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Supporting Information

Ru((*R*)-2,2'-bis(diphenylphosphino)-1,1'-binaphthyl)(H)(MeCN)(THF)$_2$](BF$_4$) - A Catalyst System for Hydrosilylation of Ketones; and for Isomerization, Intramolecular Hydrosilylation, and Hydrogenation of Olefins.

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Experimental Section

General Comments. NMR spectra were recorded on a Bruker AM-400 (^{1}H at 400.1 MHz, ^{13}C at 100.6 MHz, ^{31}P at 161.9 MHz) spectrometer. ^{1}H NMR chemical shifts were measured relative to external TMS and were referenced to residual protons in the deuterated solvent. ^{31}P NMR chemical shifts were measured relative to an 85% H_3PO_4 external reference. GC/IR experiments were conducted on a Hewlett-Packard Model 5965IRD

instrument, employing a J&W Scientific column DB5 (30 m x 0.32 mm internal diameter) having a film thickness of 1.00 μ. The carrier gas was helium and operated at a flow rate of 1.6 mL/min. Elemental analyses were performed by the micro-analytical service within the department. Optical rotations were recorded on a Perkin-Elmer 241 polarimeter at 589 nm.

The solvents, diethyl ether ($LiAlH_4$), n-hexane (CaH_2), methylene chloride (CaH_2), methylene chloride-d_2 (CaH_2), tetrahydrofuran (K, Ph_2CO), tetrahydrofuran-d_8 (K), chloroform-d_1(3Å molecular sieves), acetonitrile (3Å molecular sieves), and acetone (3Å molecular sieves) were distilled from appropriate drying agents under nitrogen or argon gas, except absolute ethanol, which was used as supplied.

The argon and nitrogen gases were dried by passage through a tube containing 3Å molecular sieves and phosphorus pentoxide. The hydrogen gas was passed through an Alltech Oxy-Trap followed by a 3Å molecular sieves-phosphorus pentoxide drying tube.

Unless stated otherwise, all reagents were used as supplied by the Aldrich Chemical Company, Incorporated. The following reagents were distilled under an inert atmosphere (nitrogen or argon) or vacuum (11mm),

depending on their boiling point: (±)-3-buten-2-ol, allyl alcohol, allyl chloride, chlorodimethylsilane, and ethyl acetoacetate. (*Z*)-α-Acetamidocinnamic acid was recrystallized from a boiling ethanol/hexanes mixture, and $[Ph_3C](BF_4)$ was recrystallized from acetonitrile.[1] Cycloocta-1,5-diene was supplied by General Intermediates of Canada and distilled from 3Å molecular sieves before use. Triethylamine and pyridine were furnished by Fisher Scientific and distilled from calcium hydride. (*R*)-(+)-2,2'-Bis(diphenylphosphino)-1,1'-binaphthyl ((*R*)-BINAP) was supplied by Strem Chemicals and recrystallized by established procedures.[2] $[Ru(COD)Cl_2]_n$,[3] $[Ru(COD)(\eta^3\text{-}C_3H_5)_2]$,[4] *cis*-$[Ru(CH_3CN)_2(COD)(\eta^3\text{-}C_3H_5)][BF_4]$,[5] dimethyl(2-propen-1-oxy)silane,[6] (*Z*)-Methyl-α-acetamidocinnamate,[7] (*R*)-(+)-α-methoxy-α-(trifluoromethyl)phenylacetyl chloride (Mosher's acid chloride), and the Mosher's esters[8] were prepared as described previously. All preparations outlined below were conducted under dry, inert conditions (argon or nitrogen) using standard Schlenk techniques. In general, ruthenium compounds were weighed into reaction vessels in an inert atmosphere dry box, and the reaction vessels transferred to a Schlenk line before the introduction of solvent.

[Ru(MeCN)((*R*)-BINAP)(1-3:5,6-η-C_8H_{11})][BF_4] (1). Acetone (25 mL) was added to a reaction vessel containing *cis*-[Ru(CH_3CN)$_2$(COD)(η^3-C_3H_5)][BF_4] (132 mg, 3.15 x 10^{-4} mol) and finely ground (*R*)-BINAP (195 mg, 3.13 x 10^{-4} mol). The suspension was stirred at room temperature for 95 minutes, forming a deep yellow solution. Removal of the solvent under reduced pressure gave a yellow solid discolored by a brown residue. The solids were then dissolved in a mixture of methylene chloride (3.5 mL) and acetonitrile (2 mL). The solution was filtered through filter paper attached to a double-ended needle, and then concentrated by removal of approximately half the solvent under reduced pressure at room temperature. Slow, drop-wise, addition of diethyl ether (10 mL) over a two hour period at room temperature afforded pale yellow needles.[9] The solvent was removed via cannula and the product washed with 3 x 1.5 mL portions of diethyl ether. The yellow solid was dried *in vacuo* for ~24 h to yield 250 mg (83%) of **1**·0.56CH_2Cl_2·0.17MeCN·0.2Et_2O. The product was stored under argon at -15°C. NMR spectroscopic data (CD_2Cl_2) indicated that **1** was isolated as a mixture of a fluxional (at room temp.) and a non-fluxional isomer in a ratio of ~ 0.9 - 1. ^{1}H (400.1 MHz, -78°C) δ -0.57 (br m, 1H),* 0.93 (br m, 1H), 1.27

(br m, 1H), 1.48-1.87 (overlapping m, 4H), 1.66 (s, 3H, CH_3CN),* 1.94 (s, 3H, CH_3CN), 2.21-2.47 (overlapping m, 4H), 2.59-2.82 (overlapping m, 2H), 2.95 (overlapping m, 2H), 3.13 (overlapping m, 2H), 3.38 (overlapping, 2H), 3.83 (m, 1H), 4.76 (m, 1H),* 4.96 (m, 1H), 5.57-8.55 (overlapping m, aromatic); $^{13}C\{^1H\}$ (100.6 MHz, -40 °C) δ 4.61 (s, CH_3CN),* 4.90 (s, CH_3CN), 19.97 (s, CH_2),* 20.62 (s, CH_2), 24.06 (s, CH_2),* 24.75 (s, CH_2), 29.81 (s, CH),* 30.79 (s, CH_2), 35.13 (s, CH),* 35.27 (s, CH_2), 55.28 (s, CH), 61.78 (s, CH), 65.64 (s, CH), 70.46 (d, J_{P-C} = 27.5, CH), 84.70 (s, CH), 89.13 (s, CH), 97.87 (d, J_{P-C} = 10.0 Hz, CH), 115.76 (d, J_{P-C} = 9.1 Hz, CH); $^{31}P\{^1H\}$ (-78 °C) δ 33.44 (d, J_{P-P} = 33.2 Hz, 1P), 35.33 (d, J_{P-P} = 35.6 Hz, 1P),* 45.69 (d, J_{P-P} = 38.6 Hz, 1P),* 46.72 (d, J_{P-P} = 33.2 Hz, 1P). Anal. Calcd. for $C_{54}H_{46}BF_4NP_2Ru \cdot 0.56CH_2Cl_2 \cdot 0.17MeCN \cdot 0.2Et_2O$: C, 65.07; H, 4.87; N, 1.59; Cl, 3.86. Found: C, 65.81; H, 4.76; N, 1.51; Cl. 3.66.

* Signals we assign to the fluxional isomer of **1**.

Hydrogenation of [Ru(MeCN)((*R*)-BINAP)(1-3:5,6-η-C_8H_{11})][BF_4] (1). Compound **1** (10.7 mg, 1.16 x 10^{-5} mol) was dissolved

in a mixture of CD_2Cl_2 (~0.1 mL) and THF-d_8 (~0.5 mL) in an NMR tube to generate a pale-yellow solution. At room temperature, dihydrogen gas (5.0 mL, 2.04 x 10^{-4} mol) was injected into the head-space of the tube, and the mixture shaken for two minutes with intermittent cooling in an ethanol/dry-ice bath giving a deep-yellow solution. Decomposition of the hydrido species was rapid in the presence of methylene chloride at room temperature. Cooling was necessary to prevent the degradation of **2** and **3**. The ruthenium-containing species detected and identified with NMR data obtained at -78 °C were [Ru((*R*)-BINAP)(H)(MeCN)(THF)$_2$][BF_4] (**2**) (44%), [Ru((*R*)-BINAP)(H)(MeCN)$_2$(THF)][BF_4] (15%), and the non-labile isomer of **1** (41%). Cyclooctene and cyclooctane[10] were also detected by ^{1}H NMR spectroscopy (~1:3). Further addition of hydrogen (5.0 mL) and shaking at room temperature for two minutes caused complete hydrogenation of cyclooctene to cyclooctane. The metal species detected in solution were **2** (59%), [Ru((*R*)-BINAP)(H)(MeCN)$_2$(THF)][BF_4] (14%), non-labile **1** (27%), and two other species in amounts too low to integrate (we tentatively identify one as *fac*-[Ru((*R*)-BINAP)(H)(MeCN)$_3$][BF_4] (**3**)). The solution was warmed to room temperature and acetonitrile (3.6 μL, 6.9 x 10^{-5} mol)

added via syringe, causing the yellow color to lighten. The solution was cooled to -78 °C for further ^1H and ^{31}P NMR spectroscopic analyses. The solution contained a product identified by NMR spectroscopy as *fac*-[Ru((*R*)-BINAP)(H)(MeCN)$_3$][BF$_4$] (**3**) (78%), and non-labile **1** (22%). NMR spectroscopic data (THF-d_8/CD_2Cl_2 (5:1)): **2**, ^1H (400.1 MHz, -78°C) δ -13.05 (app t, J = 28.0 Hz, 1H), 2.74 (s, 3H, C*H*$_3$CN), 6.00-8.72 (m, aromatic); $^{31}P\{^1H\}$ (161.9 MHz, - 78 °C) δ 72.22 (d, J_{P-P} = 49.6 Hz, 1P), 81.34 (d, J_{P-P} = 49.8 Hz, 1P); [Ru((*R*)-BINAP)(H)(MeCN)$_2$(THF)][BF$_4$], ^1H (400.1 MHz, -78 °C) δ -12.98 (overlapped by **2**), 1.96 (s, 3H, C*H*$_3$CN), 2.17 (s, 3H, C*H*$_3$CN), 6.00-8.72 (m, aromatic, overlapped by **2**); $^{31}P\{^1H\}$ (161.9 MHz, -78 °C) δ 72.22 (overlapped by **2**, 1P), 76.93 (d, J_{P-P} = 42.3 Hz, 1P); **3**, ^1H (400.1 MHz, -78 °C) δ -13.48 (app t, J = 24.1 Hz, 1H), 1.84 (s, 3H, C*H*$_3$CN), 1.87 (s, 3H, C*H*$_3$CN), 2.22 (s, 3H, C*H*$_3$CN), 6.00-8.85 (m, aromatic); $^{31}P\{^1H\}$ (161.9 MHz, -78 °C) δ 64.22 (d, J_{P-P} = 40.1 Hz, 1P), 69.89 (d, J_{P-P} = 40.5 Hz, 1P).

Catalytic Reactions. All glassware and syringes were treated with NH_4OH/EtOH, acetone, and oven-dried before use. The reactions were

performed under argon gas in rigorously dried solvents. Small-scale reactions were conducted in NMR tubes and monitored by ^{1}H NMR spectroscopy to calculate rates and chemical yields (see Table 1). The enantiomeric excess of each reaction was determined by repeating the experiment on a large-scale, and analyzing the products as described below.

Procedure for Small-scale Catalytic Reactions. Typically, compound **1** (1.2 mg, 1.25 x 10^{-6} mol) was dissolved in tetrahydrofuran-d_8 (0.48 mL) by vigorous mixing for several minutes at room temperature. Dihydrogen gas (5.0 mL, 2.04 x 10^{-4} mol) was injected into the head-space of the NMR tube, and the mixture was shaken (1-2 minutes, room temperature), resulting in an immediate color change from pale- to deep-yellow. The tube was purged with argon, and the substrate (6.25 x 10^{-5} mol) added via syringe.

Procedure for Large-scale Catalytic Reactions. The reactions were performed in a manner similar to that for the small-scale experiments, except that quantities of reactants were increased by a factor of 15, and non-

deuterated solvent[11] was used. Generation of the catalytically active solution was accomplished by bubbling dihydrogen gas (1 atm) through a stirring solution (~2.6 mM) of **1** at room temperature for 2 minutes, followed by purging the solution with argon gas (1 atm) for ~1 minute. The reactor was typically a Schlenk tube, except for the hydrogenation of (*Z*)-Methyl-α-acetamidocinnamate, when a pressure reactor was employed. The substrate was then added via syringe, and the reaction was carried out for the times and temperatures shown in Table 1. The solvents were removed at the end of the reaction by use of either a rotary evaporator, or by distillation at atmospheric pressure, depending on the boiling points of the products. The specific work-ups and product analyses are as follows.

Methyl 2-acetamido-3-phenylpropanoate was the only material detected (by ^{1}H NMR) in the residue obtained from hydrogenation of (*Z*)-methyl-α-acetamidocinnamate. The residue was dissolved in benzene, passed through a silica gel plug to remove the catalyst, and the solvent removed using a rotary evaporator. The absolute configuration of the product was determined by optical rotation.[12] The ee was determined by

mixing tris[3-(trifluoromethyl-hydroxymethylene)-(+)-camphorate europium (III) (12.7 mg, 1.42 x 10^{-5} mol) and methyl 2-acetamido-3-phenylpropanoate (50.1 mg, 2.26 x 10^{-4} mmol) in $CDCl_3$ (0.6 mL) and recording the ^1H NMR spectra. The ratio of the methoxy peaks at $\delta = 3.803$ (*R*), and $\delta = 3.697$ (*S*) were used to determine the ee (86% ee (*R*)). The ratio of these peaks was 1 : 1 for racemic Methyl 2-acetamido-3-phenylpropanoate.[13]

The isomerization of (±)-3-buten-2-ol was monitored by ^1H NMR spectroscopy. (*E*)-2-Buten-2-ol was identified by comparison to a literature spectrum.[14] 2-Butanone was identified by ^1H NMR spectroscopy and by GC-IR (the infrared spectrum was identical to that of an authentic sample). The ee of the remaining 3-buten-2-ol was determined as follows. An aliquot (5 mL) was removed with a cannula from a large-scale reaction mixture 60 minutes after the addition of substrate, and the catalyst was deactivated in the aliquot by bubbling carbon monoxide gas (1 atm) through the solution for 30 seconds. The aliquot was mixed with pentane (~ 20 mL), and passed through a Florisil plug to remove the catalyst. The solvent was removed by distillation under argon gas (T bath = 70 °C), and the residue reacted with

(*R*)-(+)-α-methoxy-α-(trifluoromethyl)phenylacetyl chloride as described in the literature.[8] The enantiomeric excess was determined by comparison of the ^{1}H NMR to that of the Mosher's ester of an authentic sample of (*S*)-(+)-3-buten-2-ol.[15] ^{1}H NMR ($CDCl_3$, 400.1 MHz); Mosher's ester of (+)-3-buten-2-ol (*R,S*) δ 1.44 (d, *J* = 6.5 Hz, 3H, C*H*$_3$CH(O)CH=CH$_2$), 3.58 (d, *J* = 1.2 Hz, 3H, OC*H*$_3$), 5.16 (app dt, *J* = 10.8, 1.4 Hz, 1H, CH$_2$=C*H*), 5.25 (app dt, *J* = 17.2, 1.0 Hz, 1H, *trans*-C*H*H=CH), 5.59 (m, 1H, CH=CHC*H*(O)(CH$_3$), overlapped by (*R,R*) diastereomer), 5.81 (ddd, *J* = 4.5, 10.5, 17.5 Hz, 1H, *cis*-C*H*H=CH), 7.34-7.72 (m, aromatic, overlapped by (*R,R*) diastereomer); Mosher's ester of (+)-3-buten-2-ol (*R,R*), δ 1.37 (d, *J* = 6.5 Hz, 3H, C*H*$_3$CH(O)CH=CH$_2$), 3.56 (d, *J* = 1.2 Hz, 3H, OC*H*$_3$), 5.22 (app dt, *J* = 10.5, 1.2 Hz, 1H, CH$_2$=C*H*), 5.35 (app dt, *J* = 17.0, 1.2 Hz, 1H, *trans*-C*H*H=CH), 5.59 (m, 1H CH=CHC*H*(O)(CH$_3$), overlapped by (*R,S*) diastereomer), 5.90 (ddd, *J* = 4.4, 10.8, 17.5 Hz, 1H, *cis*-C*H*H=CH), 7.34-7.72 (m, aromatic, overlapped by (*R,S*) diastereomer).

The material obtained from hydrosilylation of ethyl acetoacetate by chlorodimethylsilane was hydrolyzed as described in the literature.[16] The

identity of the resulting ethyl 3-hydroxybutyrate was confirmed by comparison of both its ^{1}H NMR spectrum, and its GC-IR to those of an authentic sample (Aldrich). The residue obtained from the hydrolysis was reacted with (*R*)-(+)-α-methoxy-α-(trifluoromethyl)phenylacetyl chloride as described in the literature.[8] The enantiomeric excess was determined by comparison of the ^{1}H NMR to that of the Mosher's ester of authentic ethyl (*R*)-(-)-3-hydroxybutyrate.[17] ^{1}H NMR (400.1 MHz, $CDCl_3$); Mosher's ester of 3-hydroxybutyrate (*R,R*) δ 1.23 (t, J = 7.0 Hz, 3H, OCH_2CH_3), 1.33 (d, J = 6.4 Hz, 3H, $CH_3C(O)H$), 2.56 (dd, J = 16.2, 5.0 Hz, 1H, $CH_3C(O)HCHH$), 2.72 (dd, J = 16.2, 8.5 Hz, 1H, $CH_3C(O)HCHH$), 3.55 (3H, overlapped by (*R,S*) diastereomer), 4.13 (qd, J = 7.5, 1.5 Hz, 2H, OCH_2CH_3), 5.56 (m, 1H, $CH_3C(O)H$), 7.36-7.71 (m, 5H, Ph): Mosher's ester of 3-hydroxybutyrate (*R,S*): δ 1.19 (t, J = 7.1 Hz, 3H, OCH_2CH_3), 1.43 (d, J = 6.4 Hz, 3H, $CH_3C(O)H$), 2.54 (dd, J = 16.2, 5.0 Hz, 1H, $CH_3C(O)HCHH$), 2.68 (dd, J = 16.2, 8.5 Hz, 1H, $CH_3C(O)HCHH$), 3.55 (3H, overlapped by (*R,R*) diastereomer), 4.06 (qd, J = 7.2, 1.0 Hz, 2H, OCH_2CH_3), 5.57 (m, 1H, $CH_3C(O)H$), 7.36-7.68 (m, 5H, Ph).

The isomerization and intramolecular hydrosilylation of dimethyl(2-propen-1-oxy)silane was monitored by ^{1}H NMR spectroscopy. (*Z*)-dimethyl(1-propen-1-oxy)silane,[18] (*E*)-dimethyl(1-propen-1-oxy)silane,[18] mono- and poly(2,2-dimethyl-1-oxa-2-silacyclopentane)[18,19] were identified by comparison to literature spectra.

^{1}H NMR (400.1 MHz, THF-d_8); monomeric 2,2-dimethyl-1-oxa-2-silacyclopentane δ 0.12 (s, 6H, Si(C*H*$_3$)$_2$), 0.70 (t, J = 7.2 Hz, 2H, SiC*H*$_2$CH$_2$CH$_2$), 1.82 (tt, J = 7.5, 6.5 Hz, 2H, SiCH$_2$C*H*$_2$CH$_2$), 3.74 (t, J = 6.5 Hz, 2H, OC*H*$_2$); polymerized 2,2-dimethyl-1-oxa-2-silacyclopentane δ 0.09 (m, 6H, Si(C*H*$_3$)$_2$), 0.87 (m, 2H, SiC*H*$_2$CH$_2$CH$_2$), 1.50 (m, 2H, SiCH$_2$C*H*$_2$CH$_2$), 3.24-3.87 (m, 2H, OC*H*$_2$); (*Z*)-dimethyl(1-propen-1-oxy)silane, δ 0.25 (d, J = 3.0 Hz, 6H, Si(C*H*$_3$)$_2$), 1.52 (dd, J = 6.8, 1.8 Hz, 3H, C*H*$_3$CH=C), 4.50 (dq, J = 1.0, 6.5 Hz, 1H, CH$_3$C*H*=CH), 4.69 (heptet, J = 3.0 Hz, 1H, Si*H*), 6.17 (dq, J = 6.0, 1.6 Hz, 1H, CH$_3$CH=C*H*); (*E*)-dimethyl(1-propen-1-oxy)silane, δ 0.23 (d, J = 3.0 Hz, 6H, Si(C*H*$_3$)$_2$), 1.49 (dd, J = 6.5, 1.9 Hz, 3H, C*H*$_3$CH=C), 4.99, (2 m, J = 12.0 Hz, 1H, CH$_3$C*H*=CH), 4.61(heptet, J = 3.0 Hz, 1H, Si*H*), 6.21 (dq, J = 12.1, 1.9 Hz, 1H, CH$_3$CH=C*H*).

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(9) Small amounts (~ 2 - 3%) of another product formed as well, which was detected by ^{31}P NMR spectroscopy. $^{31}P\{1H\}$ NMR (161.9 MHz, CD_2Cl_2, 25°C) δ 27.60 (d, J_{P-P} = 34.0 Hz, 1P), 40.27 (d, J_{P-P} = 36.7 Hz, 1P).

(10) Confirmed by GC/IR.

(11) Consult Table 1 for appropriate solvents.

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(13) Prepared by hydrogenation of (*Z*)-methyl-α-acetamidocinnamate by a catalyst obtained *in situ* by reaction of *cis*-[Ru(MeCN)$_2$(1-2:5-6-η-C$_8$H$_{12}$)(η^3-C$_3$H$_5$)](BF$_4$) and 1,2-bis(diphenylphosphino)ethane.

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(15) Resolution outlined in: Kenyon, J.; Snellgrove, D. *J. Chem. Soc.* **1925**, *127*, 1174 and Young, W. G.; Caserio, F. F., Jr. *J. Org. Chem.* **1961**, *26*, 245.

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(17) Purchased from Aldrich.

(18) Compared to ^{1}H NMR spectra of (*E*)- and (*Z*)-1-propen-1-ol[14] and (*E*)- and (*Z*)-trimethylsilyl-1-propen-1-ol (B. Bosnich, personal communication).

(19) B. Bosnich, personal communication, and ref. 6

SDL:SHB9401 19-Apr-95

Structure Determination Laboratory

Report on the

Complete Structure Determination and Refinement of

[(binap)Ru(η^3,η^2-C_8H_{11})(NCMe)][BF_4]•0.5$C_2H_4Cl_2$ ($C_{55}H_{48}BClF_4NP_2Ru$)

for

Professor S. H. Bergens

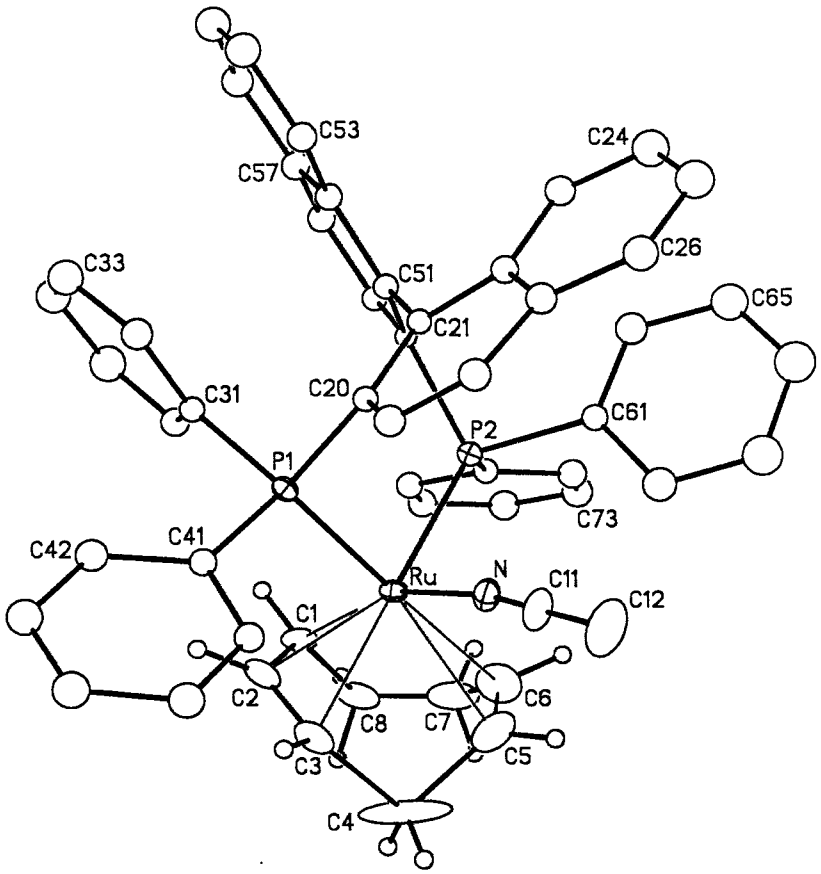
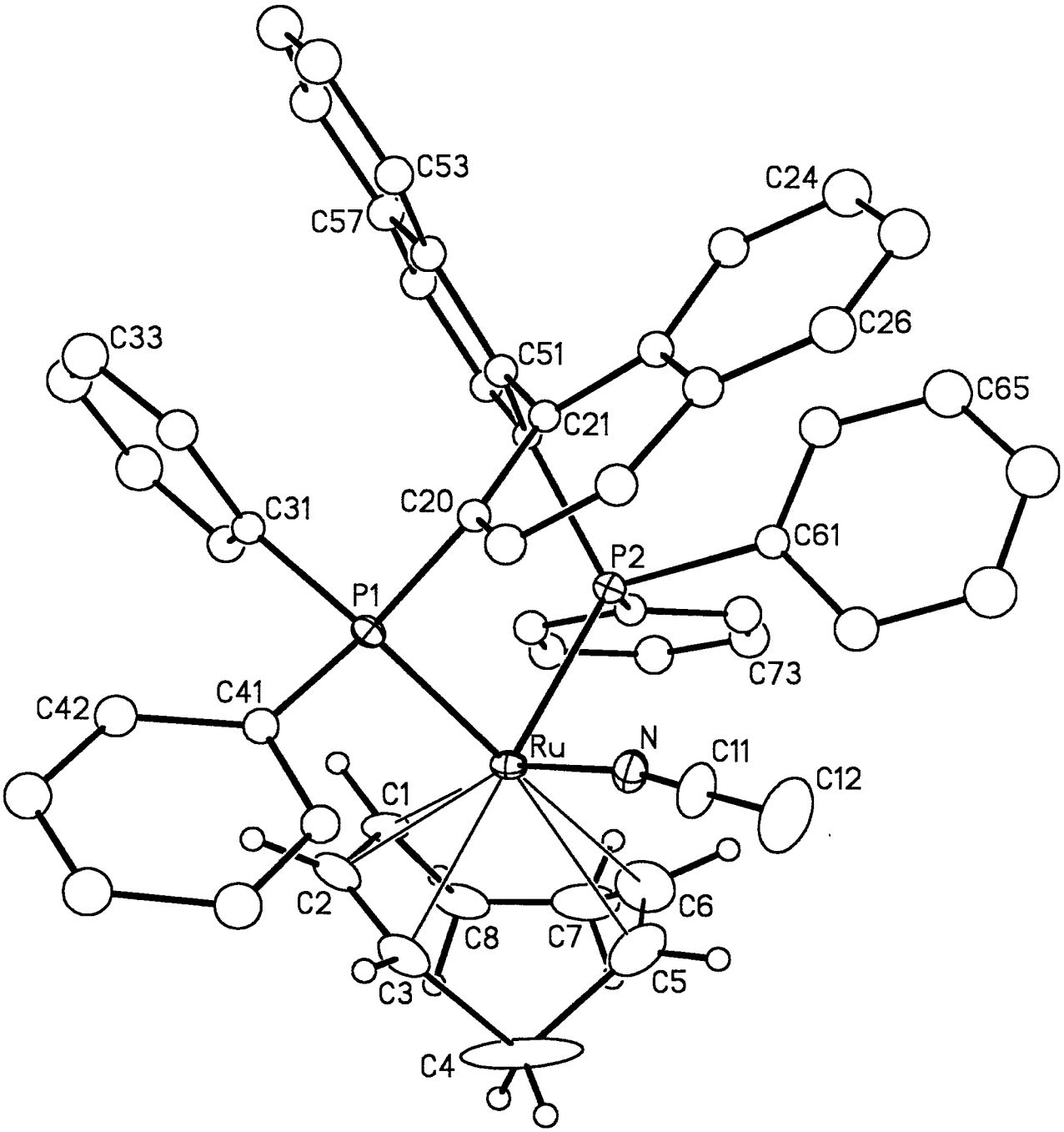


Figure Legends

Figure 1. Perspective view of the [(binap)Ru(η^3,η^2-C_8H_{11})(NCMe)]$^+$ complex cation showing the atom labelling scheme. Non-hydrogen atoms are represented by Gaussian ellipsoids at the 20% probability level. Hydrogen atoms are shown with artificially small thermal parameters for the η^3,η^2-C_8H_{11} group, and are not shown for the binap ligand and the coordinated acetonitrile.

Figure 2. Alternate view of the molecule. All hydrogen atoms and all but the ipso carbons of the binap phenyl groups have been omitted.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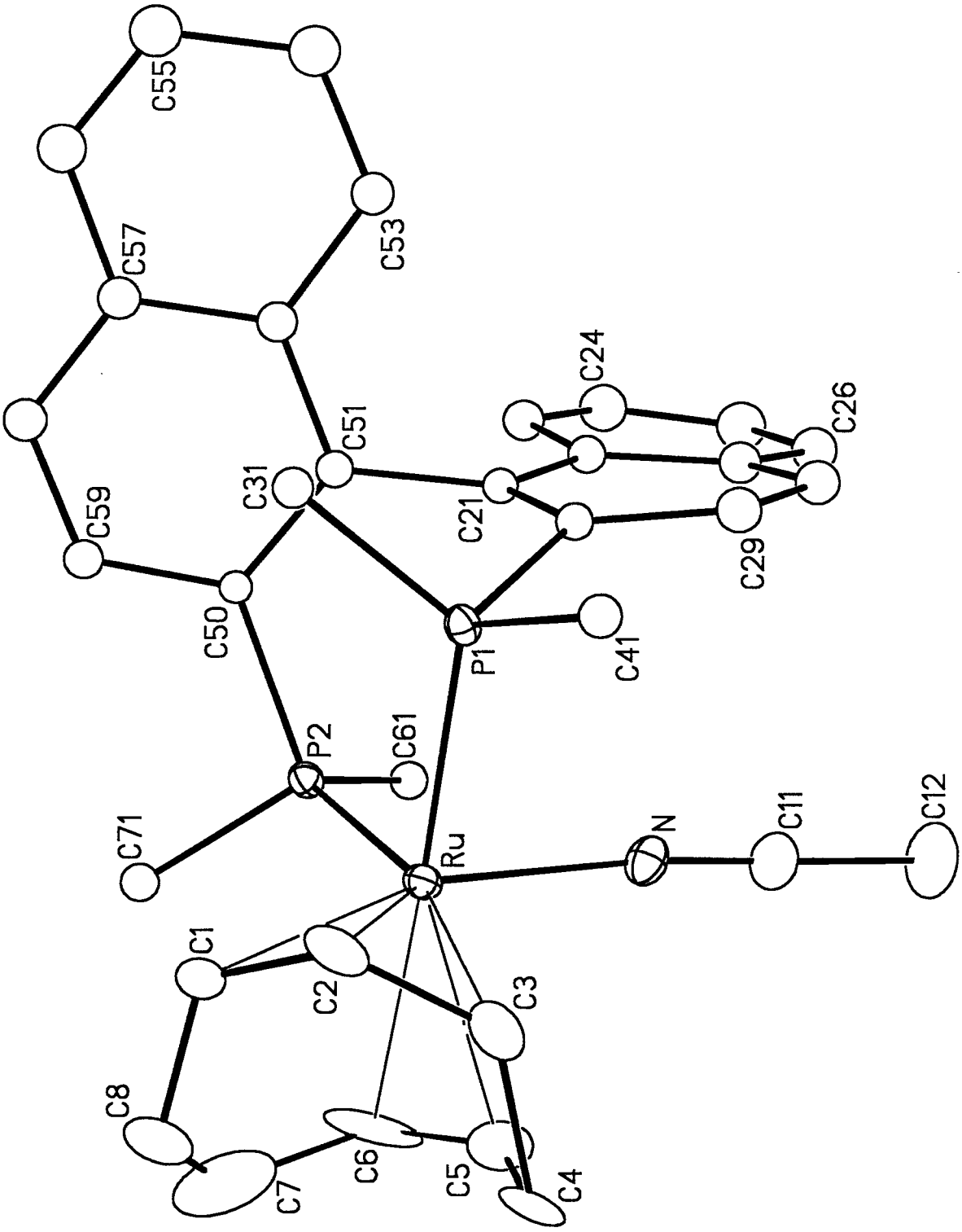
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C71
Ru
N
C11
C12
C1
C2
C3
C8
C6
C5
C7
C4

Table 1. Crystallographic Experimental Details

formula	$C_{55}H_{48}BClF_4NP_2Ru$
formula weight	1008.21
crystal dimensions (mm)	0.56 × 0.08 × 0.06
crystal system	monoclinic
space group	$P2_1$ (No. 4)
unit cell parameters[a]	
a (Å)	12.8559 (8)
b (Å)	13.0675 (8)
c (Å)	14.9401 (9)
β (deg)	91.678 (6)
V (Å^3)	2508.8 (3)
Z	2
ρ_{calcd} (g cm^{-3})	1.335
μ (mm^{-1})	4.040

B. Data Collection and Refinement Conditions

diffractometer	Siemens P4/RA[b]
radiation (λ [Å])	Cu Kα (1.54178)
monochromator	incident-beam, graphite crystal
temperature (°C)	22
scan type	θ–2θ
data collection 2θ limit (deg)	110.0
total data collected	3486 ($0 \le h \le 13$, $0 \le k \le 13$, $-15 \le l \le 15$)
independent reflections	3315

(continued)

Table 1. Crystallographic Experimental Details (continued)

structure solution method	direct methods (*SHELXS–86*[c])
refinement method	full-matrix least-squares on F^2 (*SHELXL–93*[d])
absorption correction method	semiempirical (ψ scans)
ratio of trans. factors (T_{max}/T_{min})	1.257
data/restraints/parameters	3315 [$F_o^2 \geq -3\sigma(F_o^2)$]/0/374
goodness-of-fit (S)[e]	1.076 [$F_o^2 \geq -3\sigma(F_o^2)$]
final R indices[f]	
$I>2\sigma(I)$]	$R_1 = 0.0483$, $wR_2 = 0.1283$
all data	$R_1 = 0.0506$, $wR_2 = 0.1306$
largest difference peak and hole	0.878 and –0.579 e Å^{-3}

[a]Obtained from least-squares refinement of 25 reflections with $53.7° < 2\theta < 56.0°$.

[b]Programs for diffractometer operation and data collection and reduction were those of the *XSCANS* system supplied by Siemens.

[c]Sheldrick, G. M. *Acta Crystallogr.* **1990**, *A46*, 467.

[d]Sheldrick, G. M. *SHELXL-93*. Program for crystal structure determination. University of Göttingen, Germany, 1993. Refinement on F_o^2 for all reflections (all of these having $F_o^2 < -3\sigma(F_o^2)$). Weighted R-factors wR_2 and all goodnesses of fit S are based on F_o^2; conventional R-factors R_1 are based on F_o, with F_o set to zero for negative F_o^2. The observed criterion of $F_o^2 > 2\sigma(F_o^2)$ is used only for calculating R_1, and is not relevant to the choice of reflections for refinement. R-factors based on F_o^2 are statistically about twice as large as those based on F_o, and R-factors based on ALL data will be even larger.

[e]$S = [\Sigma w(F_o^2 - F_c^2)^2/(n-p)]^{1/2}$ (n = number of data; p = number of parameters varied; $w = [\sigma^2(F_o^2) + (0.0771P)^2 + 5.5988P]^{-1}$ where $P = [\mathrm{Max}(F_o^2, 0) + 2F_c^2]/3$).

[f]$R_1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$; $wR_2 = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^4)]^{1/2}$.

Table 2. Atomic Coordinates and Equivalent Isotropic Displacement Parameters

Atom	*x*	*y*	*z*	U_{eq}, Å^2
Ru	-0.29999(4)	0.49867(5)	-0.15007(4)	0.0261(2)*
Cl1†	-0.0980(6)	0.1620(10)	-0.4501(6)	0.117(4)*
Cl2†	0.0443(11)	-0.0436(11)	-0.4757(15)	0.194(8)*
P1	-0.4252(2)	0.5992(2)	-0.08229(14)	0.0243(5)*
P2	-0.3686(2)	0.5412(2)	-0.29483(14)	0.0253(5)*
F1	-0.0466(8)	0.5040(11)	0.1479(8)	0.135(4)*
F2	0.1062(8)	0.5629(15)	0.1110(11)	0.190(8)*
F3	-0.0263(14)	0.5943(16)	0.0289(12)	0.235(11)*
F4	-0.0068(17)	0.6644(12)	0.1529(17)	0.263(13)*
N	-0.2047(6)	0.6265(7)	-0.1382(5)	0.038(2)*
C1	-0.3602(8)	0.3376(8)	-0.1331(7)	0.042(3)*
C2	-0.3366(10)	0.3852(8)	-0.0501(7)	0.047(3)*
C3	-0.2370(10)	0.4205(9)	-0.0225(7)	0.050(3)*
C4	-0.1249(15)	0.3803(12)	-0.0589(14)	0.147(11)*
C5	-0.1285(11)	0.4288(12)	-0.1602(13)	0.078(5)*
C6	-0.1767(17)	0.3935(15)	-0.2252(12)	0.094(6)*
C7	-0.2180(15)	0.2960(22)	-0.2372(14)	0.125(9)*
C8	-0.2891(11)	0.2637(10)	-0.1776(8)	0.060(3)*
C11	-0.1457(9)	0.6913(11)	-0.1231(8)	0.053(3)*
C12	-0.0716(11)	0.7740(13)	-0.1046(12)	0.088(5)*
C20	-0.4144(6)	0.7312(7)	-0.1271(6)	0.025(2)
C21	-0.4481(6)	0.7524(7)	-0.2147(5)	0.024(2)
C22	-0.4135(7)	0.8438(7)	-0.2560(6)	0.029(2)
C23	-0.4457(7)	0.8713(8)	-0.3446(7)	0.037(2)
C24	-0.4027(9)	0.9544(10)	-0.3852(8)	0.053(3)
C25	-0.3278(8)	1.0129(12)	-0.3421(7)	0.056(3)
C26	-0.2986(7)	0.9936(11)	-0.2553(6)	0.044(2)
C27	-0.3427(7)	0.9098(8)	-0.2103(6)	0.036(2)
C28	-0.3195(7)	0.8892(8)	-0.1175(7)	0.037(2)
C29	-0.3548(7)	0.8048(8)	-0.0791(7)	0.036(2)
C31	-0.5642(6)	0.5702(7)	-0.0895(6)	0.027(2)
C32	-0.6368(8)	0.6422(9)	-0.0605(6)	0.040(2)

(continued)

Table 2. Atomic Coordinates and Displacement Parameters (continued)

Atom	*x*	*y*	*z*	U_{eq}, Å^2
C33	-0.7425(9)	0.6185(10)	-0.0644(7)	0.052(3)
C34	-0.7747(9)	0.5274(9)	-0.0961(7)	0.053(3)
C35	-0.7070(9)	0.4563(10)	-0.1283(8)	0.058(3)
C36	-0.5997(7)	0.4769(7)	-0.1230(6)	0.036(2)
C41	-0.4001(7)	0.6124(7)	0.0395(6)	0.029(2)
C42	-0.4751(8)	0.5905(8)	0.1016(6)	0.039(2)
C43	-0.4526(8)	0.5980(9)	0.1935(7)	0.048(3)
C44	-0.3552(8)	0.6263(9)	0.2237(8)	0.049(3)
C45	-0.2794(9)	0.6446(9)	0.1628(7)	0.046(3)
C46	-0.3009(7)	0.6362(7)	0.0727(6)	0.032(2)
C50	-0.5030(6)	0.5943(7)	-0.3040(5)	0.021(2)
C51	-0.5265(7)	0.6870(7)	-0.2632(6)	0.027(2)
C52	-0.6315(7)	0.7229(8)	-0.2656(6)	0.031(2)
C53	-0.6609(7)	0.8169(8)	-0.2248(6)	0.035(2)
C54	-0.7632(8)	0.8492(9)	-0.2261(7)	0.050(3)
C55	-0.8405(9)	0.7883(9)	-0.2698(8)	0.052(3)
C56	-0.8157(8)	0.6988(9)	-0.3075(7)	0.045(3)
C57	-0.7108(7)	0.6631(8)	-0.3084(6)	0.035(2)
C58	-0.6833(7)	0.5720(8)	-0.3502(6)	0.036(2)
C59	-0.5819(6)	0.5402(7)	-0.3486(6)	0.030(2)
C61	-0.2982(7)	0.6316(7)	-0.3648(6)	0.030(2)
C62	-0.1923(8)	0.6490(10)	-0.3481(7)	0.049(3)
C63	-0.1380(11)	0.7176(11)	-0.4045(9)	0.069(4)
C64	-0.1897(11)	0.7634(12)	-0.4771(9)	0.072(4)
C65	-0.2918(10)	0.7391(11)	-0.4962(9)	0.061(3)
C66	-0.3460(9)	0.6767(9)	-0.4392(7)	0.045(3)
C71	-0.3776(7)	0.4299(7)	-0.3704(6)	0.029(2)
C72	-0.3205(8)	0.4231(9)	-0.4477(7)	0.041(3)
C73	-0.3245(9)	0.3353(10)	-0.4984(8)	0.050(3)
C74	-0.3844(8)	0.2535(9)	-0.4764(7)	0.049(3)
C75	-0.4410(8)	0.2591(9)	-0.4008(7)	0.042(2)
C76	-0.4396(7)	0.3461(8)	-0.3482(6)	0.035(2)

(continued)

Table 2. Atomic Coordinates and Displacement Parameters (continued)

Atom	*x*	*y*	*z*	U_{eq}, Å^2
C91†	-0.0252(40)	0.0951(45)	-0.3789(35)	0.141(17)
C92†	-0.0017(48)	0.0169(64)	-0.4065(39)	0.183(23)
B	0.0106(11)	0.5763(13)	0.1108(12)	0.067(4)*

Anisotropically-refined atoms are marked with an asterisk (*). The form of the anisotropic displacement parameter is: $\exp[-2\pi^2(h^2a^{*2}U_{11} + k^2b^{*2}U_{22} + l^2c^{*2}U_{33} + 2klb^*c^*U_{23} + 2hla^*c^*U_{13} + 2hka^*b^*U_{12})]$. Atoms of the dichloroethane solvent molecule (†) were refined with an occupancy factor of 50%.

Table 3. Selected Interatomic Distances (Å)

Atom1	Atom2	Distance	Atom1	Atom2	Distance
Ru	N	2.075(9)	C21	C51	1.492(12)
Ru	C2	2.165(11)	C22	C27	1.415(13)
Ru	C1	2.260(11)	C22	C23	1.422(14)
Ru	C3	2.289(10)	C23	C24	1.37(2)
Ru	P1	2.332(2)	C24	C25	1.37(2)
Ru	P2	2.378(2)	C25	C26	1.363(14)
Ru	C5	2.395(14)	C26	C27	1.41(2)
Ru	C6	2.402(12)	C27	C28	1.435(14)
Cl1	C91	1.65(5)	C28	C29	1.329(14)
Cl2	C92	1.44(7)	C31	C36	1.389(13)
P1	C31	1.827(8)	C31	C32	1.402(13)
P1	C41	1.847(9)	C32	C33	1.394(15)
P1	C20	1.857(9)	C33	C34	1.34(2)
P2	C61	1.834(9)	C34	C35	1.37(2)
P2	C71	1.842(9)	C35	C36	1.41(2)
P2	C50	1.863(8)	C41	C42	1.387(13)
F1	B	1.33(2)	C41	C46	1.390(13)
F2	B	1.24(2)	C42	C43	1.397(15)
F3	B	1.32(2)	C43	C44	1.37(2)
F4	B	1.33(2)	C44	C45	1.37(2)
N	C11	1.155(14)	C45	C46	1.370(15)
C1	C2	1.41(2)	C50	C59	1.390(12)
C1	C8	1.50(2)	C50	C51	1.393(13)
C2	C3	1.41(2)	C51	C52	1.428(13)
C3	C4	1.64(2)	C52	C57	1.422(13)
C4	C5	1.64(3)	C52	C53	1.428(14)
C5	C6	1.23(2)	C53	C54	1.380(15)
C6	C7	1.39(3)	C54	C55	1.42(2)
C7	C8	1.36(2)	C55	C56	1.34(2)
C11	C12	1.46(2)	C56	C57	1.427(14)
C20	C21	1.394(12)	C57	C58	1.395(14)
C20	C29	1.411(13)	C58	C59	1.368(13)
C21	C22	1.422(13)	C61	C66	1.386(14)

Table 3. Selected Interatomic Distances (continued)

Atom1	Atom2	Distance
C61	C62	1.395(14)
C62	C63	1.43(2)
C63	C64	1.39(2)
C64	C65	1.37(2)
C65	C66	1.38(2)
C71	C72	1.390(14)
C71	C76	1.399(14)
C72	C73	1.37(2)
C73	C74	1.36(2)
C74	C75	1.363(15)
C75	C76	1.382(15)
C91	C92	1.15(8)

Table 4. Selected Interatomic Angles (deg)

Atom1	Atom2	Atom3	Angle	Atom1	Atom2	Atom3	Angle
N	Ru	C2	129.1(4)	C20	P1	Ru	107.9(3)
N	Ru	C1	160.6(3)	C61	P2	C71	100.5(4)
C2	Ru	C1	37.2(4)	C61	P2	C50	101.0(4)
N	Ru	C3	95.4(4)	C71	P2	C50	102.0(4)
C2	Ru	C3	36.8(4)	C61	P2	Ru	119.5(3)
C1	Ru	C3	66.7(4)	C71	P2	Ru	112.8(3)
N	Ru	P1	85.5(2)	C50	P2	Ru	118.2(3)
C2	Ru	P1	85.4(3)	C11	N	Ru	171.7(9)
C1	Ru	P1	103.5(3)	C2	C1	C8	123.7(10)
C3	Ru	P1	97.0(3)	C2	C1	Ru	67.8(6)
N	Ru	P2	95.3(2)	C8	C1	Ru	109.6(8)
C2	Ru	P2	134.8(3)	C3	C2	C1	124.8(11)
C1	Ru	P2	101.6(3)	C3	C2	Ru	76.4(7)
C3	Ru	P2	167.0(3)	C1	C2	Ru	75.1(6)
P1	Ru	P2	91.20(8)	C2	C3	C4	126.7(10)
N	Ru	C5	76.7(5)	C2	C3	Ru	66.8(6)
C2	Ru	C5	90.1(5)	C4	C3	Ru	99.2(9)
C1	Ru	C5	88.3(5)	C5	C4	C3	100.4(9)
C3	Ru	C5	65.1(6)	C6	C5	C4	125.9(17)
P1	Ru	C5	152.9(5)	C6	C5	Ru	75.5(9)
P2	Ru	C5	110.5(5)	C4	C5	Ru	95.2(9)
N	Ru	C6	96.0(7)	C5	C6	C7	128.9(19)
C2	Ru	C6	95.2(5)	C5	C6	Ru	74.9(9)
C1	Ru	C6	75.7(6)	C7	C6	Ru	109.2(15)
C3	Ru	C6	84.9(5)	C8	C7	C6	117.5(22)
P1	Ru	C6	177.4(6)	C7	C8	C1	121.3(15)
P2	Ru	C6	86.6(5)	N	C11	C12	179.4(15)
C5	Ru	C6	29.6(6)	C21	C20	C29	119.4(8)
C31	P1	C41	102.6(4)	C21	C20	P1	119.9(7)
C31	P1	C20	104.8(4)	C29	C20	P1	119.6(7)
C41	P1	C20	104.8(4)	C20	C21	C22	118.8(8)
C31	P1	Ru	122.9(3)	C20	C21	C51	121.9(8)
C41	P1	Ru	112.2(3)	C22	C21	C51	119.1(8)

Table 4. Selected Interatomic Angles (continued)

Atom1	Atom2	Atom3	Angle	Atom1	Atom2	Atom3	Angle
C27	C22	C23	117.4(9)	C52	C51	C21	116.6(8)
C27	C22	C21	120.5(8)	C57	C52	C53	118.0(8)
C23	C22	C21	122.0(8)	C57	C52	C51	119.7(9)
C24	C23	C22	120.2(10)	C53	C52	C51	122.3(8)
C23	C24	C25	121.4(11)	C54	C53	C52	121.4(10)
C26	C25	C24	120.8(13)	C53	C54	C55	119.3(11)
C25	C26	C27	119.5(11)	C56	C55	C54	120.7(11)
C26	C27	C22	120.3(9)	C55	C56	C57	121.7(11)
C26	C27	C28	122.0(9)	C58	C57	C52	118.9(9)
C22	C27	C28	117.7(9)	C58	C57	C56	122.3(9)
C29	C28	C27	120.6(10)	C52	C57	C56	118.8(9)
C28	C29	C20	122.2(9)	C59	C58	C57	120.4(9)
C36	C31	C32	119.0(8)	C58	C59	C50	122.4(9)
C36	C31	P1	120.9(7)	C66	C61	C62	118.8(9)
C32	C31	P1	120.1(7)	C66	C61	P2	121.1(7)
C33	C32	C31	119.7(10)	C62	C61	P2	119.9(8)
C34	C33	C32	120.2(11)	C61	C62	C63	119.3(11)
C33	C34	C35	122.2(11)	C64	C63	C62	119.9(13)
C34	C35	C36	118.9(11)	C65	C64	C63	119.7(14)
C31	C36	C35	120.0(9)	C64	C65	C66	120.3(12)
C42	C41	C46	117.1(8)	C65	C66	C61	121.7(10)
C42	C41	P1	122.4(7)	C72	C71	C76	117.7(9)
C46	C41	P1	120.3(7)	C72	C71	P2	122.3(7)
C41	C42	C43	120.9(9)	C76	C71	P2	119.9(7)
C44	C43	C42	120.2(10)	C73	C72	C71	119.9(10)
C43	C44	C45	119.2(11)	C74	C73	C72	122.3(11)
C46	C45	C44	120.7(10)	C75	C74	C73	118.4(11)
C45	C46	C41	121.7(9)	C74	C75	C76	121.1(11)
C59	C50	C51	119.2(7)	C75	C76	C71	120.5(9)
C59	C50	P2	120.5(6)	C92	C91	Cl1	113.1(55)
C51	C50	P2	120.3(6)	C91	C92	Cl2	149.8(68)
C50	C51	C52	119.4(8)	F2	B	F3	110.8(18)
C50	C51	C21	123.9(8)	F2	B	F1	117.3(17)

Table 4. Selected Interatomic Angles (continued)

Atom1	Atom2	Atom3	Angle
F3	B	F1	108.9(14)
F2	B	F4	107.5(18)
F3	B	F4	102.9(19)
F1	B	F4	108.4(15)

Table 5. Torsional Angles (deg)

Atom 1	Atom 2	Atom 3	Atom 4	Angle	Atom 1	Atom 2	Atom 3	Atom 4	Angle
N	Ru	P1	C31	160.4(4)	C5	Ru	P2	C71	59.9(5)
C2	Ru	P1	C31	-69.7(5)	C6	Ru	P2	C71	41.9(7)
C1	Ru	P1	C31	-37.0(4)	N	Ru	P2	C50	-103.6(4)
C3	Ru	P1	C31	-104.7(5)	C2	Ru	P2	C50	66.7(5)
P2	Ru	P1	C31	65.2(3)	C1	Ru	P2	C50	86.0(4)
C5	Ru	P1	C31	-150.8(10)	C3	Ru	P2	C50	111.2(15)
C6	Ru	P1	C31	33.4(115)	P1	Ru	P2	C50	-18.0(3)
N	Ru	P1	C41	-76.5(4)	C5	Ru	P2	C50	178.6(5)
C2	Ru	P1	C41	53.4(4)	C6	Ru	P2	C50	160.6(7)
C1	Ru	P1	C41	86.1(4)	C2	Ru	N	C11	24.5(58)
C3	Ru	P1	C41	18.4(4)	C1	Ru	N	C11	-14.1(62)
P2	Ru	P1	C41	-171.7(3)	C3	Ru	N	C11	8.2(57)
C5	Ru	P1	C41	-27.7(10)	P1	Ru	N	C11	104.8(57)
C6	Ru	P1	C41	156.5(115)	P2	Ru	N	C11	-164.4(57)
N	Ru	P1	C20	38.5(4)	C5	Ru	N	C11	-54.6(57)
C2	Ru	P1	C20	168.4(4)	C6	Ru	N	C11	-77.2(57)
C1	Ru	P1	C20	-158.9(4)	N	Ru	C1	C2	53.1(13)
C3	Ru	P1	C20	133.4(4)	C3	Ru	C1	C2	28.8(7)
P2	Ru	P1	C20	-56.7(3)	P1	Ru	C1	C2	-63.1(6)
C5	Ru	P1	C20	87.3(10)	P2	Ru	C1	C2	-157.1(6)
C6	Ru	P1	C20	-88.5(115)	C5	Ru	C1	C2	92.3(8)
N	Ru	P2	C61	19.9(4)	C6	Ru	C1	C2	119.4(8)
C2	Ru	P2	C61	-169.7(5)	N	Ru	C1	C8	-66.3(14)
C1	Ru	P2	C61	-150.4(4)	C2	Ru	C1	C8	-119.4(10)
C3	Ru	P2	C61	-125.3(15)	C3	Ru	C1	C8	-90.5(8)
P1	Ru	P2	C61	105.6(3)	P1	Ru	C1	C8	177.5(7)
C5	Ru	P2	C61	-57.9(6)	P2	Ru	C1	C8	83.5(7)
C6	Ru	P2	C61	-75.8(7)	C5	Ru	C1	C8	-27.1(9)
N	Ru	P2	C71	137.6(4)	C6	Ru	C1	C8	0.0(8)
C2	Ru	P2	C71	-52.0(5)	C8	C1	C2	C3	38.2(17)
C1	Ru	P2	C71	-32.7(4)	Ru	C1	C2	C3	-61.2(10)
C3	Ru	P2	C71	-7.5(15)	C8	C1	C2	Ru	99.3(11)
P1	Ru	P2	C71	-136.7(3)	N	Ru	C2	C3	-27.7(8)

Table 5. Torsional Angles (continued)

Atom 1	Atom 2	Atom 3	Atom 4	Angle
C1	Ru	C2	C3	132.3(10)
P1	Ru	C2	C3	-108.2(6)
P2	Ru	C2	C3	164.7(5)
C5	Ru	C2	C3	45.1(8)
C6	Ru	C2	C3	74.3(9)
N	Ru	C2	C1	-160.0(6)
C3	Ru	C2	C1	-132.3(10)
P1	Ru	C2	C1	119.6(6)
P2	Ru	C2	C1	32.5(8)
C5	Ru	C2	C1	-87.1(8)
C6	Ru	C2	C1	-57.9(9)
C1	C2	C3	C4	-23.6(19)
Ru	C2	C3	C4	-84.2(13)
C1	C2	C3	Ru	60.6(10)
N	Ru	C3	C2	158.7(7)
C1	Ru	C3	C2	-29.1(6)
P1	Ru	C3	C2	72.6(6)
P2	Ru	C3	C2	-56.1(18)
C5	Ru	C3	C2	-128.6(8)
C6	Ru	C3	C2	-105.7(9)
N	Ru	C3	C4	-75.2(8)
C2	Ru	C3	C4	126.1(11)
C1	Ru	C3	C4	96.9(8)
P1	Ru	C3	C4	-161.4(7)
P2	Ru	C3	C4	70.0(17)
C5	Ru	C3	C4	-2.6(8)
C6	Ru	C3	C4	20.4(10)
C2	C3	C4	C5	71.4(17)
Ru	C3	C4	C5	3.5(11)
C3	C4	C5	C6	-79.1(17)
C3	C4	C5	Ru	-3.3(10)
N	Ru	C5	C6	-129.2(14)
C2	Ru	C5	C6	100.4(13)
C1	Ru	C5	C6	63.3(13)
C3	Ru	C5	C6	128.3(14)
P1	Ru	C5	C6	-179.6(10)
P2	Ru	C5	C6	-38.4(14)
N	Ru	C5	C4	105.1(9)
C2	Ru	C5	C4	-25.3(9)
C1	Ru	C5	C4	-62.5(9)
C3	Ru	C5	C4	2.5(8)
P1	Ru	C5	C4	54.6(14)
P2	Ru	C5	C4	-164.2(8)
C6	Ru	C5	C4	-125.8(16)
C4	C5	C6	C7	-16.8(30)
Ru	C5	C6	C7	-102.7(22)
C4	C5	C6	Ru	85.9(14)
N	Ru	C6	C5	49.3(13)
C2	Ru	C6	C5	-81.0(13)
C1	Ru	C6	C5	-112.8(13)
C3	Ru	C6	C5	-45.6(13)
P1	Ru	C6	C5	176.1(106)
P2	Ru	C6	C5	144.3(13)
N	Ru	C6	C7	175.9(13)
C2	Ru	C6	C7	45.6(14)
C1	Ru	C6	C7	13.7(13)
C3	Ru	C6	C7	80.9(14)
P1	Ru	C6	C7	-57.4(118)
P2	Ru	C6	C7	-89.2(14)
C5	Ru	C6	C7	126.5(20)
C5	C6	C7	C8	58.6(27)
Ru	C6	C7	C8	-27.1(20)
C6	C7	C8	C1	30.7(22)
C2	C1	C8	C7	-91.4(17)
Ru	C1	C8	C7	-15.6(16)
C31	P1	C20	C21	-60.2(7)

Table 5. Torsional Angles (continued)

Atom 1	Atom 2	Atom 3	Atom 4	Angle	Atom 1	Atom 2	Atom 3	Atom 4	Angle
C41	P1	C20	C21	-167.9(7)	C20	P1	C31	C32	-45.9(8)
Ru	P1	C20	C21	72.4(7)	Ru	P1	C31	C32	-169.2(6)
C31	P1	C20	C29	131.9(7)	C36	C31	C32	C33	1.1(14)
C41	P1	C20	C29	24.2(8)	P1	C31	C32	C33	-178.6(8)
Ru	P1	C20	C29	-95.6(7)	C31	C32	C33	C34	-0.3(16)
C29	C20	C21	C22	5.5(12)	C32	C33	C34	C35	-2.3(17)
P1	C20	C21	C22	-162.5(6)	C33	C34	C35	C36	3.9(17)
C29	C20	C21	C51	-168.8(8)	C32	C31	C36	C35	0.5(13)
P1	C20	C21	C51	23.2(11)	P1	C31	C36	C35	-179.7(8)
C20	C21	C22	C27	2.7(13)	C34	C35	C36	C31	-3.0(16)
C51	C21	C22	C27	177.2(8)	C31	P1	C41	C42	6.6(9)
C20	C21	C22	C23	-179.1(8)	C20	P1	C41	C42	115.9(9)
C51	C21	C22	C23	-4.6(13)	Ru	P1	C41	C42	-127.3(8)
C27	C22	C23	C24	4.8(14)	C31	P1	C41	C46	-179.2(8)
C21	C22	C23	C24	-173.4(9)	C20	P1	C41	C46	-70.0(8)
C22	C23	C24	C25	0.2(16)	Ru	P1	C41	C46	46.9(9)
C23	C24	C25	C26	-4.1(18)	C46	C41	C42	C43	3.6(15)
C24	C25	C26	C27	2.7(17)	P1	C41	C42	C43	177.9(8)
C25	C26	C27	C22	2.5(15)	C41	C42	C43	C44	-0.8(17)
C25	C26	C27	C28	-176.4(10)	C42	C43	C44	C45	-1.6(18)
C23	C22	C27	C26	-6.2(14)	C43	C44	C45	C46	1.0(18)
C21	C22	C27	C26	172.1(9)	C44	C45	C46	C41	2.1(17)
C23	C22	C27	C28	172.8(8)	C42	C41	C46	C45	-4.3(15)
C21	C22	C27	C28	-8.9(13)	P1	C41	C46	C45	-178.7(8)
C26	C27	C28	C29	-174.0(10)	C61	P2	C50	C59	112.4(7)
C22	C27	C28	C29	7.1(14)	C71	P2	C50	C59	9.0(8)
C27	C28	C29	C20	1.1(14)	Ru	P2	C50	C59	-115.2(6)
C21	C20	C29	C28	-7.6(14)	C61	P2	C50	C51	-70.0(7)
P1	C20	C29	C28	160.4(8)	C71	P2	C50	C51	-173.4(7)
C41	P1	C31	C36	-116.3(7)	Ru	P2	C50	C51	62.3(7)
C20	P1	C31	C36	134.4(7)	C59	C50	C51	C52	1.9(12)
Ru	P1	C31	C36	11.1(8)	P2	C50	C51	C52	-175.6(7)
C41	P1	C31	C32	63.4(8)	C59	C50	C51	C21	-179.0(8)

Table 5. Torsional Angles (continued)

Atom 1	Atom 2	Atom 3	Atom 4	Angle	Atom 1	Atom 2	Atom 3	Atom 4	Angle
P2	C50	C51	C21	3.4(11)	Ru	P2	C61	C66	-164.6(7)
C20	C21	C51	C50	-80.1(11)	C71	P2	C61	C62	-103.0(9)
C22	C21	C51	C50	105.6(10)	C50	P2	C61	C62	152.5(8)
C20	C21	C51	C52	99.0(10)	Ru	P2	C61	C62	20.9(10)
C22	C21	C51	C52	-75.3(10)	C66	C61	C62	C63	3.9(16)
C50	C51	C52	C57	0.9(13)	P2	C61	C62	C63	178.5(9)
C21	C51	C52	C57	-178.2(8)	C61	C62	C63	C64	-2.3(19)
C50	C51	C52	C53	179.7(8)	C62	C63	C64	C65	-2.6(21)
C21	C51	C52	C53	0.5(13)	C63	C64	C65	C66	5.9(21)
C57	C52	C53	C54	0.3(14)	C64	C65	C66	C61	-4.3(19)
C51	C52	C53	C54	-178.5(9)	C62	C61	C66	C65	-0.7(16)
C52	C53	C54	C55	-0.7(16)	P2	C61	C66	C65	-175.2(9)
C53	C54	C55	C56	1.9(17)	C61	P2	C71	C72	12.3(9)
C54	C55	C56	C57	-2.6(16)	C50	P2	C71	C72	116.0(8)
C53	C52	C57	C58	178.8(8)	Ru	P2	C71	C72	-116.2(8)
C51	C52	C57	C58	-2.4(13)	C61	P2	C71	C76	-171.0(7)
C53	C52	C57	C56	-0.9(14)	C50	P2	C71	C76	-67.3(8)
C51	C52	C57	C56	177.9(9)	Ru	P2	C71	C76	60.5(8)
C55	C56	C57	C58	-177.6(10)	C76	C71	C72	C73	-1.3(14)
C55	C56	C57	C52	2.2(15)	P2	C71	C72	C73	175.4(8)
C52	C57	C58	C59	1.0(14)	C71	C72	C73	C74	1.0(17)
C56	C57	C58	C59	-179.3(9)	C72	C73	C74	C75	-0.9(17)
C57	C58	C59	C50	1.9(14)	C73	C74	C75	C76	1.2(16)
C51	C50	C59	C58	-3.4(13)	C74	C75	C76	C71	-1.6(15)
P2	C50	C59	C58	174.2(7)	C72	C71	C76	C75	1.6(14)
C71	P2	C61	C66	71.5(9)	P2	C71	C76	C75	-175.2(7)
C50	P2	C61	C66	-33.1(9)	Cl1	C91	C92	Cl2	53.7(147)

Table 6. Anisotropic Displacement Parameters (U_{ij}, Å^2)

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Ru	0.0295(3)	0.0233(3)	0.0252(3)	-0.0027(3)	-0.0040(2)	0.0053(3)
Cl1	0.074(5)	0.184(11)	0.095(6)	0.059(7)	0.026(4)	0.041(6)
Cl2	0.150(11)	0.113(9)	0.325(22)	-0.015(12)	0.119(14)	-0.021(8)
P1	0.0298(12)	0.0209(12)	0.0220(11)	0.0005(10)	-0.0020(9)	-0.0017(10)
P2	0.0289(11)	0.0233(11)	0.0236(11)	-0.0024(9)	-0.0005(9)	0.0006(9)
F1	0.126(8)	0.095(7)	0.186(10)	-0.006(10)	0.049(7)	-0.045(9)
F2	0.062(6)	0.278(21)	0.231(15)	0.098(15)	0.015(7)	0.035(9)
F3	0.230(18)	0.248(22)	0.218(18)	0.099(17)	-0.146(16)	-0.083(17)
F4	0.289(23)	0.104(11)	0.405(32)	-0.073(16)	0.157(23)	-0.104(14)
N	0.031(4)	0.051(6)	0.032(4)	-0.002(4)	0.003(3)	-0.007(5)
C1	0.043(6)	0.027(5)	0.057(7)	0.003(5)	-0.015(5)	0.009(5)
C2	0.078(8)	0.027(6)	0.036(6)	0.005(5)	-0.001(6)	0.016(6)
C3	0.079(8)	0.033(6)	0.037(6)	0.001(5)	-0.011(5)	0.003(6)
C4	0.168(18)	0.063(10)	0.202(21)	-0.088(13)	-0.150(17)	0.091(12)
C5	0.050(8)	0.065(10)	0.120(14)	-0.013(10)	0.011(9)	0.013(7)
C6	0.131(16)	0.071(12)	0.082(12)	0.001(9)	0.043(12)	0.077(12)
C7	0.094(13)	0.182(27)	0.098(14)	0.042(16)	-0.028(11)	0.052(16)
C8	0.089(9)	0.042(7)	0.048(7)	-0.004(6)	-0.016(7)	0.028(7)
C11	0.039(6)	0.066(8)	0.054(7)	-0.009(6)	0.013(5)	-0.015(7)
C12	0.056(8)	0.091(12)	0.117(13)	-0.017(10)	0.013(8)	-0.044(9)
B	0.043(8)	0.061(11)	0.097(12)	0.003(9)	-0.011(8)	-0.016(7)

The form of the anisotropic displacement parameter is:

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

Table 7. Derived Atomic Coordinates and Displacement Parameters for Hydrogen Atoms

Atom	x	y	z	U_{eq}, Å^2
H1	-0.4346	0.3282	-0.1472	0.051
H2	-0.3946	0.3999	-0.0110	0.056
H3	-0.2339	0.4628	0.0316	0.060
H4A	-0.0670	0.4071	-0.0231	0.177
H4B	-0.1212	0.3062	-0.0600	0.177
H5	-0.0733	0.4777	-0.1734	0.094
H6	-0.1549	0.4239	-0.2817	0.112
H7A	-0.2504	0.2930	-0.2966	0.150
H7B	-0.1608	0.2476	-0.2358	0.150
H8A	-0.2510	0.2276	-0.1303	0.072
H8B	-0.3328	0.2135	-0.2082	0.072
H12A	-0.0020	0.7481	-0.1080	0.106
H12B	-0.0822	0.8007	-0.0457	0.106
H12C	-0.0821	0.8274	-0.1480	0.106
H23	-0.4961	0.8327	-0.3752	0.044
H24	-0.4246	0.9717	-0.4431	0.064
H25	-0.2967	1.0663	-0.3726	0.067
H26	-0.2500	1.0354	-0.2259	0.053
H28	-0.2795	0.9353	-0.0840	0.044
H29	-0.3396	0.7938	-0.0187	0.043
H32	-0.6145	0.7054	-0.0388	0.048
H33	-0.7908	0.6659	-0.0450	0.062
H34	-0.8454	0.5120	-0.0962	0.063
H35	-0.7317	0.3954	-0.1532	0.070
H36	-0.5524	0.4282	-0.1420	0.043
H42	-0.5413	0.5704	0.0819	0.047
H43	-0.5040	0.5839	0.2342	0.057
H44	-0.3405	0.6331	0.2847	0.059
H45	-0.2128	0.6627	0.1829	0.055
H46	-0.2478	0.6468	0.0328	0.039
H53	-0.6102	0.8573	-0.1966	0.042
H54	-0.7812	0.9103	-0.1986	0.060

Table 7. Derived Parameters for Hydrogen Atoms (continued)

Atom	*x*	*y*	*z*	U_{eq}, Å^2
H55	-0.9092	0.8107	-0.2725	0.062
H56	-0.8682	0.6588	-0.3336	0.054
H58	-0.7342	0.5327	-0.3793	0.043
H59	-0.5651	0.4803	-0.3785	0.036
H62	-0.1576	0.6163	-0.3008	0.059
H63	-0.0680	0.7317	-0.3927	0.083
H64	-0.1553	0.8103	-0.5125	0.087
H65	-0.3246	0.7647	-0.5478	0.073
H66	-0.4163	0.6647	-0.4510	0.054
H72	-0.2797	0.4779	-0.4652	0.050
H73	-0.2850	0.3316	-0.5495	0.060
H74	-0.3866	0.1952	-0.5122	0.058
H75	-0.4812	0.2034	-0.3843	0.051
H76	-0.4801	0.3489	-0.2977	0.042
H91A†	0.0372	0.1338	-0.3637	0.169
H91B†	-0.0630	0.0851	-0.3244	0.169
H92A†	-0.0680	-0.0181	-0.4029	0.220
H92B†	0.0383	-0.0085	-0.3551	0.220

†Hydrogen atoms of the dichloroethane solvent molecule were included with an occupancy factor of 50%.