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THE STRUCTURE OF HEXAFLUOROACETONE AZINE BIS(TRIPHENYLPHOSPHINE)PLATINUM

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Summary

The crystal structure of hexafluoroacetone azine bis(triphenylphosphine)-platinum, $C_{42}H_{30}F_{12}N_2P_2Pt$, has been determined by single-crystal X-ray diffraction techniques. The compound crystallizes in the monoclinic space group $C2/c$ with a 31.155(3), b 13.091(1), c 21.828(2) Å, β 109.23(1)° and $Z = 8$. Manual diffractometer methods (Cu radiation, λ 1.54050 Å) were used to obtain 5046 reflections with $I_{net} \geqslant 8$ counts sec^{-1} at 21°C. The structure was solved by the heavy atom method and was refined, including CH_2Cl_2 at occupancy 0.4 on a crystallographic 2-fold axis, by block-diagonal least-squares methods to $R = 0.056$. Location of the phenyl hydrogen atoms was not attempted. The platinum atom is σ-bonded to one C=N of the azine ligand forming a PtCN three-membered ring. Coordination about platinum consists of a slightly distorted square-planar arrangement of the phosphorus atoms and the bonded carbon and nitrogen atoms of the unsaturated ligand. The observed difference in Pt—P bond lengths is rationalized in terms of the *trans*-influence.

Introduction

Stone and coworkers have isolated a stable, crystalline product from the reaction of *trans*-stilbenebis(triphenylphosphine)platinum with bistrifluoromethyldiazomethane [1]. The IR and NMR spectra of this product, as well as the experimentally determined molecular weight, are consistent with the formulation of the platinum—azine complex (I) for this product. However, a serious difficulty with this structure is that the observed ^{19}F NMR spectrum suggests that the trifluoromethyl groups bonded to the carbon adjacent to platinum are in

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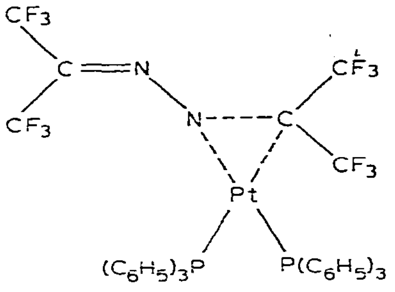

(I)

identical environments. In order to confirm the identity of this complex and to enable a more complete interpretation of the observed spectra, a single-crystal X-ray diffraction study was undertaken. This structure was reported in a preliminary form, together with a rationalization of the observed NMR spectrum [1]. We present here the full details of the structure solution and refinement, and a discussion of the structural results.

Experimental

Pale yellow prismatic crystals for the X-ray study were kindly supplied by Professor F.G.A. Stone of the University of Bristol. One of these was mounted with [010] parallel to the ϕ axis of the goniometer. All X-ray studies were carried out using a General Electric XRD-5 diffractometer equipped with a single crystal orienter, with Cu-K_α radiation, at ambient room temperature (~21°C).

Intensity weighted plots of the reciprocal lattice revealed that the crystal symmetry was $2/m(C_{2h})$ with systematic absences consistent with the two space groups $C2/c$ and Cc. Cell constants listed in Table 1 were obtained from a least-squares refinement of 43 independent 2θ values with $K\text{-}\alpha_1$ and $K\text{-}\alpha_2$ peaks well resolved, measured at 1° takeoff angle with a 0.05° diffracted beam slit.

TABLE 1

EXPERIMENTAL SUMMARY

Crystal data
Formula weight 1047.72, monoclinic
a 31.155(3) Å, b 13.091(1) Å, c 21.828(2) Å
β 109.24(1)°
d_{cald} 1.68 g cm^{-3}, d_{obs} 1.71 ± 0.01 g cm^{-1} (flotation, aqueous $ZnCl_2$)
Systematic absences: hkl, $h + k = 2n + 1$; $h0l$, $l = 2n + 1$; $0k0$, $k = 2n + 1$
Space group: $C2/c$ (No. 15), $Z = 8$
$F(000) = 4163.2e$ [c]
λ(Cu): $K_{\alpha 1}$, 1.54050; $K_{\alpha 2}$, 1.54433 Å
Data collection
Crystal dimensions: 0.184 × 0.177 × 0.164 mm (of the forms {100}, {101} and {111}, respectively)
μ(Cu-K_α) 80.8 cm^{-1} [c]
5793 symmetry independent reflections for $0 < 2\theta \leqslant 115.00°$

[c] Including the methylene chloride molecule on the 2-fold axis with an occupancy factor of 0.4.

The calculated crystal density, assuming eight formula weights of I per unit cell with methylene chloride located on the 2-fold axes of $C2/c$ with an occupancy factor of 0.4 (see below), is 1.68 g cm^{-3}. The average of three independent density determinations, 1.71 ± 0.01 g cm^{-3}, agrees more favorable with the density calculated on the basis of unit occupancy for the methylene chloride (1.72 g cm^{-3}), indicating that its occupancy factor may vary among crystals.

Omega scans (5° take-off angle, 0.05° receiving slit) of reflections along each reciprocal axis revealed peaks that were single, narrow (<0.4° wide) and symmetrical, indicating that the crystal was single and possessed suitable quality for data collection by the stationary-crystal, stationary-counter technique (5° take-off angle, 1° receiving slit) using balanced Ross filters. Periodic monitoring of six reflections revealed variations which were not statistically significant and which did not constitute any trend, indicating that the crystal has suffered neither serious radiation damage nor misalignment during the 18 days of data collection. These data were reduced in the usual way by applying correction factors for Lorentz and polarization effects and for α_1—α_2 splitting [2]. An additional correction for absorption as a function of crystal shape was applied [3]. The calculated values for the absorption correction factors for the data ranged between 2.94 and 5.06. Standard deviations in the observed structure factor amplitudes, $\sigma(|F_o|)$, based on counting statistics, were calculated according to Evans [4].

After refinement of the structure was completed and the correct occupancy factor for the methylene chloride molecule was known, the absorption coefficient, μ, was recalculated. However, checks of 10% of the reflections, distributed throughout the region of reciprocal space covered by the data set, revealed no changes larger than 2% in relative intensities resulting from this alteration in μ. The expected errors in the measured dimensions of the crystal would produce variations of at least this magnitude; therefore, reprocessing of the entire data set followed by a repeated refinement was not considered worthwhile.

The coordinates of the platinum and the two phosphorous atoms could be deduced from a three-dimensional Patterson map assuming the space group $C2/c$, thereby also resolving the space group amibiguity. The remaining 56 non-hydrogen atoms were found in a Fourier map phased by these three heavy atom positions. Refinement of the positional parameters and isotropic temperature factors for these 59 atoms by block-diagonal least squares methods reduced $R = \Sigma||F_o| - |F_c||/\Sigma|F_o|$ to 0.10. A difference density map calculated at this point contained one large peak only 1.4 Å from the 2-fold axis at $(\frac{1}{2}, y, \frac{1}{4})$. This peak was located more than 3.5 Å from any other atom, except its own 2-fold image. The distance between these two peaks is consistent with the known chlorine—chlorine distance in methylene chloride, the solvent from which the sample had been recrystallized. The height of this peak in the map was only about one-half the peak height expected for a chlorine atom. This was also consistent with the observation that there were no prominent peaks in the Patterson map which could be associated with platinum—chlorine interactions. On this basis it was determined that there was a methylene chloride molecule located on the crystallographic 2-fold axis and that the occupancy factor of these sites was approximately 0.5. Because the least squares program did not have the capability of refining occupancy factors, it was decided to complete the refinement of the

TABLE 2

FRACTIONAL ATOMIC COORDINATES AND ANISOTROPIC THERMAL PARAMETERS (× 10^4) [a] FOR THE NON-HYDROGEN ATOMS OF $C_{42}H_{30}F_{12}N_2P_2Pt$. [Estimated standard deviations are given in parentheses. The temperature factor expression was $\exp -(h^2\beta_{11} + k^2\beta_{22} + l^2\beta_{33} + 2hk\beta_{12} + 2hl\beta_{13} + 2kl\beta_{23})$]

	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Pt [a]	13173(2)	12383(4)	3840(2)	79(1)	401(3)	176(1)	1(2)	31(1)	0(2)
P(1)	1112(1)	2763(2)	665(1)	8(0)	45(2)	21(1)	0(1)	4(0)	0(1)
P(2)	1751(1)	691(2)	1411(1)	9(0)	40(2)	18(1)	0(1)	3(0)	−1(1)
N(1)	1333(3)	157(7)	−326(4)	13(1)	42(7)	19(3)	2(3)	5(2)	0(4)
N(2)	1100(3)	−736(8)	−435(5)	12(1)	61(8)	20(3)	1(3)	4(2)	3(2)
C(1)	1041(4)	1018(9)	−585(5)	11(2)	43(10)	22(3)	−1(3)	4(2)	0(5)
C(2)	1316(4)	−1572(9)	−256(5)	12(2)	56(10)	21(3)	−7(3)	6(2)	−9(5)
C(3)	555(5)	872(10)	−881(6)	17(2)	64(12)	28(4)	8(4)	−2(3)	−7(6)
C(4)	1268(5)	1590(11)	−1024(6)	27(3)	67(13)	30(5)	16(5)	11(3)	14(6)
C(5)	1028(5)	−2539(11)	−423(7)	17(2)	70(13)	44(5)	1(5)	9(3)	−4(7)
C(6)	1813(4)	−1746(10)	78(6)	17(2)	61(11)	28(4)	6(4)	9(3)	−1(6)
F(1)	361(2)	414(6)	−490(4)	12(1)	114(8)	43(3)	−6(3)	5(1)	−6(4)
F(2)	419(3)	320(6)	−1434(4)	23(2)	96(8)	31(3)	4(3)	−3(2)	−15(4)
F(3)	324(3)	1752(6)	−1056(4)	19(1)	81(7)	48(3)	10(3)	−3(2)	−8(4)
F(4)	1065(3)	2475(7)	−1219(4)	34(2)	101(9)	66(4)	24(3)	27(2)	46(5)
F(5)	1699(3)	1779(7)	−728(4)	19(1)	116(9)	58(4)	−2(3)	17(2)	19(4)
F(6)	1233(4)	1092(8)	−1568(4)	47(2)	158(11)	29(3)	−4(4)	24(2)	−8(5)
F(7)	2059(2)	−1167(7)	−207(4)	15(1)	99(8)	50(3)	3(3)	11(2)	6(4)
F(8)	1962(3)	−1525(7)	679(3)	18(1)	179(11)	24(2)	19(3)	0(1)	−22(4)
F(9)	1941(3)	−2698(6)	5(4)	18(1)	70(7)	58(3)	11(3)	8(2)	4(4)
F(10)	601(3)	−2341(7)	−607(5)	15(1)	92(8)	94(5)	−13(3)	0(2)	16(5)
F(11)	1109(3)	−3190(6)	59(4)	29(2)	77(7)	43(3)	−5(3)	10(2)	22(4)
F(12)	1112(3)	−3055(7)	−891(4)	40(2)	86(8)	51(3)	−28(3)	20(2)	−32(4)
Cl	4545(3)	930(8)	2448(3)	12(1)	174(12)	7(2)	9(3)	2(1)	2(4)

[a] All values for Pt are presented × 10^5.

original 59 atoms and then to determine the positional and thermal parameters and the occupancy factor for the methylene chloride molecule.

After converting the temperature factors for all atoms, except for the carbon atoms of the phenyl groups, to anisotropic form, the refinement was continued to $R = 0.09$. An electron-density map calculated at this point yielded accurate positional parameters for the carbon and chlorine atoms of the methylene chloride molecule. In order to arrive at the proper occupancy factor for these atoms, electron-density and difference-density maps were calculated corresponding to several occupancy factors for the methylene chloride. From graphical comparisons of the peak heights and peak shapes in these maps the "best" occupancy factor was determined to be 0.4.

Three cycles of isotropic refinement of the other 59 atoms, with the parameters for methylene chloride held constant, produced an R value of 0.07. Addi-

TABLE 3

FRACTIONAL ATOMIC COORDINATES (X 10^4) AND ISOTROPIC THERMAL PARAMETERS FOR $C_{42}H_{30}F_{12}N_2P_2Pt$

	x	y	z	$B(A^2)$
C	5000(0)	1712(33)	2500(0)	3.7(9)
C(7)	1482(4)	4729(10)	1014(6)	4.1(3)
C(8)	1846(4)	5453(10)	1162(6)	4.9(3)
C(9)	2272(4)	5143(11)	1178(6)	5.4(3)
C(10)	2367(4)	4121(10)	1046(6)	5.1(3)
C(11)	2003(4)	3372(9)	886(5)	4.0(3)
C(12)	1575(4)	3712(9)	889(5)	3.6(2)
C(13)	890(4)	2662(9)	1328(5)	3.6(3)
C(14)	592(4)	1832(10)	1285(6)	5.0(3)
C(15)	398(5)	1703(11)	1798(7)	6.4(4)
C(16)	504(5)	2423(12)	2327(7)	6.4(4)
C(17)	809(5)	3235(12)	2336(7)	6.6(4)
C(18)	1012(4)	3375(10)	1842(6)	4.4(3)
C(19)	669(4)	3456(9)	52(5)	3.8(3)
C(20)	801(4)	4101(10)	−370(6)	5.0(3)
C(21)	440(5)	4684(11)	−869(7)	6.2(4)
C(22)	−18(5)	4547(11)	−892(6)	5.8(3)
C(23)	−141(4)	3896(11)	−475(6)	5.4(3)
C(24)	207(4)	3338(10)	14(6)	4.8(3)
C(25)	1806(4)	1357(9)	2163(5)	3.4(2)
C(26)	1555(4)	1034(9)	2568(5)	4.1(3)
C(27)	1605(4)	1557(9)	3162(6)	4.3(3)
C(28)	1904(4)	2390(10)	3339(6)	4.2(3)
C(29)	2158(4)	2706(10)	2969(6)	4.1(3)
C(30)	2109(4)	2201(9)	2368(5)	3.7(3)
C(31)	1544(4)	−547(9)	1573(5)	3.4(3)
C(32)	1109(4)	−840(10)	1182(6)	4.5(3)
C(33)	931(5)	−1799(11)	1331(7)	6.3(4)
C(34)	1169(5)	−2390(11)	1829(6)	5.9(4)
C(35)	1591(5)	−2083(11)	2220(7)	6.0(4)
C(36)	1799(4)	−1149(10)	2092(6)	4.6(3)
C(37)	2344(4)	569(9)	1432(5)	3.1(2)
C(38)	2703(3)	428(9)	2025(5)	3.9(3)
C(39)	3149(4)	347(10)	2024(6)	4.4(3)
C(40)	3246(4)	412(10)	1466(6)	4.9(3)
C(41)	2888(4)	550(11)	883(6)	5.5(3)
C(42)	2435(4)	621(10)	862(6)	4.3(3)

tional least squares calculations, holding the methylene chloride parameters fixed and varying anisotropic thermal parameters for all atoms, except for the phenyl carbon atoms, resulted in an R value of 0.06. Subsequent least-squares calculations, varying the overall scale factor, all positional parameters, isotropic temperature factors for the phenyl carbon atoms and the carbon atom of methylene chloride, and anisotropic temperature factors for the remaining 24 atoms served to complete the refinement at $R = 0.055$ and $R_w = [\Sigma w(|F_o| - |F_c|)^2 / \Sigma w|F_o|^2]^{1/2} = 0.059$. Final shifts in all coordinates were less than 0.4 σ.

A final difference density map contained no peak greater than 0.5 e $Å^{-3}$, except in regions near the platinum, phosphorus and chlorine atoms. The atomic scattering factors used were those of Cromer and Waber [5]. Anomalous dispersion corrections for the platinum, phosphorus and chlorine atoms were included [6]. Only the 5046 reflections with $I_{net} \geqslant 8$ count sec^{-1} were utilized in the refinement. The function minimized in the least squares computation was $\Sigma w(|F_o| - |F_c|)^2$ where $w = \sigma^{-2}(|F_o|)$. Calculations were carried out on a Control Data 6600/6400 computer system. Structure factor and least squares calculations were done using a local modification of a program by Kartha [7]. All Fourier computations were performed using a program by Rao [8]. Various other data processing programs of local origin were also used.

Final atomic parameters are given in Tables 2 and 3 *. Estimated standard deviations were calculated from the inverse matrix of the final least squares cycle.

Discussion

The molecular geometry of I is shown by the stereoscopic drawings [9] in Fig. 1 and corresponds to the proposed platinum—azine complex. These stereoscopic drawings show 25 percent equiprobability ellipsoids derived from the anisotropic thermal parameters.

The three planes defined by the carbon atoms of the phenyl groups attached to P(1) intersect to form angles of 124.6, 117.9, and 100.4°; likewise the three planes defined by the carbon atoms of the phenyl groups attached to P(2) intersect to form angles of 121.7, 120.4, and 113.9°. These relative geometries are representative of those of triphenylphosphines in similar complexes.

Coordination about the platinum atom consists of a distorted square-planar arrangement of the phosphorus atoms and the atoms C(1) and N(1). The angle formed by the interaction of the plane defined by the platinum atom and the two phosphorus atoms with the plane defined by the platinum atom and the atoms C(1) and N(1) is 4.8°. Similar distortions from planarity have been observed in other bistriphenylphosphine complexes with platinum [10,11].

In order to classify the type of bonding between the platinum atom and the

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* A table of observed and calculated structure factor amplitudes is available in the microfilm copy of Dr. Oliver's Ph. D. dissertation, August, 1971, The University of Texas at Austin. Copies may be obtained through Manuscript Publications, University Microfilms, 300 N. Zeeb Road, Ann Arbor, Michigan, 48016 (U.S.A.).

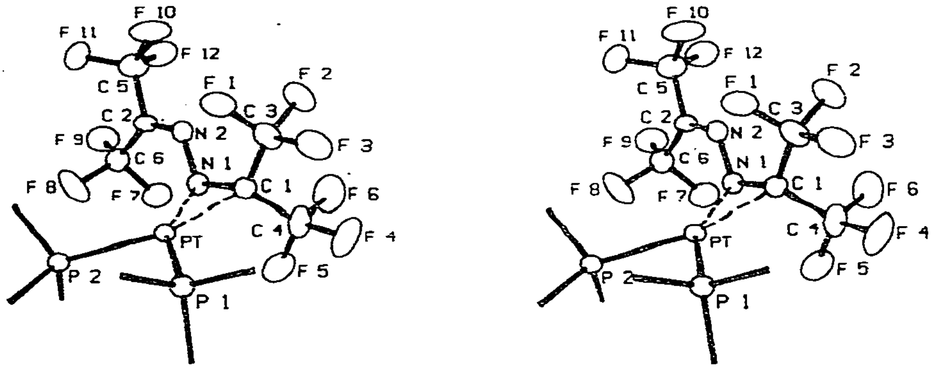

Fig. 1. Stereoscopic view of $C_{42}H_{30}F_{12}N_2P_2Pt$. Phenyl rings are omitted for clarity, but P—C bonds are shown.

atoms C(1) and N(1), consider the three planes A, B, and C defined below:

Plane	Atom	rms, displacement from plane (Å)
A	Pt, C(1), N(1)	0
B	C(1), N(1), C(4)	0
C	N(1), N(2), C(1), C(2), C(3), C(5), C(6)	0.02

The interplanar angles between planes A and V, planes A and C, and planes B and C are 113.7, 119.2, and 127.1°, respectively. In the event that the platinum atom were π-bonded to the organic ligand, the expected values for these interplanar angles would be 90, 90, and 180°, respectively; however, the existance of a σ-bonded three-membered ring would favor a uniform value of 120° for these interplanar angles. It is thus evident that the observed interplanar angles are most consistent with the formulation of the σ-bonded three-membered ring system. An alternative method which has been used [12] for determining the mode of bonding between metal and olefinic ligand is not applicable to I, due to coordination in this compound of platinum to one trivalent atom and one tetravalent atom rather than to two tetravalent atoms.

The bond lengths and bond angles of I are given in Tables 4 and 5. The average values of the phenyl C—C distances, the rms deviation from this average distance and the average value of the estimated standard deviations in these distances are 1.41, 0.03, and 0.02 Å, respectively. These values suggest that, as usual, the deviations calculated from the least-squares matrices are underestimates. The average value of the twelve C—F distances is 1.32 ± 0.03 Å, representative of the values found in perfluoromethyl substituents [13].

The two distances Pt—P(1) and Pt—P(2) are 2.243 and 2.318 Å, respectively. The highly significant difference in these distances (0.075 Å) is attributable to the *trans*-influence [14]. Kinetic studies have indicated that the *trans*-labilizing ability of a methyl group is greater than that of a triphenylphosphine [15]. In agreement with this observation, it is the distance Pt—P(2) opposite C(1) which is the longer distance. This longer P—P distance *trans* to Pt—C is in good agreement with those in the related compound $Pt(CF_3CFCFCF_3)(PPh_3)_2$, Pt—P 2.322(3)

TABLE 4

BOND DISTANCES (Å) [a]

i j	*D(ij)*	*i j*	*D(ij)*	*i j*	*D(ij)*
Pt—P(1)	2.243(3)	C(4)—F(6)	1.33(2)	C(21)—C(22)	1.43(2)
Pt—P(2)	2.318(3)	C(6)—F(7)	1.37(2)	C(22)—C(23)	1.39(2)
Pt—N(1)	2.112(9)	C(6)—F(8)	1.28(2)	C(23)—C(24)	1.45(2)
Pt—C(1)	2.02(1)	C(6)—F(9)	1.33(2)	C(24)—C(19)	1.42(2)
Pt—C(12)	1.84(1)	C(5)—F(10)	1.28(2)	C(25)—C(26)	1.42(2)
P(1)—C(13)	1.80(1)	C(5)—F(11)	1.31(2)	C(26)—C(27)	1.43(2)
P(1)—C(19)	1.82(1)	C(5)—F(12)	1.32(2)	C(27)—C(28)	1.40(2)
P(2)—C(25)	1.82(1)	C—Cl	1.722(9)	C(28)—C(29)	1.37(2)
P(2)—C(31)	1.82(1)	C(7)—C(8)	1.43(2)	C(29)—C(30)	1.43(2)
P(2)—C(37)	1.84(1)	C(8)—C(9)	1.38(2)	C(30)—C(25)	1.43(2)
C(1)—N(1)	1.44(2)	C(9)—C(10)	1.42(2)	C(31)—C(32)	1.40(2)
N(1)—N(2)	1.36(1)	C(10—C(11)	1.45(2)	C(32)—C(33)	1.45(2)
N(2)—C(2)	1.28(2)	C(11)—C(12)	1.41(2)	C(33)—C(34)	1.34(2)
C(1)—C(3)	1.45(2)	C(12)—C(7)	1.41(2)	C(34)—C(35)	1.37(2)
C(1)—C(4)	1.56(2)	C(13)—C(14)	1.41(2)	C(35)—C(36)	1.45(2)
C(2)—C(5)	1.53(2)	C(14)—C(15)	1.45(2)	C(36)—C(31)	1.39(2)
C(2)—C(6)	1.50(2)	C(15)—C(16)	1.44(2)	C(37)—C(38)	1.42(2)
C(3)—F(1)	1.34(2)	C(16)—C(17)	1.42(2)	C(38)—C(39)	1.40(2)
C(3)—F(2)	1.35(2)	C(17)—C(18)	1.43(2)	C(39)—C(40)	1.35(2)
C(3)—F(3)	1.35(2)	C(18)—C(13)	1.41(2)	C(40)—C(41)	1.40(2)
C(4)—F(4)	1.31(2)	C(19)—C(20)	1.41(2)	C(41)—C(42)	1.40(2)
C(4)—F(5)	1.31(2)	C(20)—C(21)	1.49(2)	C(42)—C(37)	1.37(2)

[a] Average C—C (Phenyl) distance = 1.41 ± 0.03 Å.

and 2.302(3) Å, in which both phosphines are *trans* to carbon [16]. The difference in C—Ni distances (1.876(4) Å *trans* to carbon, 1.819(5) Å *trans* to nitrogen) for the two t-BuNC ligands in the ketenimine complex Ni(t-BuN=C=C(CN)$_2$)(t-BuNC)$_2$ has also been discussed by Ibers et al. [17] in terms of metal—ligand back-bonding.

This same *trans*-influence may serve to explain the observed distance Pt—N(1) which is 0.12 Å longer than the average value (1.99 ± 0.05 Å) for the Pt—N distances observed in several structures [18—21]. Kinetic studies have indicated that the *trans*-labilizing ability of triphenylphosphine is greater than that of both pyridine and ammonia [17]. The observed Pt—N distance suggests that triphenylphosphine is also a stronger *trans*-labilizer than is NR$_2$. In agreement with this observation, the distance Pt—P(1) is not significantly different from the Pt—P distance (2.247 Å) observed in the complex *cis*-Pt(P(CH$_3$)$_3$)$_2$Cl$_2$, in which the phosphorous atoms are opposite weak *trans*-labilizers [22].

The distance C(1)—N(1) is not significantly different from the C—N distance observed in azirane (1.488 Å) [23]. The C(2)—N(2) distance and the N(1)—N(2) distance are similar to the analogous C—N distance (1.28 Å) and N—N distance (1.41 Å) observed in anisaldehyde azine [24].

Three of the C—CF$_3$ distances are not significantly different from their average value (1.53 Å). The remaining such distance, C(1)—C(3) (1.45 Å), is 0.08 Å shorter than this average distance. In terms of the Cruickshank criteria [25], this difference is 2.7 σ, but if the estimated standard deviations in these distances are multiplied by the ratio of the observed rms deviation to the average

TABLE 5

BOND ANGLES (°)

i j k	Angle (*ijk*)	*i j k*	Angle (*ijk*)
C(1)—Pt—N(1)	40.7(4)	C(1)—C(4)—F(6)	113(1)
C(1)—Pt—P(1)	110.6(3)	C(2)—C(5)—F(10)	112(1)
C(1)—Pt—P(2)	151.4(3)	C(2)—C(5)—F(11)	114(1)
N(1)—Pt—P(1)	151.1(3)	C(2)—C(5)—F(12)	111(1)
N(1)—Pt—P(2)	110.8(3)	C(2)—C(6)—F(7)	110(1)
P(1)—Pt—P(2)	98.0(1)	C(2)—C(6)—F(8)	116(1)
Pt—P(1)—C(12)	113.8(4)	C(2)—C(6)—F(9)	113(1)
Pt—P(1)—C(13)	111.9(4)	F(1)—C(3)—F(2)	105(1)
Pt—P(1)—C(19)	117.3(4)	F(1)—C(3)—F(3)	105(1)
C(12)—P(1)—C(13)	108.1(5)	F(2)—C(3)—F(3)	103(1)
C(12)—P(1)—C(19)	101.4(5)	F(4)—C(4)—F(5)	108(1)
C(13)—P(1)—C(19)	103.2(5)	F(4)—C(4)—F(6)	105(1)
Pt—P(2)—C(25)	125.2(4)	F(5)—C(4)—F(6)	107(1)
Pt—P(2)—C(31)	109.4(4)	F(10)—C(5)—F(11)	107(1)
Pt—P(2)—C(37)	108.2(4)	F(10)—C(5)—F(12)	108(1)
C(25)—P(2)—C(31)	100.7(5)	C(12)—C(7)—C(8)	118(1)
C(25)—P(2)—C(37)	103.0(5)	C(7)—C(8)—C(9)	120(1)
C(31)—P(2)—C(37)	109.6(5)	C(8)—C(9)—C(10)	112(1)
Pt—C(1)—N(1)	72.9(6)	C(9)—C(10)—C(11)	119(1)
Pt—C(1)—C(3)	120.4(9)	C(10)—C(11)—C(12)	117(1)
Pt—C(1)—C(4)	116.6(8)	C(11)—C(12)—C(7)	124(1)
Pt—N(1)—C(1)	66.4(6)	C(14)—C(13)—C(18)	124(1)
Pt—N(1)—N(2)	124.6(7)	C(13)—C(14)—C(15)	118(1)
C(1)—N(1)—N(2)	111.9(9)	C(14)—C(15)—C(16)	120(1)
P(1)—C(12)—C(7)	119.7(9)	C(15)—C(16)—C(17)	118(1)
P(1)—C(12)—C(11)	116.4(9)	C(16)—C(17)—C(18)	123(1)
P(1)—C(13)—C(14)	114.9(9)	C(17)—C(18)—C(13)	116(1)
P(1)—C(13)—C(18)	121.0(9)	C(20)—C(19)—C(24)	112(1)
P(1)—C(19)—C(20)	117.8(9)	C(19)—C(20)—C(21)	118(1)
P(1)—C(19)—C(24)	119.9(9)	C(20)—C(21)—C(22)	118(1)
P(2)—C(25)—C(26)	120.7(9)	C(21)—C(22)—C(23)	123(1)
P(2)—C(25)—C(30)	120.8(8)	C(22)—C(23)—C(24)	119(1)
P(2)—C(31)—C(32)	117.3(9)	C(23)—C(24)—C(19)	119(1)
P(2)—C(31)—C(36)	121.2(9)	C(30)—C(25)—C(26)	118(1)
P(2)—C(37)—C(38)	121.2(9)	C(25)—C(26)—C(27)	120(1)
P(2)—C(37)—C(42)	118.7(9)	C(26)—C(27)—C(28)	119(1)
N(1)—C(1)—C(3)	120(1)	C(27)—C(28)—C(29)	122(1)
N(1)—C(1)—C(4)	105(1)	C(28)—C(29)—C(30)	120(1)
C(3)—C(1)—C(4)	114(1)	C(29)—C(30)—C(25)	120(1)
F(11)—C(5)—F(12)	105(1)	C(36)—C(31)—C(32)	121(1)
F(7)—C(6)—F(8)	106(1)	C(31)—C(32)—C(33)	118(1)
F(7)—C(6)—F(9)	103(1)	C(32)—C(33)—C(34)	122(1)
F(8)—C(6)—F(9)	108(1)	C(33)—C(34)—C(35)	120(1)
Cl—C—Cl	107(2)	C(34)—C(35)—C(36)	122(1)
N(1)—N(2)—C(2)	119(1)	C(35)—C(36)—C(31)	118(1)
C(1)—C(3)—F(1)	114(1)	C(42)—C(37)—C(38)	120(1)
C(1)—C(3)—F(2)	116(1)	C(37)—C(38)—C(39)	120(1)
C(1)—C(3)—F(3)	113(1)	C(38)—C(39)—C(40)	121(1)
C(1)—C(4)—F(4)	111(1)	C(39)—C(40)—C(41)	119(1)
C(1)—C(4)—F(5)	113(1)	C(40)—C(41)—C(42)	122(1)
		C(41)—C(42)—C(37)	118(1)

esd for the phenyl carbon—carbon distances (1.5), then this difference in the C—CF_3 bond distances becomes 1.8 σ, representing only a possibly significant difference.

No intermolecular contact distances are shorter than the sum of the nonbonded Van der Waals radii for these atoms, indicating no unusual packing effects.

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