 2192 2192 2192 219	2010 1748 2477 2504 2504 2504 2730 21948 2192 2192 2192 2192 2192 2192 2192 219	2010 1748 2477 2504 2504 2504 2504 2504 2504 2519 2519 2519 2519 2519 255 255 255 255 255 255 255 255 255 25	2010 1748 2504 2504 2504 2504 2949 2949 2033 2949 1023 1023 2949 1023 2033 2950 2957 2957 2957 2957 2957 29565 29565 2957 2957 2005 2005 2005 2005 2005 2005 2005 20	2010 1748 2504 2504 2504 2504 2089 2089 2019 2019 2019 2029 2029 2029 2029 202
×	3325	-277	-1108	-129	-1123	-665	2408	-2229	-2490	-1511	-1954	-1496		10	10 1688	10 -1688 -1464	10 1688 1464 2112	10 1688 1464 2112 1128	10 -1688 -1464 -2112 -1128 -1631	10 -1688 -1464 -1464 -1128 -1128 -1631	10 -1688 -1464 -1464 -1128 -1128 -1371 -1371	10 -1688 -1464 -1464 -1128 -1128 -1128 -1371 -118 -118	10 -1688 -1464 -1464 -1128 -1128 -1128 -1128 -1128 -1128 -1128 -1128 -116	10 -1688 -1464 -1464 -1128 -1128 -1128 -1128 -1128 -1128 -1128 -1128 -116 -118 -116 -116	-10 -1688 -1464 -1464 -1128 -1128 -1128 -1128 -1128 -1128 -1128 -1371 -118 -118 -116 -118 -116 -1880	-10 -1688 -1464 -1464 -1128 -1	-10 -1688 -1464 -1464 -1128 -1128 -1128 -1128 -1128 -1128 -1128 -1128 -1280 -134 -134	-10 -1688 -1464 -1464 -1128 -1128 -1128 -1128 -1128 -1128 -1371 -134 -880 -134 -880 -134		-10 -1688 -1464 -1464 -1128 -1128 -1128 -1128 -1128 -1128 -1128 -1371 -118 -138 -880 -881 -881 -881 -1681 -1681 -1681 -1681 -1681 -1682 -1688 -1688 -1688 -1688 -17888 -1788 -1788 -1788 -1788 -1788 -1788 -1788 -1788 -1788 -1788 -	-10 -1688 -1464 -1464 -1128 -1128 -1128 -1128 -1128 -1128 -1128 -1281 -128 -134 -134 -192	10 -1688 -1464 -1464 -11631 -1128 -1128 -1128 -1128 -1128 -1128 -1371 -1128 -1128 -1128 -1128 -1128 -1128 -11631 -11631 -11631 -1166 -1166 -11631 -1168 -11631 -1166 -1168 -1166 -1168 -1168 -1168 -1166 -1168 -1168 -1166 -1168 -1166 -1168 -1168 -1168 -1168 -1166 -1168 -1166 -1168 -1166 -1168	$\begin{array}{c} 10\\ -1688\\ -1464\\ -1464\\ -11631\\ -1128\\ -1371\\ -1128\\ -1371\\ -1631\\ -106\\ -1880\\ -880\\ -1880\\ -192\\ -166\\ -192\\ -978\end{array}$	$\begin{array}{c} 10\\ -1688\\ -1464\\ -1464\\ -1371\\ -1128\\ -1371\\ -1371\\ -1371\\ -1681\\ -880\\ -880\\ -880\\ -881\\ -880\\ -881\\ -978\\ -978\\ -978\\ -978\\ -681\\ -978\\ -681\\ -978\\ -681\\ -978\\ -681\\ -978\\ -681\\ -978\\ -916\\ -978\\ -916\\ -978\\ -916$	$\begin{array}{c} 10\\ -1688\\ -1464\\ -1464\\ -1464\\ -1128\\ -1371\\ -1128\\ -1371\\ -1631\\ -1378\\ -138\\ -138\\ -198\\ -192\\ -192\\ -192\\ -192\\ -192\\ -192\\ -192\\ -192\\ -192\\ -192\\ -192\\ -192\\ -192\\ -192\\ -192\\ -192\\ -10$	$\begin{array}{c} 10\\ -1688\\ -1464\\ -1464\\ -1464\\ -1128\\ -1631\\ -1631\\ -1631\\ -1681\\ -880\\ -880\\ -880\\ -880\\ -881\\ -880\\ -978\\ -978\\ -978\\ -916\\ -918\\ -192\\ -192\\ -192\\ -192\\ -192\\ -108$	$\begin{array}{c} 10\\ -1688\\ -1688\\ -1464\\ -1688\\ -1371\\ -1631\\ -1631\\ -1631\\ -1688\\ -138\\ -880\\ -880\\ -880\\ -880\\ -880\\ -106\\ -681\\ -192\\ -192\\ -192\\ -110\\ -192\\ -110\\ -100$	$\begin{array}{c} 10\\ -1688\\ -1688\\ -1688\\ -1681\\ -1681\\ -1681\\ -1681\\ -1681\\ -168\\ -880\\ -880\\ -880\\ -880\\ -168\\ -192\\ -166\\ -192\\ -1681\\ -192\\ -1681\\ -$
Atom	3C6H2	4CIH1	4C2H1	4C2H2	4C3H1	4C3H2	4C4H1	4C4H2	4C5H1	4C5H2	4C6H1	4C6H2	ECULT1	111100	5C2H1	5C2H2 5C2H2	5C2H1 5C2H2 5C3H1	5C2H1 5C2H2 5C3H1 5C3H1 5C3H2	502H1 502H2 503H1 503H2 604H1	502H1 502H2 503H1 503H2 503H2 504H1 504H2	502H1 502H1 503H1 503H2 503H2 504H1 504H2 505H1	502H1 502H1 502H1 503H2 503H2 504H1 503H1 505H1	502011 502011 502011 502011 502011 502011 502011 502011 502011	50201 50201 50201 50201 50201 50201 50201 50201 50201 50201 50201	502011 502011 502012 503011 503011 503011 503012 503011 503011 60111	502011 502011 502012 503011 503011 503011 503011 503011 603101 603101 603011	502011 502011 502012 503011 503011 503011 503011 503011 603011 603011 603011 603011 603011	6C2H1 5C2H1 5C2H1 5C2H2 5C3H1 5C3H1 5C3H1 5C6H1 6C6H1 6C2H1 6C2H1 6C2H1 6C3H1	502011 502011 502012 502011 502011 502011 502011 602011 602012 602011 602012 602012 602012 602012 602012	502011 502011 502012 502011 502011 502011 502012 602011 602012 6020000000000	502011 502011 502011 502011 502011 502011 502011 602010 602010 602010 602010 6020000000000	502011 502011 502011 502011 502011 502011 502011 602010 602010 602010 602010 602010 6020000000000	6C3H1 6C3H1 5C2H1 5C2H1 5C3H1 5C3H1 5C5H1 5C5H1 6C3H1 6C3H1 6C3H1 6C3H1 6C3H1 6C3H1 6C3H1 6C3H1 6C3H1 6C3H1	502011 502011 502011 502011 502011 502011 502011 602012 6020000000000	502011 502011 502011 502011 502011 502011 502011 602012 6020000000000	5000 5000 5000 5000 5000 5000 5000 500	50000 50000 50000 50000 50000 50000 50000 50000 50000 50000 6000000	5000 5000 5000 5000 5000 5000 5000 500
B (Å <sup>2</sup> )	7.06	7,06	7.06	8.24	8.24	8.24	3.95	4.90	4.90	6.07	6.07	6.73	<b>C L C</b>	57.0	6.32	6.32 6.32 6.32	6.32 6.32 5,21	6.73 6.32 5.21 5.21	6.32 6.32 5.21 5.21 3.79	6.73 6.32 5.21 5.21 5.38 5.38	6.73 6.32 5.21 5.21 5.38 5.38	6.73 6.32 5.21 5.21 5.38 5.38 6.01	6.73 6.32 5.21 5.21 5.38 5.38 6.01	6.73 6.32 5.21 5.21 5.38 5.38 6.01 7.26	6.2.3 6.32 5.21 5.21 5.23 5.38 5.21 5.38 6.01 7.26 6.01	6,5,3 6,5,2 5,21 5,21 5,21 5,21 5,21 6,01 6,01 6,01 6,01 6,01	6.23 6.32 5.21 5.21 5.21 5.21 5.21 6.01 6.01 6.01 6.96 6.96	6,2,3 6,221 5,21 5,21 5,21 5,21 5,26 6,01 6,01 6,01 6,96 6,96 5,12	6.23 6.23 5.21 5.21 5.21 5.21 5.21 6.01 6.01 6.01 6.96 6.96 6.96 5.12	6.23 6.23 5.21 5.21 5.21 5.21 5.21 5.12 6.01 6.01 5.12 5.12 5.12 5.12	6.23 6.23 6.221 5.21 5.21 5.21 5.21 5.21 5.12 6.01 6.01 5.12 5.12 5.12 5.47	6.23 6.23 6.32 5.21 5.21 5.21 5.21 5.21 7.26 6.01 6.01 5.38 5.47 5.47 5.47 5.47	6.23 6.23 6.32 5.21 5.21 5.21 5.21 7.26 6.01 6.01 5.38 5.12 5.12 5.47 5.47 5.47 5.47 5.68	6.23 6.23 6.32 5.21 5.21 5.21 5.21 6.01 6.01 5.28 6.01 5.28 5.47 5.47 5.68 5.47 5.68	6.23 6.23 6.32 5.21 5.21 5.21 5.21 6.01 5.28 6.01 5.47 5.47 5.47 5.47 5.68 5.47 5.68 5.47 5.68 5.47 5.68 5.73	6.23 6.23 6.32 5.21 5.21 5.21 5.21 5.21 5.28 5.21 5.28 5.47 5.47 5.47 5.68 5.47 5.68 5.73 5.68 5.73	6,23 6,23 6,221 5,221 5,221 5,221 5,238 5,238 5,23 5,47 5,47 5,47 5,47 5,47 5,47 5,47 5,47	6,23 6,23 6,221 5,221 5,221 5,221 5,238 5,23 5,23 5,23 6,23 6,23 6,23 6,23
2	-2204	-3374	-3300	4097	-4202	-3482	1738	1682	1045	2819	2817	3847	C 7 L 7	4543	4543 4487	4243 4487 3850	4043 4487 3850 2715	4543 4487 3850 2715 2713	4543 4487 3850 2715 2713 1145	4543 4487 3850 2715 2713 1145 	4543 4487 3850 2715 2713 1145 1173 1685	4543 4487 2715 2713 2713 1145 -685 -795	4543 4487 2715 2713 2713 1145 -1173 -685 -795 483	4543 4487 2715 2713 2713 2713 1145 -145 -1795 -158 -558	4543 4487 2715 2713 2713 2713 145 -145 -1795 -158 -1558 309	4543 4543 3850 2715 2715 2713 -1455 -1455 -1455 -1455 -1455 -1455 -1583 -1558 -1258	4543 4543 8487 8487 2715 2715 2713 - 685 - 685 - 685 - 7685 - 758 - 209 1277 - 1765	4543 48487 8850 2715 2715 2713 2713 -685 -145 -685 -173 -558 -1258 109 109	4543 4543 8487 8487 2715 2715 1145 -685 -685 -685 -758 309 1277 1265 1109 1386	4543 4543 8487 8487 2715 2713 2713 685 -1145 -1173 -558 -1273 109 1277 -940	4543 4543 8487 8487 2715 2715 2713 2713 685 -1145 -1145 -1245 -126	4543 4543 8487 8487 2715 2715 2713 2713 2713 1145 -685 -685 -7558 -7558 -369 11277 109 1277 -940 -347 -347 -347 -347 -347 -347 -347 -347	+543 856 8487 8487 2715 2715 2713 2713 -685 -685 -685 -795 -795 1176 -309 1277 -347 -347 -108	+543 4543 8487 8487 8487 2715 2715 1145 -685 -1173 12558 -558 -558 -558 -1455 -1108 -301 -301 -301 -301 -1403	4543 4543 8487 8487 8487 2715 2713 2713 2713 1145 1145 1145 1265 1108 1265 1108 12630 1108 12630	A543 4543 8487 8487 8487 2715 2713 2713 2713 1145 1145 1145 1145 1265 1108 1265 1108 1265 1108 1265 1108 1265 1108 1265 108 1265 108 1265 108 1265 1265 1265 1265 1265 1265 1265 1265	+543 4543 8487 8487 8487 2715 2713 2713 2713 1145 -1145 -1145 1265 -1108 -309 -304 -1108 -364 -368 -368 -368	4543         4543         8487         8487         8487         8487         8487         8487         1145         1145         1145         1145         1145         1145         1145         1145         1145         1145         1145         1145         1258         1265         1108         1108         1265         1109         1265 <t< td=""></t<>
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Atom	С4Н1 <sup>а</sup>	C4H2	C4H3	C5H1	C5H2	C5H3	1C1H1	1C2H1	1C2H2	1C3H1	1 C3H2	1C4H1		1 C4H2	1 C4H2 1 C6H1	1C4H2 1C5H1 1C5H2	1C4H2 1C5H1 1C5H2 1C6H1	1C4H2 1C5H1 1C5H2 1C6H1 1C6H1	1C4H2 1C5H1 1C5H2 1C5H2 1C6H1 1C6H2 2C1H1	1C4H2 1C5H1 1C5H2 1C5H2 1C6H1 1C6H2 2C1H1 2C2H1	1C4H2 1C5H1 1C5H2 1C6H1 1C6H1 1C6H2 2C1H1 2C2H1 2C2H2	1C4H2 1C6H1 1C6H2 1C6H2 1C6H1 1C6H2 2C1H1 2C2H1 2C2H2 2C3H1	1C4H2 1C6H1 1C6H2 1C6H2 1C6H2 2C1H1 2C2H1 2C2H1 2C3H1 2C3H2 2C3H2	1 (24H2 1 (56H1 1 (56H2 1 (66H2 1 (66H2 2 (21H1 2 (21H1 2 (21H1 2 (23H1 2 (23H1 2 (24H1	1 (24H2 1 (56H1 1 (56H2 1 (66H2 1 (66H2 2 (21H1 2 (21H1 2 (21H1 2 (21H1 2 (21H1 2 (21H1 2 (21H1 2 (21H1 2 (21H1) 2 (21H1	1 (24H2 1 (56H1 1 (56H2 1 (66H2 1 (66H2 2 (21H1 2 (21H1 2 (21H1 2 (21H1 2 (21H1 2 (21H1 2 (21H1 2 (21H1 2 (21H1 2 (21H1) 2 (21H1)	1 (24H2 1 (56H1 1 (56H2 1 (56H2 2 (21H1 2 (22H1 2 (23H1 2 (23H1 2 (24H1 2 (24H1 2 (24H1 2 (24H1 2 (25H1 2 (25H2	1 C4H2 1 C6H1 1 C6H1 1 C6H2 1 C6H2 2 C1H1 2 C2H1 2 C2H1 2 C3H2 2 C3H2 2 C3H2 2 C6H1 2 C6H1 2 C6H1 2 C6H1	1C4H2 1C6H1 1C6H2 1C6H2 1C6H2 2C1H1 2C2H1 2C3H1 2C3H1 2C3H2 2C3H2 2C6H1 2C6H1 2C6H2 2C6H2	1C4H2 1C6H1 1C6H2 1C6H2 1C6H2 2C1H1 2C2H1 2C3H1 2C3H2 2C3H2 2C3H2 2C6H1 2C6H1 2C6H1 2C6H2 3C1H1	1C4H2 1C6H1 1C6H1 1C6H2 1C6H2 2C1H1 2C2H1 2C3H1 2C3H2 2C3H2 2C4H1 2C6H1 2C6H1 2C6H1 2C6H1 3C1H1 3C1H1	1C4H2 1C6H1 1C6H1 1C6H2 1C6H2 2C1H1 2C2H1 2C3H1 2C3H2 2C3H2 2C4H1 2C4H1 2C6H1 2C6H1 2C6H1 3C1H1 3C2H2 3C1H1	1C4H2 1C6H1 1C6H2 1C6H2 1C6H2 2C1H1 2C2H1 2C2H1 2C3H2 2C3H2 2C3H2 2C6H1 2C6H1 2C6H1 2C6H2 3C1H1 3C2H2 3C1H1 3C2H2 3C3H1 3C2H2 3C3H1	1C4H2 1C6H1 1C6H1 1C6H2 1C6H2 2C1H1 2C2H1 2C2H1 2C3H2 2C3H1 2C4H1 2C6H1 2C6H1 2C6H1 2C6H1 3C1H1 3C2H1 3C3H1 3C3H1 3C3H1 3C3H2 3C3H1 3C3H1 3C3H2 3C3H1 3C3H2 3C3H1 3C3H2 3C3H1 3C3H2 3C3H1 3C3H2 3C3H1 3C3H2 3C3H1 3C3H2 3C3H1 3C3H2 3C3H1 3C3H2 3C3H1 3C3H2 3C3H1 3C3H2 3C3H1 3C3H2 3C3H1 3C3H2 3C3H1 3C3H2 3C3H1 3C3H2 3C3H1 2C3H2 2C3H1 2C3H2 2C3H1 2C3H2 2C3H1 2C3H2 2C3H1	1C4H2 1C6H1 1C6H2 1C6H2 1C6H2 2C1H1 2C2H1 2C2H1 2C3H2 2C3H2 2C6H1 2C6H1 2C6H1 2C6H1 3C1H1 3C2H2 3C1H1 3C2H2 3C3H1 3C2H2 3C3H1 3C2H2 3C3H1 3C2H2 3C3H1 3C2H2 3C3H1 3C2H1 3C2H2 3C3H1 3C2H1 3C2H1 3C2H1 3C2H1 3C2H1 3C3H1 3C2H1 3C3H1	1C4H2 1C6H1 1C6H1 1C6H2 1C6H2 2C1H1 2C2H1 2C2H1 2C2H1 2C2H1 2C6H1 2C6H1 2C6H1 2C6H1 3C2H1 3C2H1 3C3H1 3C2H1 3C3H1	104H2 106H1 106H1 106H2 106H2 201H1 202H1 202H1 202H1 202H1 206H1 206H1 206H1 302H1	104H2 106H1 106H1 106H2 106H2 202H1 202H1 202H1 202H1 202H1 206H1 206H1 206H1 302H1 302H1 302H1 302H1 302H1 302H1 302H1 302H1 302H1 302H1 302H1 302H1

TABLE 7

Atoms	Distance (Å)	Atoms	Angle (deg)	
Pt-P(1)	2.332(2)	P(1)-Pt-P(2)	163.79(8)	
Pt-P(2)	2.329(2)	P(1)-Pt-C(1)	96.1(3)	
Pt-C(1)	2.305(10)	P(1)PtC(2)	98.3(3)	
PtC(2)	2.233(10)	P(2)-PtC(1)	96.7(3)	
		P(2)PtC(2)	97.9(3)	
C(1)C(2)	1,367(14)			
C(2)C(3)	1.290(15)	C(1)-Pt-C(2)	35.0(4)	
C(3)C(4)	1.533(17)	C(1)-C(2)-Pt	75.3(6)	
C(3)C(5)	1.516(17)	C(1)-C(2)-C(3)	160(1)	
		C(2)-C(1)-Pt	69.6(6)	
		C(2) - C(3) - C(4)	127(1)	
P(1)-1C(1)	1.838(6)	C(2)-C(3)-C(5)	120(1)	
P(1)-2C(1)	1.831(7)	C(3)-C(2)-Pt	125.1(9)	
P(1)-3C(1)	1.852(7)	C(4)-C(3)-C(5)	114(1)	
P(2)-4C(1)	1.845(6)			
P(2)-5C(1)	1.835(7)	Pt-P(1)-1C(1)	108.9(2)	
P(2)-6C(1)	1.845(6)	Pt-P(1)-2C(1)	111.9(2)	
		Pt-P(1)-3C(1)	115.3(2)	
		1C(1) - P(1) - 2C(1)	104.0(3)	
		1C(1)-P(1)-3C(1)	103.9(3)	
		2C(1)P(1)3C(1)	111.8(3)	
	•	Pt-P(2)-4C(1)	115.9(2)	
		Pt - P(2) - 5C(1)	111.9(2)	
		Pt - P(2) - 6C(1)	109.2(2)	
		4C(1)-P(2)-5C(1)	111.4(3)	
		4C(1) - P(2) - 6C(1)	103,8(3)	
		5C(1)-P(2)-6C(1)	103.6(3)	

## SELECTED INTERATOMIC DIMENSIONS

# TABLE 8

# SELECTED LEAST-SQUARES PLANES

Coefficier	nts of the plane equ	uation, $Ax + By +$	Cz - D = 0		
Plane	A	В	с	D	
1	14.80	0.802	0.051	2.514	 
2	0.105	-21.73	7.829	-2.756	
Deviation	s of atoms from th	e planes (Å).			
Atom	Plane 1	Plane 2			

Atom	Plane 1	Plane 2	
Pt	0.0000(4)	-0.0056(4)	
P(1)		0.075(2)	
P(2)		0.081(3)	
C(1)	-0.004(10)	0.89(1)	
C(2)	0.014(10)	0.48(1)	
C(3)	0.002(11)		
C(4)	-0.010(12)		
C(5)	0.025(14)		

The phosphine ligands are bent towards the probable location of the hydride ligand with a P-Pt-P angle of  $163.79(8)^{\circ}$ . All cyclohexyl rings are in the chair conformation, with a mean P-C(1) distance of 1.841(7) Å. Dimensions within the PF<sub>6</sub> anion show small deviations from  $0_h$  symmetry, with P-F distances ranging from 1.52(1) to 1.566(8) Å, mean 1.55(2) Å, and angle means of 90(3) and  $177(2)^{\circ}$ . The solvent molecule is planar within experimental error, with a mean C-Cl distance of 1.676(6) Å.

It has been noted previously [21] that, in other square planar complexes in which the allene ligand is perpendicular to the coordination plane, the midpoint of the coordinated C=C bond does not lie on the plane. A similar geometry is adopted in this study, Table 8. However, the uncertainty in the position of the hydride ligand precludes detailed comparisons of the present geometry with other studies.

## Discussion

The cation is believed to be representative of the intermediate in the insertion of an allene into a metal—H bond. However, unlike the reaction with allene itself [22], formation of the  $\eta^3$ -allyl product was not observed in this case. This failure of insertion to occur is believed to be due to steric constraints resulting from the methyl substituents on the allene and the bulky phosphine ligands. It has been shown that the *cis* coordination of two PCy<sub>3</sub> ligands involves considerable "intermeshing" of the cyclohexyl rings. For example, the P- Pt- P angle of 100.17(4)° in Pt(PPh<sub>3</sub>)<sub>2</sub>(F<sub>3</sub>CC=CCF<sub>3</sub>) [23] increases to 110.28(6)° in Pt(PCy<sub>3</sub>)<sub>2</sub>(F<sub>3</sub>CC=CCF<sub>3</sub>), and a different conformation is observed for the phosphine substituents [24]. In Pt(PCy<sub>3</sub>)<sub>2</sub>( $\eta^3$ -C<sub>3</sub>H<sub>5</sub>) [25] preliminary indications are that the P-Pt-P angle is 111°. Thus the  $\eta^3$ -allyl product from the insertion of 3-methyl-1,2-butadiene may be too large to be coordinated along with the PCy<sub>3</sub> ligands.

The metal—allene bonding is thought to be similar to metal—olefin bonding, and may be discussed in terms of the Dewar—Chatt—Duncanson [26—28] bonding model with the incorporation of an additional  $\pi$ -back-bonding overlap between the filled metal  $d_{xy}$  orbital and the empty  $\pi^*$  orbital of the uncoordinated double bond [10,21].

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