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The Reduction of C* Bonds Proceeds with Retention of Configuration: Stereochemical Investigation of the Heterogeneous Reduction by Dideuterium of (Homohypostrophene)neopentyl(2-norbornyl)platinum(II) Complexes on Platinum Black.[1]

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with retention of configuration. Approximately 20% of the *exo*-2-norbornyl* moieties undergo β-H activation at rates competitive with reductive elimination as norbornanes-d_n; in contrast, approximately 35% of the *endo*-2-norbornyl* moieties undergo α-H activation and epimerization to *endo*-2-norbornyl* at rates competitive with reductive elimination as norbornanes-d_n. These results are rationalized on the basis of steric interactions between the norbornyl moieties and the surface of platinum. The reduction by D_2 of homohypostrophene incorporates deuterium exclusively into the *exo* positions of the product tetracyclo[6.3.0.0^{4,11}.0^{5,9}]undecane (HOPH); analogous reduction of **1** and **2** incorporates deuterium predominantly into the *endo* positions of HOPH. These results argue that the reduction of (diolefin)dialkylplatinum(II) complexes proceeds via adsorption of the platinum atom to the surface of the catalyst. Neopentane-d_1 is the major isotopomer of neopentane produced from the reductions of **1** and **2** by D_2.

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Technical Report # 5

The Reduction of C* Bonds Proceeds with Retention of Configuration: Stereochemical Investigation of the Heterogeneous Reduction by Dideuterium of (Homohypostrophene)neopentyl(2-norbornyl)-platinum(II) Complexes on Platinum Black

by

T. Randall Lee, Derk A. Wierda, and George M. Whitesides

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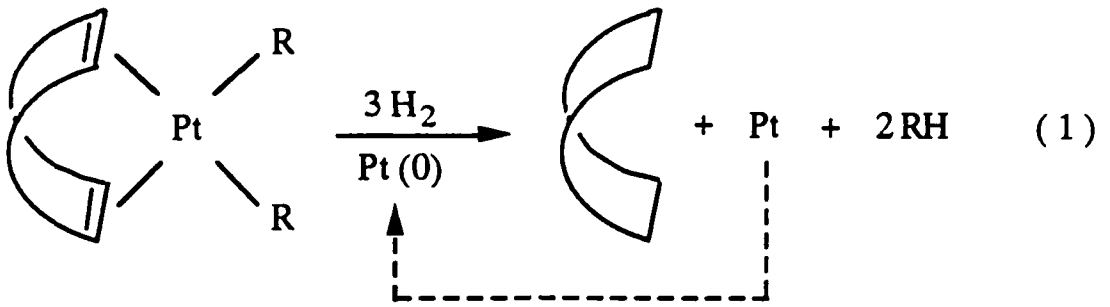
Introduction

The stereochemical outcome of heterogeneous hydrogenations of olefins on noble metal catalysts has been examined extensively.[2] Despite these efforts and the continuing progress in understanding the structures of hydrocarbons on metal surfaces,[3-24] the stereochemistry of reduction of the C* bond has been defined only by inference.[25] Determining the stereochemistry of reduction of C* bonds using the reduction of olefins requires a critical assumption since the *initial* stereochemistry of the C* bond is not known. Studies of the hydrogenation of olefins (that have no particular face selectivity) have showed that, over most metals, H_2 adds predominantly *cis* to the double bonds.[26-32] In addition, "anchoring" at sites remote from the double bonds increases the selectivity toward *cis* addition of H_2.[33-41] Several studies showed that *cis* addition of H_2 occurs to the less hindered face of the olefin.[42-49] These last reports constitute the most definitive, albeit indirect, characterization of the stereochemistry of reduction of C* bonds: the olefins *probably* coordinate by presenting their least hindered face to the surface of the metal; since H_2 adds to this face, the stereochemistry of the reduction of the C* bond proceeds with retention of configuration. We wanted to provide an independent and more direct determination of the stereochemistry of this reaction.

We have been studying the mechanisms of the heterogeneous hydrogenation of olefins and of organoplatinum compounds.[50-55] In this research, we showed that the heterogeneous, platinum-catalyzed hydrogenation of (diolefin)dialkylplatinum(II) complexes ($DOPtR_2$) on platinum black produces diolefin-H_4, two equivalents of R-H, and platinum(0). The platinum(0) becomes part of the surface of the catalyst (eq 1). This reaction involves (i) adsorption of dihydrogen and the components of $DOPtR_2$ on the surface of the catalyst, (ii) generation of platinum-

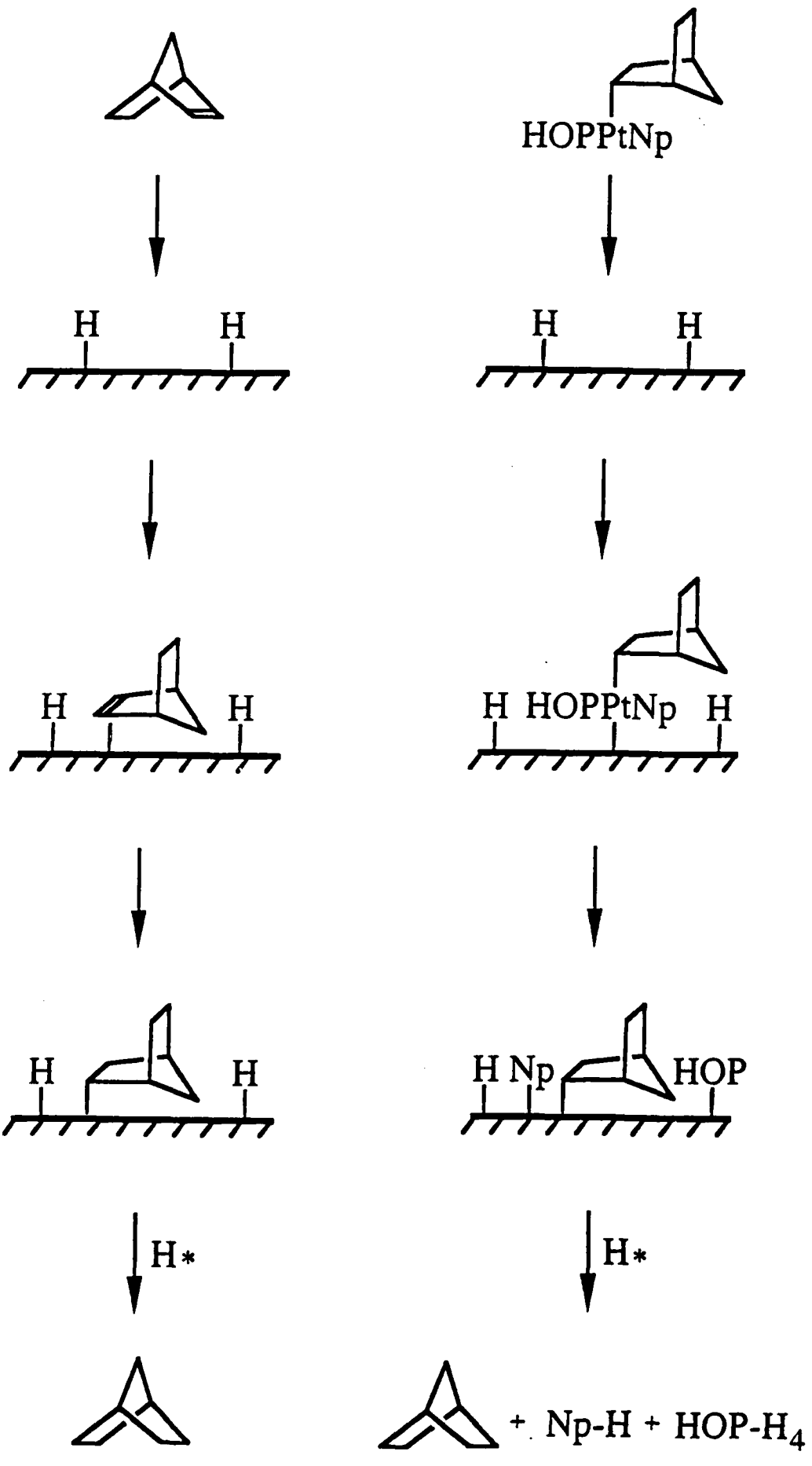


surface alkyls (DO* and R*) from the alkyl and diolefin moieties originally present in the organometallic complex, and (iii) final reaction of the surface alkyls with surface hydrides to produce alkanes by reductive elimination. This reaction can be used to generate R* of known *initial* structure.[51,52] The intermediate surface alkyls generated from these reductions are related to those generated in heterogeneous hydrogenations of olefins (Scheme I).

In alkane solvents at relatively low temperatures (*ca* -20 °C) and high pressures of H_2 (*ca* 2.5 atm), the rate-determining step in production of alkane is an unspecified reaction occurring on the surface.[50] Under these conditions, the reduction by dideuterium of (1,5-cyclooctadiene)di-*n*-propylplatinum(II) produces 1-propane-d_1 as the major product, and that of (1,5-cyclooctadiene)di-*iso*-propylplatinum(II) produces 2-propane-d_1 as the major product. These observations suggest that the regiochemistries of the propyl-Pt moieties are preserved on transfer to the surface and on reduction.[52]

In the work presented here, we used this system to characterize the stereochemistry of the reduction of C* bonds in heterogeneous hydrogenations. We synthesized (homohypostrophene)neopentyl(*exo*-2-norbornyl)platinum(II) **1** and (homohypostrophene)neopentyl(*endo*-2-norbornyl)platinum(II) **2**, and confirmed their structures using X-ray crystallography. We selected

Scheme I. Proposed Analogy Between Surface Alkyls Derived from Norbornene (left) and Those Derived from (Homohypostophene)neopentyl(*exo*-2-norbornyl)platinum(II) (right). HOP = Homohypostrophene; Np = Neopentyl.


HOPPtNp
H
H
H
H
H
H HOPPtNp H
H
H
H
Np
HOP
H*
H*
+ Np-H + HOP-H4

homohypostrophene (HOP) as the diolefin for three reasons. First, we could examine the stereochemistry of the reduction of the HOP moiety in $HOPPtR_2$. Second, HOP cannot form surface π-allyl groups, and is thus a relatively inert surface species that should not interfere in the reactions of coadsorbed alkyls.[56] Third, we could readily obtain crystals of complexes containing HOP. We chose 2-norbornyl groups as ligands for two reasons. First, norbornyl groups substituted at C(2) exist as two epimers (*exo* and *endo*, each enantiomeric); we wanted to synthesize both epimers to simplify interpretation of the data from reductions of the platinum complexes.[57] Second, the location of the deuterium atoms in the product norbornanes (*exo* vs *endo*) could be easily resolved using ^1H (or ^2H) NMR spectroscopy.[47,51,61] Neopentyl groups were chosen as ligands (rather than methyl groups) for two reasons. First, the reaction of mixtures of *exo*- and *endo*-2-norbornylmagnesium bromide (*ca* 45% *exo* and 55% *endo*) with HOPPtNpCl was selective (≥ 90% **1** produced), but analogous reaction with HOPPtMeCl was not. Second, the substitution of neopentyl groups for methyl groups improved the crystallinity of $DOPtR_2$ complexes.

Reduction of these complexes with dideuterium over platinum black in alkane solvents generated deuterated norbornanes via intermediate 2-norbornyl* moieties of known stereochemistry. We determined the location of the deuterium atoms in these norbornanes using ^1H and ^2H NMR spectroscopy, and analyzed the alkanes produced in these reductions by GC/MS. Finally, ^1H NMR spectroscopy was used to locate the deuterium atoms in the homohypostrophanes produced in the reductions by D_2 of samples containing **1** and **2**, and of free homohypostrophene.

Experimental Section

General. We purchased *n*-pentane (99+%, anhydrous, sure-seal bottle) from Aldrich, and stored it under argon. *n*-Heptane (Aldrich, 99.9%, HPLC grade) was distilled from Na/K, and diethyl ether (Mallinckrodt) was distilled from Na/benzophenone. We purchased platinum black (lot numbers 10410HT and 03019KT), neopentyl chloride (99%), *exo*-2-bromonorbornane (98%), norbornene (99%), benzophenone (99%), pentacyclo[5.4.0.0^{2,6}.0^{3,10}.0^{5,9}]-undecane-8,11-dione (98%), *tert*-butyllithium (1.7*M* in *n*-pentane), $LiAlH_4$ (1*M* in diethyl ether), and 10% $AgNO_3$ on silica gel from Aldrich, and used them without further purification. Di-μ-chloro-dichlorobis(ethylene)di-platinum(II) (Zeise's dimer, Strem), and dideuterium (99.5 atom % D, Matheson) were used as received. Cyclopentadiene was distilled from dicyclopentadiene (Aldrich, 97%), and benzoquinone (Baker) was recrystallized from petroleum ether.

We collected the ^2H NMR spectra on a Bruker WM 300 spectrometer operating at 46.03 MHz with broadband ^1H decoupling, and referenced to C_6D_6 (δ 7.15 ppm). Melting points were obtained in capillaries sealed under vacuum. We used a Hewlett Packard 5992A GC/MS (70 eV electron impact ionization) to measure mass spectra, and collected these data using the software for Selected Ion Monitoring from Hewlett Packard. The UV absorbance spectra of samples containing predominantly **1** and predominantly **2** were obtained with a Perkin-Elmer 552 spectrophotometer, and are included as supplementary material. We measured the UV absorbances of aliquots from kinetics runs on a Gilford 240 single-beam spectrophotometer at 286 and 292 nm for reductions of samples containing predominantly **1** and **2**, respectively (*vide infra*). The methods used to collect the X-ray structures of **1** and **2** are included as supplementary material. The lowest energy conformation and ^1H NMR coupling constants of

homohypostrophane (HOPH) were calculated with Macromodel V2.0 using the MM2(85) parameter set.[62] Oneida Research Co performed the elemental analyses.

Procedure for Reductions. Each reduction was performed as follows. A 20-mL pressure-bottle reactor (purchased from Lab Glass, and silanized as described previously[50]) was charged with 30 mg of platinum black and a football-shaped (10 x 6 mm) magnetic stirring bar. The vessel was capped with a neoprene septum, purged with argon, and immersed to within ~ 1 cm of its metal crown cap in a large bath of water/ethylene glycol (1:1, v:v) thermostatted by a Neslab Cryocool at -20 ± 1 °C. Solvent (1 mL) was added, and dideuterium was admitted to the reactor through a syringe needle inserted into the septum. The vessel was purged for 15 s, then pressurized to 2.4 atm (as monitored by inserting a syringe needle equipped with a pressure gauge through the septum of the reactor; the pressure reported is probably accurate to ± 5%). Stirring was started and maintained at 1800 RPM (the number of revolutions per minute of the magnetic stir bar as measured by a calibrated strobe light). After 10 minutes, we stopped the stirrer, allowed the catalyst to settle to the bottom of the vessel, and removed the solvent through a cannula. A yellow solution of the platinum complex (25 mg of dissolved in 4 mL of *n*-pentane or *n*-heptane)[63] was cooled to -20 °C and admitted to the vessel via cannula. We started the stirrer, and allowed the reaction to proceed for 90 min; after this time, we observed that the solution was clear, and that it showed no UV absorbance.

For reductions in *n*-pentane, the hydrocarbon products (with the exception of neopentane) were separated from the solvent by preparative GC on a F&M 700 instrument. We used a 1/4 in X 6 ft UCW-98 column operated at 150 °C with a helium flow of *ca* 30 mL/min. Elution times were: *n*-pentane, 30-90 s;

norbornane, *2*-3 min; homohypostrophane (the product of the reduction of homohypostrophene) 30-35 min.

Kinetics of Reductions. Previous studies of the kinetics of reduction of $DOPtR_2$ in alkane[50] and protic solvents[53] under the conditions used here showed that for a wide variety of diolefins and R groups (where R is alkyl) the kinetic features---- rates of reduction and zero-order dependence on the concentration of substrate----were similar. Based on these observations, and because we had only limited quantities of **1** and **2**, we did not perform an explicit investigation of the kinetics of reduction of these compounds. Nevertheless, since some platinum complexes, for example (norbornadiene)dimethylplatinum(II), react autocatalytically with H_2,[50] we needed to establish whether an autocatalytic reaction was important in the reductions of **1** and **2**.

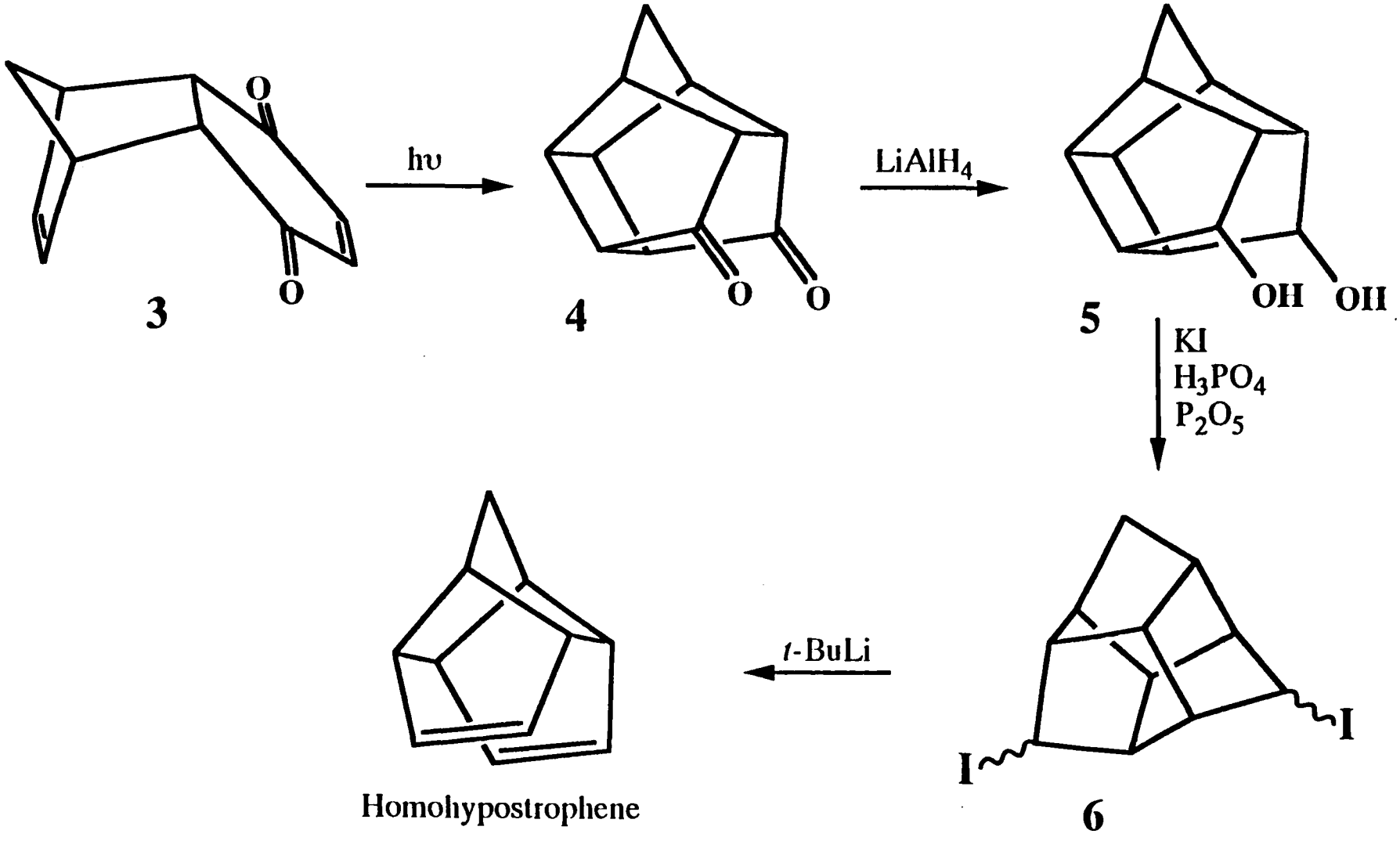
In order to test for autocatalysis, we ran blank reductions of mixtures of **1** and **2** in *n*-heptane without any catalyst present. Immediately after addition of the dissolved platinum complexes, we removed an aliquot ($t = 0$)[64] from the reactor. Stirring was resumed; simultaneously, a stopwatch was started. Every 15 min, an aliquot was removed until 90 min had elapsed. We diluted the aliquots under air by a factor of 100 by transferring 50 μL (using a 50 μL disposable glass micropipet) to a 5-mL volumetric flask, and filling it to the mark with solvent. The diluted solution was transferred to a 3.0-mL quartz cuvette (10 x 10 x 30 mm) using a disposable glass pipet. The UV absorbances did not diminish for either substrate during the 90-min period; therefore, autocatalysis did not contribute to the reduction of these compounds under the conditions employed here.

Synthesis of Tetracyclo[6.3.0.0^{4,11}.0^{5,9}]undeca-2,6-diene (Homohypostrophene). We synthesized homohypostrophene (*ca* 50-g scale) using the method of Smith and Barborak (Scheme II).[65] Since we observed that homohypostrophene decomposes to an insoluble white material on storage at -6 °C, we chose **6** as the immediate target of the large-scale synthesis, and converted this compound directly to homohypostrophene when desired.

1α, 4α, 4aβ, 8aβ-Tetrahydro-1,4-methanonaphthalene-5,8-dione, 3. From 154 mL of dicyclopentadiene (at 170 °C), we distilled 39 mL (470 mmol) of cyclopentadiene (at 44 °C) into a 50-mL graduated cylinder cooled to 0 °C. The cyclopentadiene was diluted into 90 mL of toluene at -78 °C. In a 1-L flask equipped with a magnetic stir bar, we dissolved 50.0 g (463 mmol) of benzoquinone in 370 mL of toluene. We added the cooled solution of cyclopentadiene dropwise to the stirred solution of benzoquinone via cannula, and periodically cooled the mixture with a dry ice/acetone bath. After the addition was complete, the solution was stirred for 34 h at room temperature. Removal of the solvent by rotary evaporation yielded 82 g of crude product whose ^{1}H NMR was consistent with that reported for **3**:[66] ($CDCl_3$, 250 MHz) δ 6.55 (s, 2 H), 6.05 (t, 2 H, J = 2 Hz), 3.53 (br s, 2 H), 3.20 (t, 2 H, J = 2 Hz), 1.47 (J_{AB} = 9 Hz, 2 H).

Pentacyclo[5.4.0.0^{2,6}.0^{3,10}.0^{5,9}]undecane-8,11-dione, 4. We dissolved 82 g of **3** in a minimum amount of ethyl acetate, and irradiated this solution in a quartz tube for 8 h with a 450 watt Ace-Hanovia mercury arc lamp. Irradiation produced an off-white precipitate that was collected and recrystallized from acetone to give, after drying, 64.6 g of **4**. The ^{1}H NMR spectrum of the product was consistent with that of a sample of **4** purchased from Aldrich: ($CDCl_3$, 300 MHz) δ 3.17 (br s, 2 H), 2.92 (m, 2 H,), 2.80 (m, 2 H), 2.70 (s, 2 H), 1.95 (J_{AB} = 11 Hz, 2 H).

Scheme II. Synthesis of Homohypostrophene.



3 4 5 6

Homohypostrophene

Pentacyclo[5.4.0.0^{2,6}.0^{3,10}.0^{5,9}]undecane-8,11-diol, 5. Into a 2-L round-bottomed flask equipped with a magnetic stir bar, we transferred 64.6 g of the dione **4** using 500 mL of diethyl ether. To this stirred solution under argon, we added dropwise 800 mL of $LiAlH_4$ (1*M* solution in diethyl ether). The mixture was stirred overnight, then excess hydride was decomposed by the slow addition of 30 mL of H_2O followed by 60 mL of 15% NaOH solution. We added *ca* 200 mL of 50% H_2SO_4, and separated the phases. We extracted the aqueous phase with $CHCl_3$ (6 x 100 mL), combined the organic phases, and dried them over magnesium sulphate. Filtration followed by rotary evaporation yielded 63 g of crude **5**. The ^1H NMR spectrum was consistent with that reported in the literature:[67] ($CDCl_3$, 250 MHz) δ 5.73 (s, 2 H; this resonance disappears upon addition of D_2O), 3.96 (br s, 2 H), 2.2-2.8 (m, 8 H) 1.37 (J_{AB} = 12 Hz, 2 H).

Diiodopentacyclo[6.3.0.0^{2,6}.0^{3,10}.0^{5,9}]undecane, 6. To a 2-L three-necked flask, we added 192 g of P_2O_5 and 253.5 g of 85% H_3PO_4. After the evolution of heat subsided, we added 364 g of KI, and 63 g of the diol **5**. After attaching a mechanical stirrer, a reflux condenser, and a drying tube to the flask, we placed it in an oil bath at 108 °C for 12 h with stirring. The mixture was allowed to sit at room temperature for *ca* 12 h, after which it was transferred to a 2-L separatory funnel using diethyl ether and H_2O. The layers were separated, and the aq phase was extracted with diethyl ether (2 x 500 mL). We washed the combined organic phases with 10% aq $Na_2S_2O_3$, and twice with H_2O. After drying the organic phase with magnesium sulphate, we added decolorizing carbon (Norit), and filtered the solution. Removal of the solvent by rotary evaporation provided a yellow oil. Trituration of this oil with acetone gave, after drying under vacuum, 89.3 g (224 mmol, 48% yield based on benzoquinone) of a white solid; the ^1H NMR spectrum of this material was consistent with that reported for a mixture of

stereoisomers, *syn*-4, *anti*-7- and *syn*-4, *syn*-7-diiodopentacyclo-[6.3.0.0^{2,6}.0^{3,10}.0^{5,9}]undecane:[65] ($CDCl_3$, 300 MHz) δ 3.95 (br s, 2 H), 2.0-3.4 (complex m, 8 H), 1.45 (m, 2 H).

Homohypostrophene. Under an atmosphere of argon, a flame-dried 100-mL round-bottomed flask containing a magnetic stir bar was charged with 5.03 g (12.6 mmol) of **6** and 50 mL of diethyl ether. To this solution, we slowly added 15 mL (26 mmol) of *tert*-butyllithium (1.7*M* in *n*-pentane) via cannula. The solution was stirred for 10 min after the addition was completed. We added H_2O carefully to quench any excess lithium reagent. A sufficient amount of water was then added to produce a clear biphasic solution. We extracted the aqueous phase with diethyl ether, combined the organic extracts, and dried them with magnesium sulphate. Filtration followed by careful rotary evaporation at 0 °C yielded a waxy white semi-solid which was sublimed. The sublimate was chromatographed on 10% $AgNO_3$ on silica gel. Elution with *n*-pentane provided 0.247 g of pentacyclo-[6.3.0.0^{2,6}.0^{3,10}.0^{5,9}]undecane (trishomocubane).[65] ^{1}H NMR: ($CDCl_3$, 300 MHz) δ 1.98 (m, 8H), 1.30 (s, 6H). Elution with 1:1 *n*-pentane/diethyl ether produced, after careful removal of the solvent, 0.885 g (6.1 mmol, 48% yield) of homohypostrophene.[65] ^{1}H NMR ($CDCl_3$, 250 MHz): δ 5.90 (s, 2 H), 3.14 (s, 2 H), 2.32 (s, 4 H), 1.63 (s, 2 H).

Tetracyclo[6.3.0.0^{4,11}.0^{5,9}]undecane, (Homohypostrophane, HOPH). Homohypostrophane (HOPH) is the product from the reduction of homohypostrophene with H_2 over platinum black. Following the procedure for reductions outlined above, we hydrogenated 0.104 g (0.72 mmol) of homohypostrophene in *n*-pentane. The solution was separated from the catalyst using a pipet, and the solvent was carefully removed by a flow of argon.

Sublimation of the resulting white solid gave 0.077 g (0.52 mmol; 72%) of HOPH as a waxy white solid. Mp: sub. MS: m/e (rel. int.) 148 (38) M^+, 119 (49), 91 (55), 81 (51), 80 (64), 79 (76), 77 (40), 67 (100), 66 (95), 41 (76), 39 (94), 27 (68). ^1H NMR ($CDCl_3$, 500 MHz, Figure 7):[68] δ 2.06 (m, 2 H, H_a), 1.98 (br s, 4 H, H_b), 1.56 (br d, 4 H, J = 8 Hz, H_c), 1.46 (s, 2 H, H_d), 1.45 (br d, 4 H, J ~ 8 Hz, H_e). ^{13}C NMR ($CDCl_3$, 100.6 MHz): 48.1, 43.0, 30.4, 25.5. Anal. Calcd. for $C_{11}H_{16}$: C, 89.12; H, 10.88; Found: C, 88.85; H, 10.90.

Synthesis of Grignard Reagents. Neopentylmagnesium chloride. Into a 500-mL round-bottomed flask containing a magnetic stir bar, we placed 10.0 g (0.411 mol) of magnesium chips. We wired a rubber septum onto the flask, and flame-dried the flask under a flow of argon. After the flask cooled, we added *ca* 300 mL of diethyl ether, initiated stirring, and slowly added via cannula 30.0 mL (0.244 mol) of neopentyl chloride in *ca* 50 mL diethyl ether. We replaced the septum with a reflux condenser, and refluxed the solution for *ca* one week under argon. Titration[69] showed that the solution was 0.6*M* in neopentylmagnesium chloride (*ca* 74% yield).

2-Norbornylmagnesium bromide. We synthesized this Grignard reagent several times using the following procedure. We placed 5.0 g (0.206 mol) of magnesium chips and a magnetic stir bar to a 200-mL round-bottomed flask. The flask was capped with a rubber septum, and flame-dried under argon. Diethyl ether (*ca* 80 mL) was added, stirring was started, and 10.0 mL (0.078 mol) of *exo*-2-bromonorbornane in *ca* 20 mL diethyl ether was added via cannula. A slow rate of addition was maintained so that the temperature of the flask was warm, but the solution was not refluxing. After the addition was complete, the solution was stirred for one hour, then allowed to sit overnight.

Titration[69] of these solutions typically showed the solutions to be 0.6-0.7*M* in 2-norbornylmagnesium bromide (*ca* 60-70% yield). According to prior reports, these solutions contained a mixture of *ca* 40% *exo*- and 60% *endo*-2-norbornyl-magnesium bromide.[58,60]

***endo*-2-Norbornylmagnesium bromide.** This Grignard reagent was synthesized using a variation on established procedures.[58,60] We transferred under argon 50.0 mL (30.0 mmol) of a 0.6*M* solution of 2-norbornylmagnesium bromide to a flame-dried, 100-mL Schlenk flask capped with a rubber septum, and containing a magnetic stir bar. The flask was placed in a -10 °C salt/ice-water bath, and stirring was initiated. We added via cannula a solution of 3.28 g of benzophenone (18.0 mmol, 0.6 equiv) in 10 mL of diethyl ether. The solution rapidly turned dark pink; a white precipitate was observed. The solution was allowed to stir for 5 min at -10 °C, after which the flask was placed in a dry ice/acetone bath at -78 °C. We attached a medium glass frit having male ground-glass joints at both ends to a 100-mL Schlenk flask. This filtration apparatus was flame-dried under a purge of argon. After cooling, we attached the apparatus under a flow of argon to the Schlenk flask containing the dark-pink solution. The solution was filtered quickly through the frit; the filtrate, presumably containing *endo*-2-norbornylmagnesium bromide,[58,60] was stored at -78 °C, and used shortly thereafter.

Synthesis of Platinum Complexes. (Homohypostrophene)-platinum(II)dichloride, HOPPtCl$_2$. Under argon, we added 3.51 g of a 3:1 mixture of homohypostrophene/trishomocubane (*ca* 18 mmol of homohypostrophene) in 25 mL of benzene to an orange suspension of 5.0 g (8.5 mmol) of Zeise's dimer in 100 mL of benzene in a 250-mL round-bottomed flask

equipped with a magnetic stir bar. Stirring for 24 h at room temperature produced a precipitate of white needles in a dark brown solution. We collected the precipitate by filtration through a medium glass frit, and washed it with benzene. Recrystallization of the precipitate from hot chloroform yielded 3.68 g (8.97 mmol) of $HOPPtCl_2$ as white needles (53% yield based on platinum). Mp: 252-300 °C dec. ^1H NMR ($CDCl_3$, 400 MHz): δ 6.38 ("t" with Pt satellites, J_{Pt-H} = 78 Hz, 4H), 3.27 ("t" with Pt satellites, J_{Pt-H} = 26 Hz, 4H) 3.21 (m, 2 H), 1.85 (s, 2 H). Anal. Calcd for $C_{11}H_{12}PtCl_2$: C, 32.21; H, 2.95. Found: C, 32.22; H, 2.65.

(Homohypostrophene)platinum(II)diiodide, $HOPPtI_2$. The diiodide was obtained in quantitative yield from $HOPPtCl_2$ by suspending the dichloride in acetone, saturating the solution with KI (the solution turned from clear to yellow immediately upon addition of KI), and stirring for three days. Aqueous workup and extraction with chloroform followed by recrystallization from hot chloroform yielded $HOPPtI_2$ as yellow needles. Mp: 247-285 °C dec. ^1H NMR ($CDCl_3$, 500 MHz): δ 6.47 ("t" with Pt satellites, J_{Pt-H} = 78 Hz, 4H), 3.30 (sep, J = 1.8 Hz, 2 H), 3.15 ("t" with Pt satellites, J_{Pt-H} = 27 Hz, 4H), 1.93 (s, 2 H). Anal. Calcd for $C_{11}H_{12}PtI_2$: C, 22.28; H, 2.04. Found: C, 22.25; H, 2.02.

(Homohypostrophene)dineopentylplatinum(II), $HOPPtNp_2$. This compound could be synthesized from either the corresponding dichloride or diiodide; we present here a representative example. In a flame-dried 100-mL Schlenk flask equipped with a magnetic stir bar, a suspension of 0.902 g (1.88 mmol) of $HOPPtI_2$ in diethyl ether (50 mL) was cooled to -78 °C under an atmosphere of argon. We added a 0.6*M* solution of neopentylmagnesium chloride (7.2 mL, 4.3 mmol) dropwise via cannula. The solution was stirred and allowed to warm slowly to 0 °C. Analysis by TLC (1:1 *n*-pentane/diethyl ether) showed the

reaction to be complete. We added excess H_2O slowly to quench excess Grignard reagent. The aqueous phase was extracted with diethyl ether, and the extracts dried over magnesium sulfate. Decolorizing carbon (Norit) was added, and the solution was filtered into a 250-mL round-bottomed flask. We concentrated the solution to dryness on a rotary evaporator, and obtained a yellow-green solid. This solid was chromatographed on silica gel using *n*-pentane as the eluant. The first fraction off the column (yellow, absorbs in the UV) contained the product. Removal of solvent followed by recrystallization from diethyl ether/methanol yielded yielded 0.683 g (1.42 mmol; 76% yield) of $HOPPtNp_2$. Mp: 106-107 °C. ^1H NMR (C_6D_6, 400 MHz): δ 5.62 ("t" with Pt satellites, J_{Pt-H} = 51 Hz, 4H), 2.66 (sep, J = 1.8 Hz, 2 H), 2.43 (br s, 4 H), 2.12 ("t" with Pt satellites, J_{Pt-H} = 92 Hz, 4H), 1.37 (s, J_{Pt-C} = 124 Hz, 18 H), 1.09 (s, 2 H). Anal. Calcd for $C_{21}H_{34}Pt$: C, 52.37; H, 7.12. Found: C, 52.63; H, 6.95.

(Homohypostrophene)neopentylplatinum(II)chloride, HOPPt(Np)Cl. In a 50-mL round-bottomed flask equipped with a magnetic stir bar, we dissolved 1.05 g (2.18 mmol) of $HOPPtNp_2$ in a minimum amount of *n*-pentane, and added 1 mL of conc HCl. After stirring for 2 h at room temperature, a white precipitate had formed, and analysis by TLC (1:1 *n*-pentane/diethyl ether) indicated that the reaction was complete. The solution was neutralized by adding saturated sodium bicarbonate solution. We extracted the aqueous phase with diethyl ether and dried the organic phase over magnesium sulfate. After filtration, rotary evaporation yielded an off-white solid that was chromatographed on silica gel (1:1 *n*-pentane/diethyl ether), and recrystallized from diethyl ether to afford glassy pale yellow plates of HOPPt(Np)Cl (0.888 g, 1.99 mmol; 91% yield). Mp: 148-149 °C. ^1H NMR ($CDCl_3$, 500 MHz): δ 6.40 (t of "t" with Pt satellites, J = 3, J_{Pt-H} = 44 Hz, 2H), δ 5.20 (t of "t" with Pt satellites, J = 3, J_{Pt-H} = 91 Hz, 2H), 3.23 (m, 1 H), 3.10

(m, 1 H), 3.04 (br m, 2 H), 2.91 (br m, 2 H), 1.66 ("t" with Pt satellites, J_{Pt-H} = 76 Hz, 2 H), 1.59 (br s, 2 H), 1.07 (s, J_{Pt-C} = 124 Hz, 9H). Anal. Calcd for $C_{16}H_{23}PtCl$: C, 43.10; H, 5.20; Found: C, 43.21; H, 4.99.

(Homohypostrophene)neopentylplatinum(II)iodide, HOPPt(Np)I. This compound was obtained from the corresponding chloride in quantitative yield by dissolving the chloride in a minimum amount of acetone, saturating the solution with KI (the solution turns from clear to yellow immediately upon addition of KI), and stirring for two days. Aqueous workup and extraction with diethyl ether followed by recrystallization from hot diethyl ether yielded HOPPt(Np)I as yellow prisms. Mp: 143-144 °C. ^{1}H NMR ($CDCl_3$, 500 MHz): δ 6.41 (t of "t" with Pt satellites, J = 3, J_{Pt-H} = 48 Hz, 2H), δ 5.35 (t of "t" with Pt satellites, J = 3, J_{Pt-H} = 89 Hz, 2H), 3.26 (m, 1 H), 3.10 (m, 1 H), 2.97 (br m, 2 H), 2.91 (br m, 2 H), 2.07 ("t" with Pt satellites, J_{Pt-H} = 81 Hz, 2 H), 1.63 (br s, 2 H), 1.09 (s, J_{Pt-C} = 124 Hz, 9H). Anal. Calcd for $C_{16}H_{23}PtI$: C, 35.76; H, 4.31; Found: C, 35.87; H, 4.21.

(Homohypostrophene)neopentyl(*exo*-2-norbornyl)platinum(II), 1. Under argon, we added via cannula 30.0 mL (18.0 mmol) of a 0.6*M* solution of 2-norbornylmagnesium bromide (*ca* 45% *exo* and 55% *endo*) in diethyl ether to a flame-dried 100-mL Schlenk flask equipped with a magnetic stir bar. We placed the flask in a -10 °C salt/ice-water bath, and added dropwise via cannula a yellow solution containing 0.252 g (0.565 mmol) of HOPPt(Np)Cl in diethyl ether. After one h, the solution had darkened; analysis by TLC (1:1 diethyl ether/*n*-pentane) showed that the reaction was complete. We added H_2O to destroy the excess Grignard reagent, extracted the aqueous phase with diethyl ether, combined the organic phases, and dried them over magnesium sulphate. After adding decolorizing carbon (Norit), the solution was filtered into a round-bottomed flask,

and concentrated to a yellow oil. This oil was chromatographed on silica gel using *n*-pentane as the eluant. The first few fractions (yellow, absorbs in the UV) were collected, and evaporated to dryness. The resulting yellow solid, recrystallized from diethyl ether/methanol, gave 83 mg (0.164 mmol, 29% yield) of a mixture of **1** (96%) and **2** (4%).[70] Mp: 98-120 °C dec. ^{1}H NMR (C_6D_6, 500 MHz): δ 5.55-5.80 (complex m, 4 H), 2.6-2.7 (complex m, 2 H), 2.35-2.55 (complex m, 6H), 2.02 (m, 1 H), 1.88-1.98 (complex m, 1 H),1.88 & 1.85 (J_{AB} = 11 Hz, 2 H), 1.79 (m, 1 H), 1.63 (m, 1 H), 1.51 (m, 2 H), 1.40 (m, 1 H), 1.35 (s, 9 H), 1.28 (m, 1 H), 1.09 (s, 2 H). ^{13}C NMR (C_6D_6, 125.8 MHz): 108.0, 107.3, 106.9, 105.8, 69.23, 69.18, 54.6, 54.0, 53.9, 53.8, 53.5, 46.4, 43.3, 42.4, 41.5, 41.3, 39.7, 38.8, 37.5, 36.13, 36.06, 35.97, 29.9. Anal. Calcd. for $C_{23}H_{34}Pt$: C, 54.64; H, 6.78; Found: C, 54.62; H, 6.56.

A similar synthesis on somewhat larger scale (0.256 g, 0.574 mmol HOPPt(Np)Cl) produced 0.163 g (32.2 mmol, 58% yield) of a mixture of **1** (90%) and **2** (10%). We used this mixture for the isotopic reductions.

(Homohypostrophene)neopentyl(*endo*-2-norbornyl)platinum(II), 2. A flame-dried, septum-capped 200-mL round-bottomed flask equipped with a magnetic stir bar was charged with 0.253 g (0.471 mmol) of HOPPt(Np)I in a minimum amount of diethyl ether, and cooled under argon to -10 °C in a salt/ice water bath. To this stirred solution, we added via cannula the solution containing the *endo*-2-norbornylmagnesium bromide (*ca* 18 mmol; *vide supra*). Over the course of 1 hr at -10 °C, the solution turned from yellow to brown. Analysis by TLC (1:1 *n*-pentane/diethyl ether) showed the presence of starting material (R_f ~ 0.5), and possible product (R_f ~ 0.9). Elution with *n*-pentane showed that the spot at R_f ~ 0.9 had several components. We quenched the reaction by adding H_2O.

The aqueous phase was extracted with diethyl ether; the extracts were combined and dried with magnesium sulphate. We added decolorizing carbon (Norit), and filtered the solution into a round-bottomed flask. Rotary evaporation yielded a yellow oil which was chromatographed on silica gel using *n*-pentane as the eluant. The first yellow fractions showed absorbance in the UV, and contained the desired product. Recrystallization from diethyl ether/methanol yielded 30 mg (0.059 mmol, 13% yield) of **2** as yellow needles. Mp: 111-180 °C dec. ^{1}H NMR (C_6D_6, 500 MHz): δ 5.79 (t of "t" with Pt satellites, J = 4.5, J_{Pt-H} = 52 Hz, 2 H), 5.62 (t of "t" with Pt satellites, J = 4.5, J_{Pt-H} = 48 Hz, 1 H), 5.55 (t of "t" with Pt satellites, J = 4.5, J_{Pt-H} = 52 Hz, 1 H), 3.03 (br s, 1 H), 2.80 ("t" with Pt satellites, J_{Pt-H} = 96 Hz, 1H), 2.66 (m, 2 H), 2.47 (m, 2 H), 2.42 (m, 2 H), 2.37 (m, 1 H), 1.99 & 1.84 (J_{AB} = 11 Hz, 2 H), 1.92 (complex m, 3 H), 1.70 (m, 2 H), 1.35-1.55 (complex m, 4H), 1.33 (s, 9 H), 1.09 (s, 2 H). Anal. Calcd. for $C_{23}H_{34}Pt$: C, 54.64; H, 6.78; Found: C, 54.58; H, 6.60.

Two subsequent syntheses produced 0.091 g (0.18 mmol, 39% yield) of a mixture of **2** (98%) and **1** (2%), and 0.062 g (12 mmol, 26% yield) of a mixture of **2** (97%) and **1** (3%).[70] We used the last mixture for the isotopic reductions.

Isotopic Analysis of Alkanes-d_n. For analyses by GC/MS, we used the average content of deuterium, d_{av} (eq 2), to describe the isotopic compositions of

$$d_{av} = 1/100 \sum_{n=1}^{m} n\,(\,\%\ \text{alkane-}d_n) \qquad (2)$$

the alkanes produced in the reductions.[51-53] In analyses by NMR, the values of d_{av} simply reflect the content of deuterium derived from integrations (*vide infra*). We believe that all values of d_{av} are accurate to ± 5% absolute.

Isotopic analyses by GC/MS were conducted using procedures analogous to those described earlier.[51-53] The relevant mass spectral data (m/e (rel. int.)) are: for norbornane, 96 (100.0) M^+, 95 (29.7), 97 (7.2); for HOPH, 148 (100.0) M^+, 149 (8.7). Distributions of the ions from norbornane were corrected for $(M - 1)^+$ by iteratively subtracting from the $(n - 1)$th peak the $(M - 1)^+$ percentage of the corrected value for the (n)th peak, and normalizing the resulting distribution. Distributions of ions for both molecules were corrected for natural abundance ot ^{13}C by iteratively subtracting from the nth peak the $(M + 1)^+$ percentage of the corrected value for the $(n - 1)$th peak, and normalizing the resulting distribution.[71] No other fragment ions with relative abundances > 1.0 fell within the range of relevant m/z.

In analyses by ^1H NMR, standard integration techniques using Bruker software were used to determine the isotopic content of norbornane-d_n and HOPH-d_n. For analyses of norbornane-d_n, we integrated H_b and $(H_c + H_d)$ relative to H_a (Fig 4); for analyses of HOPH-d_n, we integrated $(H_d + H_e)$ and H_c relative to H_a and H_b (Fig 7). We used a 10 s relaxation delay (relaxation delay + acquisition time totalled 13 s) to acquire the spectra.

We used an acquisition time of 2 s to collect the ^2H spectra of the norbornanes. No relaxation delay was employed. The resonances at δ 1.12 and 1.42 were integrated relative to each other by enlarging the printed spectra, cutting out the peak areas, and weighing them. Triplicate analyses of each spectrum differed by no more than 0.7%.

Results and Discussion

X-Ray Crystal Structures. Figures 1 and 2 are ORTEP plots of the structures of **1** and **2**. These structures are detailed in the Supplementary Material to this paper. Each compound clearly possesses a unique stereochemistry of bonding of the norbornyl group to platinum. The fact that the thermal ellipses in Figure 1 are larger than those in Figure 2 might reflect the fact that we collected the structure of **1** at 0 °C, and the structure of **2** at -58 °C.

NMR Spectra of the Norbornanes. Figure 3 shows the ^{1}H and ^{2}H NMR spectra of the norbornanes resulting from the reductions by D_2 of samples containing 90% **1** and 10% **2**, and 97% **2** and 3% **1**. These data show that the reduction of **1** incorporates deuterium into the *exo* position of norbornane, and the reduction of **2** incorporates deuterium predominantly into the *endo* position of norbornane. Assuming that the stereochemistry of bonding of the norbornyl moieties to platinum is maintained upon transfer to the surface(*vide infra*), these results argue that the reduction of C* bonds proceeds with predominant retention of configuration.

Formation of 2-Norbornyl* Occurs Without Loss of the Stereochemistry of Bonding Between the 2-Norbornyl Moieties and Platinum(II). Stereochemical[51] and kinetic[50] data provide support for this contention: the stereochemistry of reduction of the diolefin moieties of $DOPtR_2$ complexes indicate that the mechanism for reduction of these complexes occurs by initial adsorption at platinum (*vide infra*). Adsorption at platinum should not invert the stereochemistry of the norbornyl-Pt bond.

Figure 1. ORTEP drawing (30% probability level, showing atomic labelling scheme) for (homohypostrophene)neopentyl(*exo*-2-norbornyl)platinum(II) **1**. The molecule crystallizes in a non-centrosymmetric space group; the absolute configuration (*R*) was determined crystallographically. The data for this structure were collected at 0 °C.

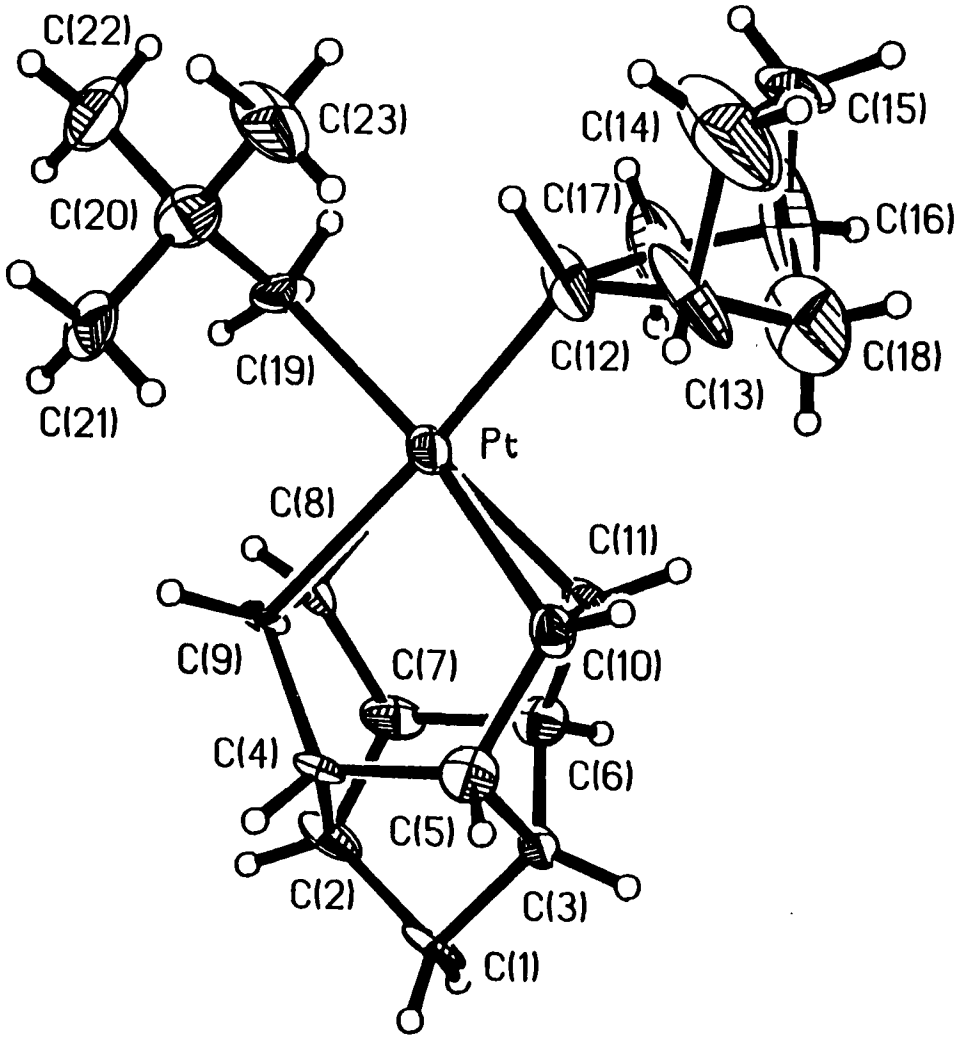

C(22)
C(23)
C(20)
C(21)
C(19)
C(17)
C(14)
C(15)
C(16)
C(12)
C(13)
C(18)
Pt
C(8)
C(11)
C(9)
C(7)
C(10)
C(4)
C(6)
C(5)
C(2)
C(3)
C(1)

Figure 2. ORTEP drawing (30% probability level, showing atomic labelling scheme) for (homohypostrophene)neopentyl(*endo*-2-norbornyl)platinum(II) **2**. The molecule crystallizes in a centrosymmetric space group; the *S* enantiomer is shown. The data for this structure were collected at -58 °C.

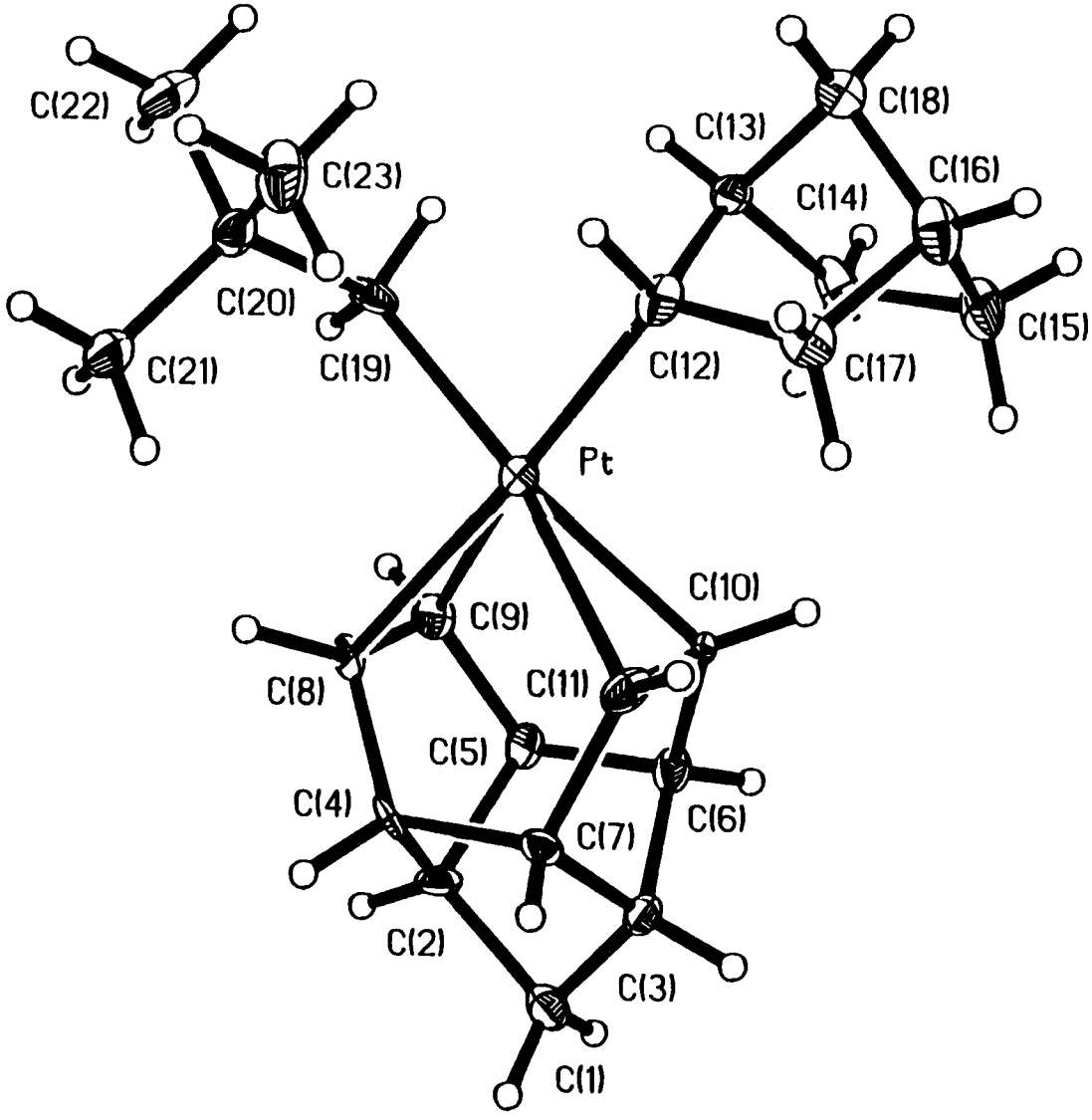
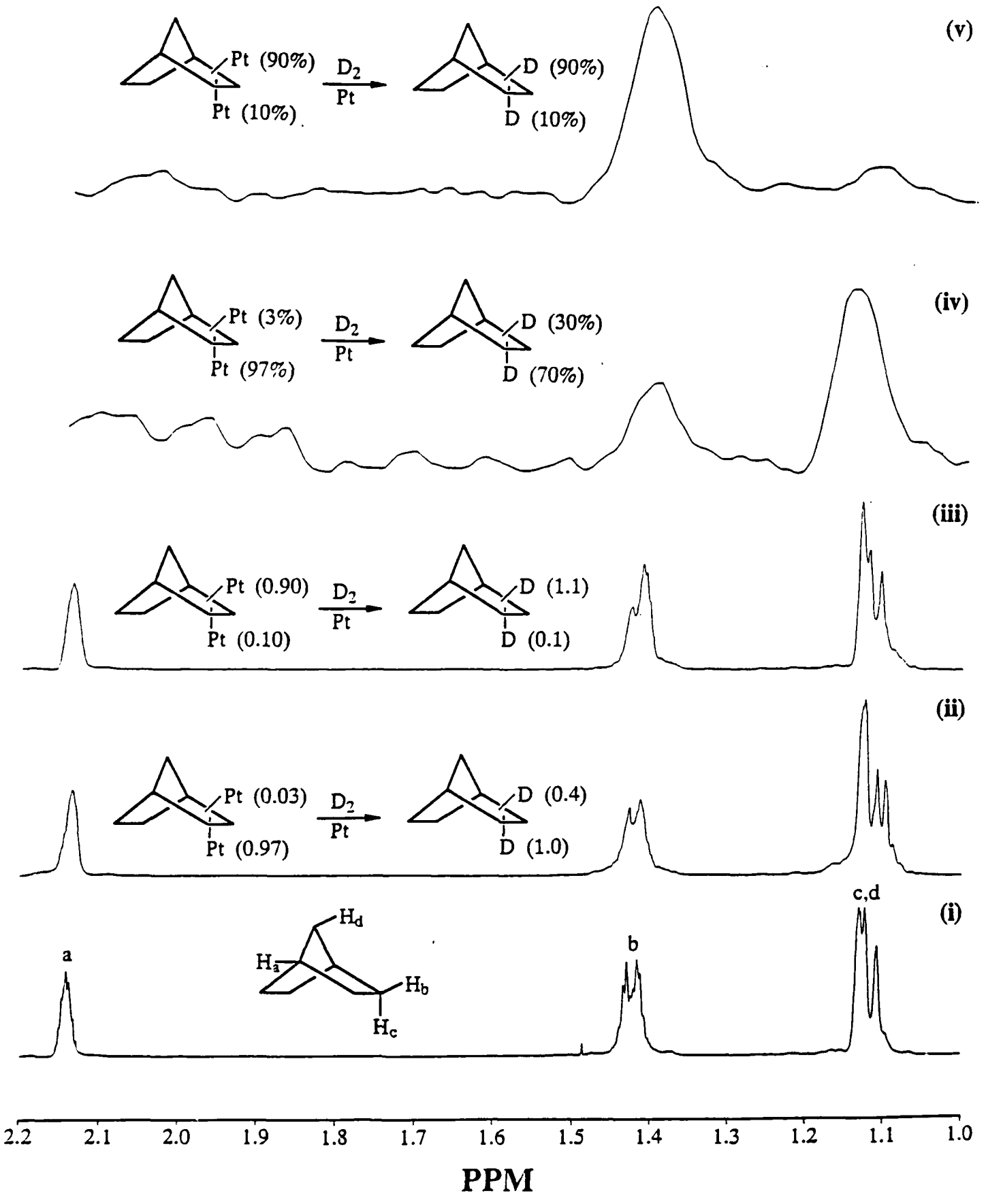

C(22)
C(23)
C(20)
C(21)
C(19)
C(18)
C(13)
C(16)
C(14)
C(15)
C(12)
C(17)
Pt
C(10)
C(9)
C(8)
C(11)
C(5)
C(4)
C(6)
C(7)
C(2)
C(3)
C(1)

Figure 3. ^{1}H NMR spectra (C_6D_6, 500 MHz) of (i) norbornane-d_0, and the norbornanes from the reduction by D_2 of samples containing (ii) 97% **2** and 3% **1**, and (iii) 90% **1** and 10% **2**. ^{2}H NMR spectra (C_6D_6, 46.03 MHz) of the norbornanes from the reductions by D_2 of samples containing (iv) 97% **2** and 3% **1**, and (v) 90% **1** and 10% **2**.


(v)
Pt (90%)
Pt (10%)
D2
Pt
D (90%)
D (10%)
(iv)
Pt (3%)
Pt (97%)
D2
Pt
D (30%)
D (70%)
(iii)
Pt (0.90)
Pt (0.10)
D2
Pt
D (1.1)
D (0.1)
(ii)
Pt (0.03)
Pt (0.97)
D2
Pt
D (0.4)
D (1.0)
(i)
a
Hd
Ha
Hb
Hc
b
c,d
2.2
2.1
2.0
1.9
1.8
1.7
1.6
1.5
1.4
1.3
1.2
1.1
1.0
PPM

The rate determining step in the heterogeneous hydrogenations of $DOPtR_2$ complexes has not been unambiguously identified, but the activation energy for the reduction of (1,5-cyclooctadiene)dimethylplatinum(II) ($CODPtMe_2$) over platinum black in *n*-heptane is 15 ± 2 kcal/mol,[50] and that for inversion at a methyl carbon (e.g. S_N2 displacement on MeI and MeBr) is typically 15-20 kcal/mol.[72] The similar magnitude of these activation energies suggests that if inversion at carbon occurs in the reductions of $DOPtR_2$ complexes, the rates of these reductions should be influenced by structure in ways similar to those well established for S_N2 reactions. In fact, the reductions of $CODPtR_2$ complexes and S_N2 displacements on alkyl iodides (taken as a representative set) follow very different patterns of relative rates. For the former reaction, the relative rates of reduction are $CODPtMe_2$ (1.0), $CODPtEt_2$ (1.0), CODPt(*iso*-Pr)$_2$ (0.69), CODPt(*iso*-Bu)$_2$ (0.40), $CODPtNp_2$ (0.23), and $CODPtPh_2$ (0.60);[50] for the latter, the relative rates of displacement by Cl^- with inversion at carbon are MeI (1.0), EtI (0.090), *iso*-PrI (0.0029), *iso*-BuI (0.0034), NpI (0.0000013), and PhI (0.0).[72] The absence of a correlation between the rates of reduction of $DOPtR_2$ complexes and the rates of inversion at carbon in S_N2 reactions is compatible with the hypothesis that the mechanism for the reduction of the platinum complexes involves retention at C1 of the R group in the reaction R-Pt ----> R*.

Mass Spectral Analysis of the Norbornanes. Figure 4 provides the mass spectral data for the norbornanes produced in the reductions by D_2 of norbornene, and samples containing 90% **1** and 10% **2**, and 97% **2** and 3% **1**. Norbornane-d_1 is the major product from reductions of the platinum complexes, and norbornane-d_2 is the major product from the reduction of norbornene.

Figure 4. Isotopic distributions determined from the mass spectra of the norbornanes produced from the reductions by D_2 of norbornene (bottom), a mixture of 97% **2** and 3% **1** (middle), and a mixture of 90% **1** and 10% **2** (top). The percent composition shown for each isotopomer is probably accurate to ± 5%.

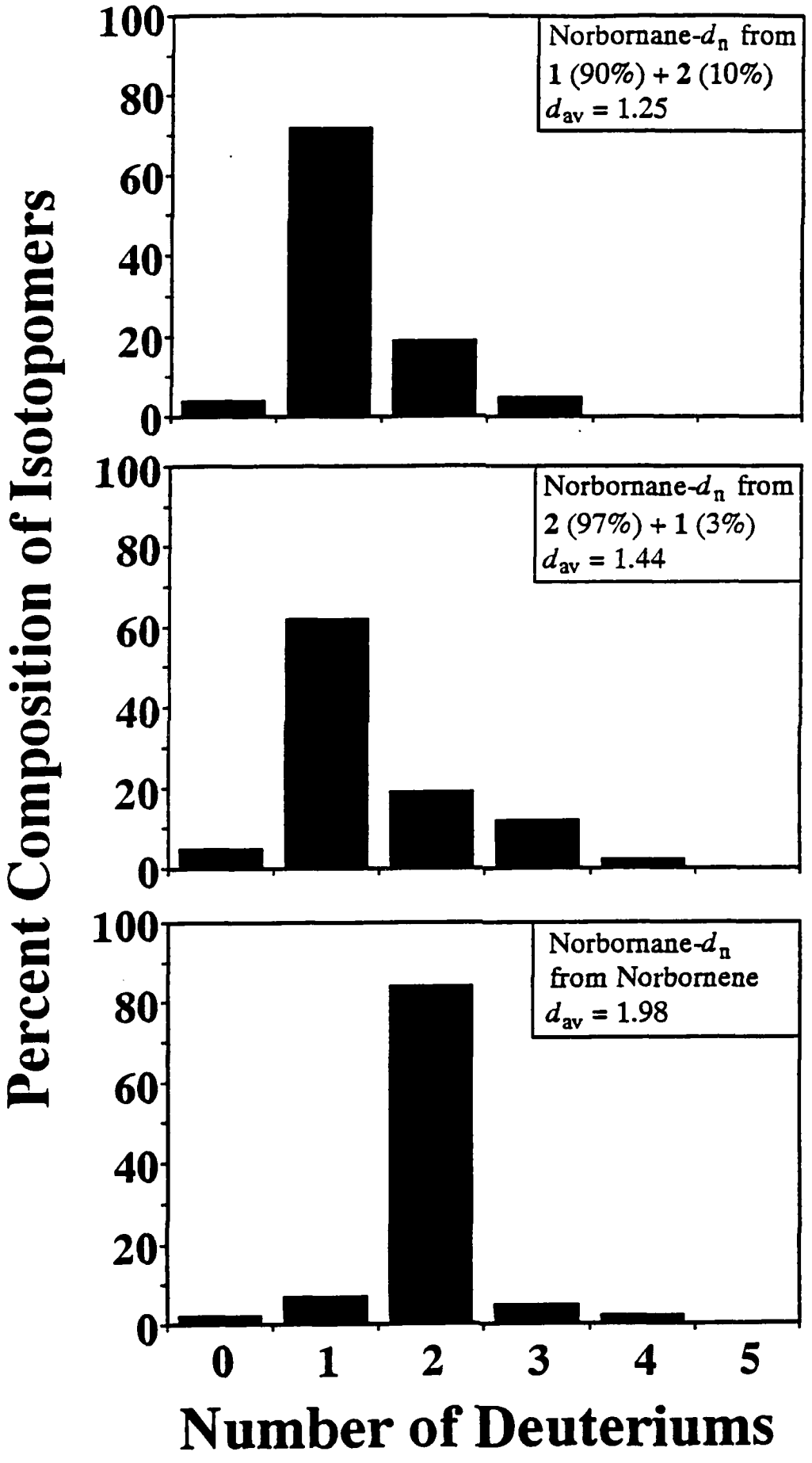
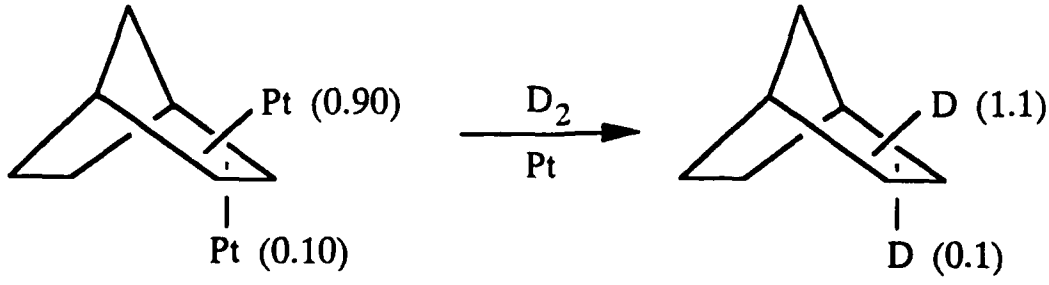
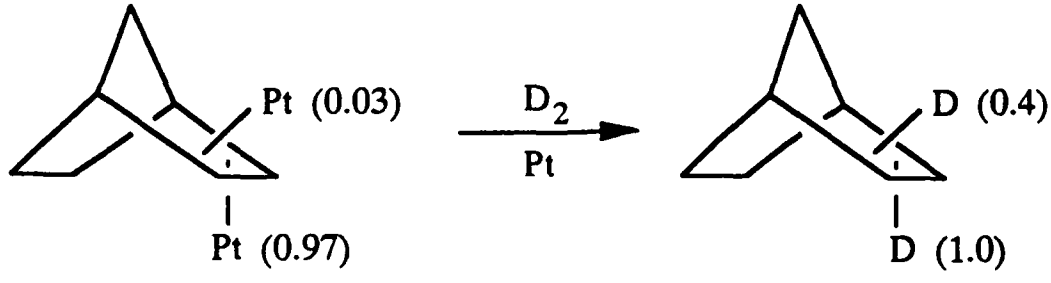

Norbornane-d_n from
1 (90%) + 2 (10%)
d_{av} = 1.25
Norbornane-d_n from
2 (97%) + 1 (3%)
d_{av} = 1.44
Norbornane-d_n
from Norbornene
d_{av} = 1.98
Percent Composition of Isotopomers
100
80
60
40
20
0
0 1 2 3 4 5
Number of Deuteriums

Table I gives the values of d_{av} determined from the mass spectral and ^1H NMR analyses of these norbornanes, and shows that the results from both analytical methods are in good agreement. The fact that these numbers agree indicates that activation of H_a and H_d (more correctly, incorporation of deuterium into these positions) does not occur. The norbornanes produced from both platinum complexes contain more than 1.0 D (the number of equivalents of deuterium expected based on the stoichiometry of the reaction). The incorporation of excess deuterium probably results from either (or both) α-H activation, or β-H activation (β-H elimination) prior to reductive elimination of 2-norbornyl* moieties from the surface (*vide infra*). Since it is unlikely under these conditions that every activation of a C-H bond results in subsequent incorporation of deuterium, we define an activation-incorporation event as an incorporation (e.g. α incorporation or β incorporation).

The isotopic composition of the norbornanes produced from the reductions by D_2 of samples containing **1** and **2** are summarized in eq 3 and 4. Equation 3



(3)



(4)

Table I. Isotopic Compositions (d_{av}) of the Alkanes-d_n from the Reductions by D_2 of **1**, **2**, Homohypostrophene, and Norbornene.[a]

Alkane-d_n	Substrate	d_{av} (MS)	d_{av} (^{1}H NMR)
Homohypostrophane	**1** (90%) + **2** (10%)	5.22	5.26
	2 (97%) + **1** (3%)	5.24	5.30
	homohypostrophene	3.88	3.84
Norbornane	**1** (90%) + **2** (10%)	1.25	1.17
	2 (97%) + **1** (3%)	1.44	1.35
	norbornene	1.98	-----[b]

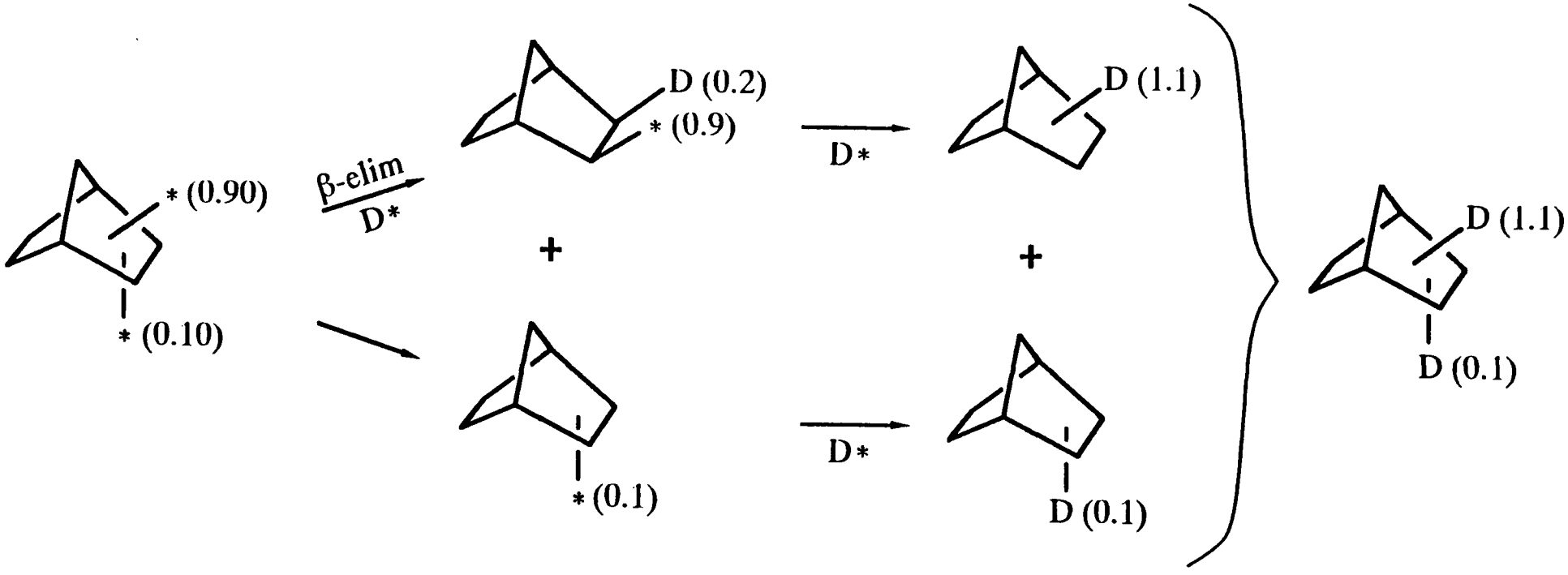
[a] The values of d_{av} are probably accurate to ± 5% absolute.

[b] The content of deuterium was not determined by ^{1}H NMR for this substrate.

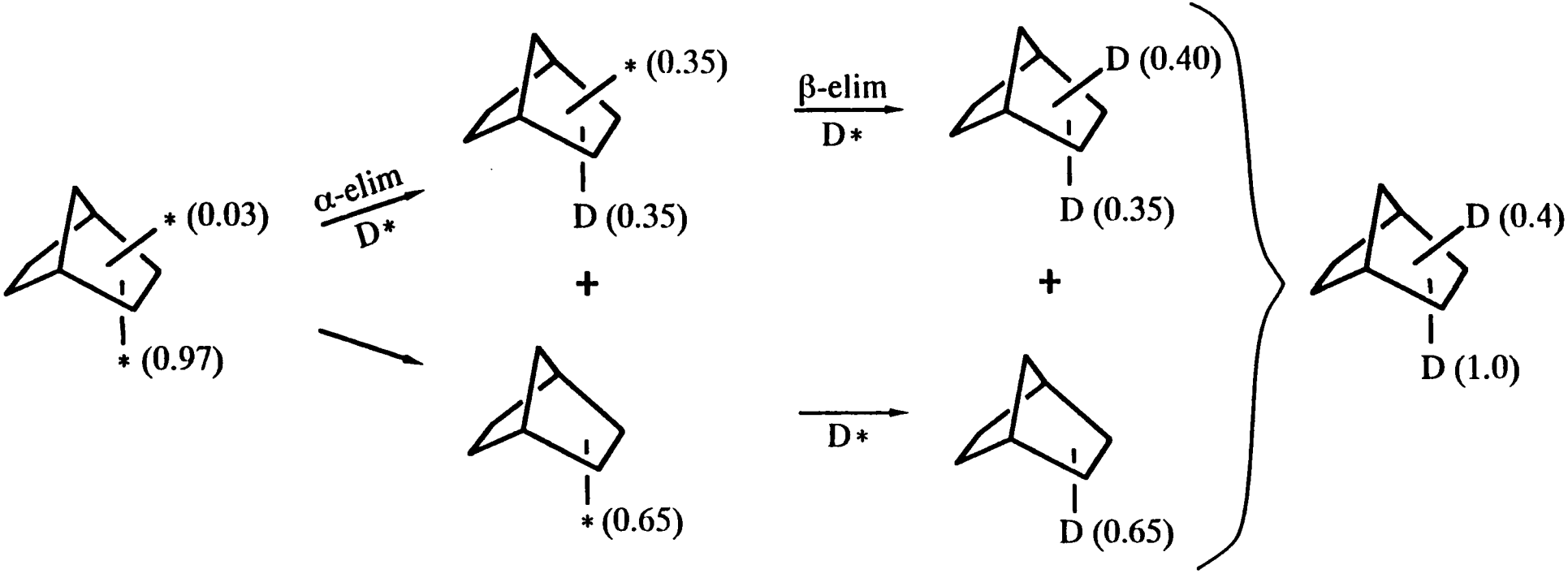
shows, for example, that the reduction by D_2 of a mixture of 90% **1** and 10% **2** produces norbornanes containing 1.1 D in the *exo* position, and 0.1 D in the *endo* position. The observation that the incorporation of deuterium into the *endo* position does not exceed 0.1 D suggests that excess deuterium does not result from β incorporation into *endo*-2-norbornyl* (*endo**) moieties, nor from α incorporation into *exo*-2-norbornyl* (*exo**) moieties. On the other hand, β incorporation[73] of deuterium into *ca* 20% of the *exo** moieties adequately accounts for the excess deuterium, and is qualitatively consistent with the isotopic distributions shown in Figure 4 (*ca* 70% norbornane-d_1 and 20% norbornane-d_2) for the reduction of this mixture. Scheme III summarizes these proposals.

In the reduction of samples containing 97% **2** and 3% **1**, the presence of 3% **1** is not significant, and will not be considered in the discussion here. The total amount of deuterium incorporated into the norbornanes (1.4 D) is described by eq 4: 1.0 D in the *endo* position, and 0.4 D in the *exo* position. Since the incorporation of deuterium into the *endo* position does not exceed 1.0 D, the excess deuterium probably does not result from β incorporation of deuterium into the *endo** moieties. Scheme IV summarizes the proposed reactions occurring in the reduction of *endo**. The incorporation of excess deuterium into *endo** moieties probably occurs via α incorporation[74,75] and epimerization of *ca* 35% of the *endo** moieties to *exo** moieties followed by β incorporation of deuterium into *ca* 20% of the subsequent *exo** moieties.[76] The reactions shown in Scheme IV rationalize the incorporation of excess deuterium, and are in qualitative agreement with the isotopic distributions shown in Figure 4 (e.g. *ca* 60% norbornane-d_1, 20% norbornane-d_2, and 10% norbornane-d_3) for the reduction of 97% **2** and 3% **1**.

Scheme III. Proposed Reactions of the Norbornyl* Moieties Generated in the Reduction by D_2 of a Mixture of 90% **1** and 10% **2**. Hydrogen atoms have been omitted for clarity.


* (0.90)
* (0.10)
β-elim
D*
D (0.2)
* (0.9)
+
* (0.1)
D*
D (1.1)
+
D*
D (0.1)
D (1.1)
D (0.1)

Scheme IV. Proposed Reactions of the Norbornyl* Moieties Generated in the Reduction by D_2 of a Mixture of 97% **2** and 3% **1**. Hydrogen atoms have been omitted for clarity.


* (0.03)
* (0.97)
α-elim
D*
* (0.35)
D (0.35)
+
* (0.65)
β-elim
D*
D (0.40)
D (0.35)
+
D*
D (0.65)
D (0.4)
D (1.0)

In summary, the reduction of *exo** moieties occurs via (1) simple reductive elimination (major pathway, *ca* 70%), and (2) β incorporation of deuterium prior to reductive elimination (minor pathway, *ca* 20%). Since α incorporation into *exo** does not occur, α-H activation of *exo** probably does not occur; hence, epimerization from *exo** to *endo** does not occur. The reduction of *endo** moieties occurs via (1) simple reductive elimination (major pathway, *ca* 65%), and (2) α-H activation and epimerization to *exo** followed by reduction of *exo** as described above (minor pathway, *ca* 35%). Since β incorporation into *endo** does not occur, β-H activation (β-H elimination) of *endo** probably does not occur. The reasons for the differences in reactivity between *exo** and *endo** cannot be determined from the experimental data. In the following two paragraphs, we provide largely speculative rationalizations[77] for the apparent differences in reactivity between these surface moieties assuming that the rate-limiting step for production of norbornane is reductive elimination from the surface.[50-52]

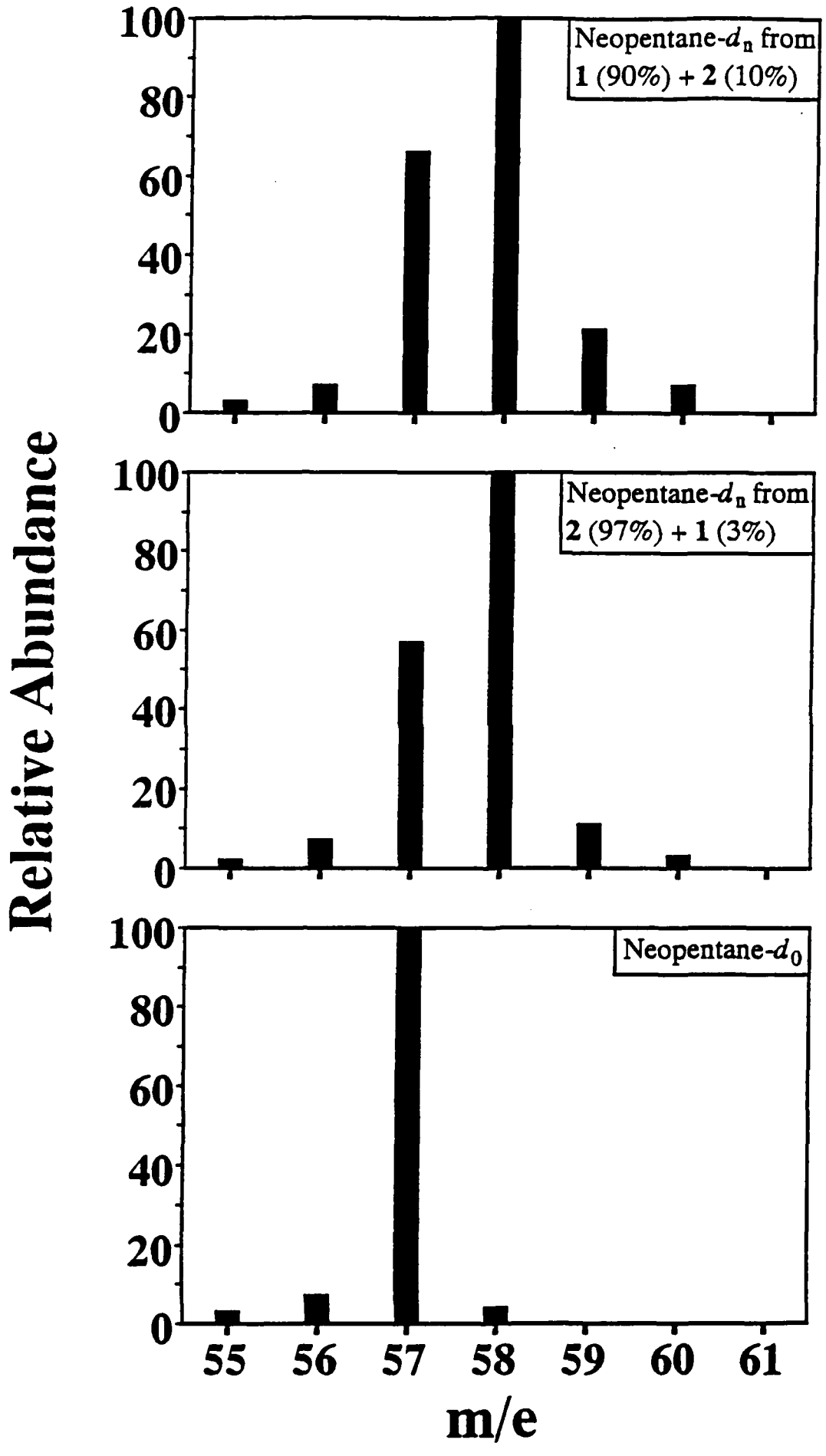
The observation of α-H activation/epimerization of *endo** to *exo**, but not of *exo** to *endo**, can be rationalized on the usual basis of a steric preference for *exo* by large substituents (here the surface of platinum). The following argument assumes that the barriers to reductive elimination as norbornane are similar for *exo** and *endo**. A norbornyl group bonded *endo* to Pt* is probably energetically destabilized (relative to *exo**) due to the unfavorable steric interactions between the distal *endo* hydrogens and the surface of platinum; consequently, the barrier to conversion of *endo** to *exo** is relatively low, and the rate of conversion of *endo** to *exo** is competitive with the rate of reductive elimination of *endo*-2-norbornyl groups from the surface. The transition states for conversion of *endo** to *exo** and *exo** to *endo** are probably the same. Since *exo** is a more stable surface species than *endo**, the barrier to conversion of *exo** to *endo** is relatively high, and the

rate of conversion of *exo** to *endo** is negligible in comparison to the rate of reductive elimination of *exo*-2-norbornyl groups from the surface.

The observation of β-H elimination in *exo** moieties, but not in *endo** moieties can also be rationalized assuming that that the barriers to reductive elimination as norbornane are similar for *exo** and *endo**. The formation of *endo*-norbornene* from *endo** is disfavored because *endo*-norbornene* is destabilized relative to *endo** due to increased steric repulsions between the distal *endo* hydrogens and the surface of platinum. Consequently, the energy of the transition state for β-H elimination in *endo** is high relative to that for reductive elimination of this moiety; the rate of β-H elimination is, therefore, negligible compared to the rate of reductive elimination. Formation of *exo*-norbornene* from *exo** is probably less sterically demanding than formation of *endo*-norbornene* from *endo**. As a consequence, the transition state for β-H elimination in *exo** is comparable in energy to that for reductive elimination of this species; hence, the rate of β-H elimination is competitive with that for reductive elimination.

Mass Spectra of the Neopentanes. Figure 5 shows the mass spectral data for neopentane-d_0, and the neopentanes produced in the reductions by D_2 of mixtures of **1** and **2**. No M^+ ion is observed in the mass spectrum of neopentane: the base peak is the expected $(M\text{-}CH_3)^+$ ion. We are reluctant to infer detailed isotopic compositions of the neopentanes from these data since we do not know isotope effects for loss of, for example, CH_3 relative to CH_2D; we can, however, infer qualitatively that the neopentanes from the reduction of **1** and **2** are predominantly composed of neopentane-d_1, and that the same isotopic species is (are) produced from both **1** and **2**.

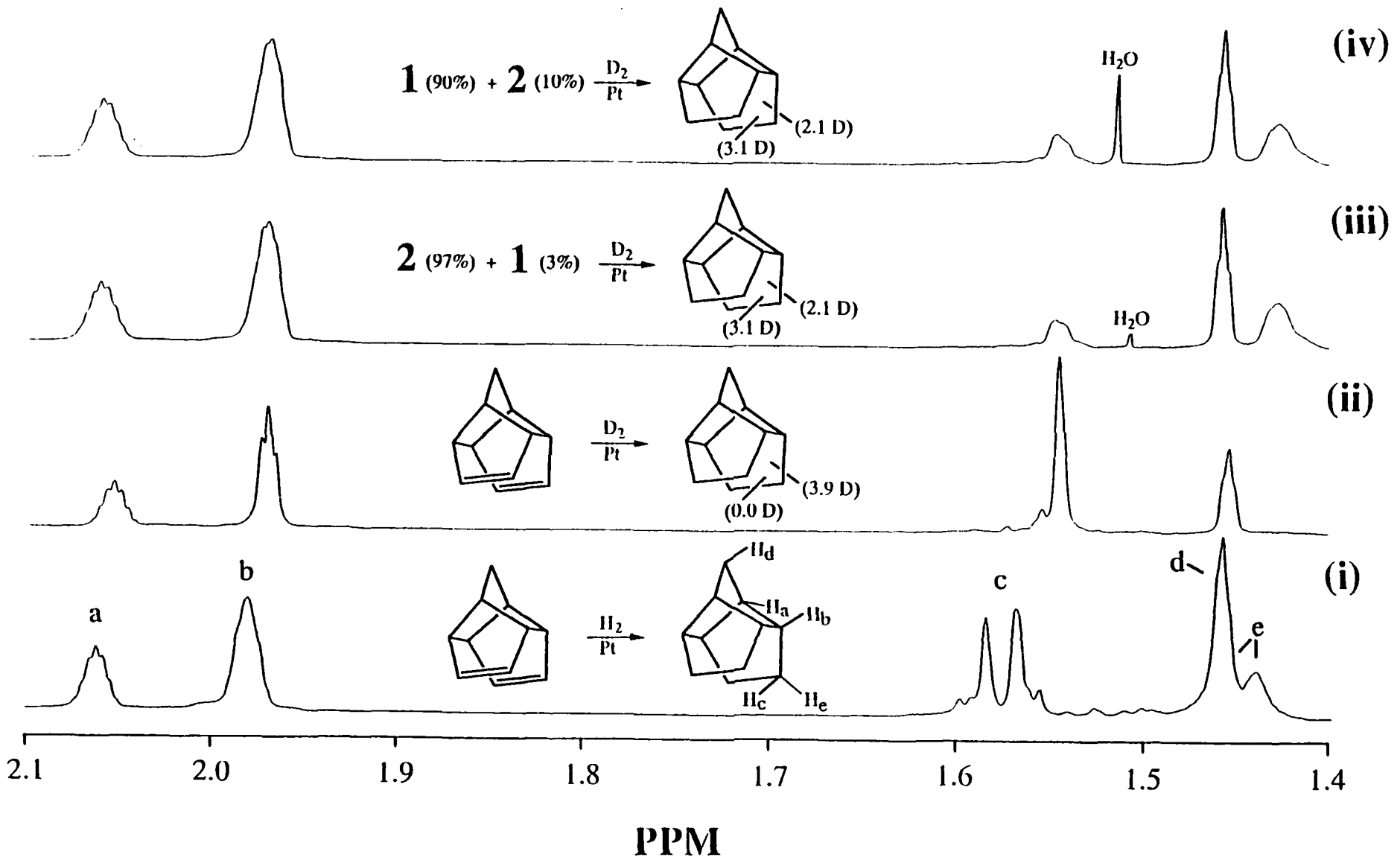
Figure 5. Mass spectra of neopentane-d_0 (bottom), and the neopentanes produced from the reductions by D_2 of a mixture of 97% **2** and 3% **1** (middle), and 90% **1** and 10% **2** (top).


Relative Abundance
100
80
60
40
20
0
Neopentane-d_n from
1 (90%) + 2 (10%)
Neopentane-d_n from
2 (97%) + 1 (3%)
Neopentane-d_0
55
56
57
58
59
60
61
m/e

^{1}H NMR Spectra of the Homohypostrophanes. Figure 6 compares the ^{1}H NMR spectra of the homohypostrophanes (HOPH-d_n) produced in the reduction by H_2 of homohypostrophene, and the reductions by D_2 of homohypostrophene and samples containing **1** and **2**. The assignments of the ^{1}H resonances are described in the experimental section.[68,78] In the reduction by D_2 of homohypostrophene, the loss of the resonance attributed to the *exo* protons of HOPH indicates that deuterium adds exclusively to the *exo* positions of HOPH. Analogous reductions of samples containing predominantly **1** and predominantly **2** are indistiguishable from one another, and less isotopically clean than reduction of homohypostrophene. In the reductions of **1** and **2**, the loss of the resonance attributed to the *endo* protons of HOPH predominates: *ca* 3.1 H are lost from the *endo* positions and 2.1 H are lost from the *exo* positions. This observation indicates that deuterium adds predominantly to the *endo* positions of the HOP moiety originally coordinated to platinum in **1** and **2**.

Qualitatively, these data suggest that the reduction of coordinated homohypostrophene proceeds with stereochemistry that is predominantly opposite to that of the reduction of free homohypostrophene. The observation that deuterium is incorporated predominantly into the faces of the olefins in homohypostrophene that were coordinated to the platinum atoms in **1** and **2** indicates that adsorption on the surface occurs through initial attachment of the platinum atoms of these complexes. These results provide further support for our proposal that the mechanism of reduction of $DOPtR_2$ proceeds via initial association of the platinum atom with the surface of the catalyst: we observed previously that the reduction of coodinated norbornadiene (NBD) in $NBDPtMe_2$ proceeds with stereochemistry that is predominantly opposite to that of the reduction of free NBD.[51] Taken together, these results argue that the adsorption of the diolefin

Figure 6. ^{1}H NMR spectra ($CDCl_3$, 500 MHz) of the tetracyclo[6.3.0.0^{4,11}.0^{5,9}]-undecanes (homohypostrophanes) from: (i) the reduction by H_2 of homohypostrophene,[68] the reduction by D_2 of (ii) homohypostrophene, (iii) samples containing 97% **2** and 3% **1**, and (iv) samples containing 90% **1** and 10% **2**. The changes in chemical shifts between (i)-(iv) are probably due to isotope effects.[78]

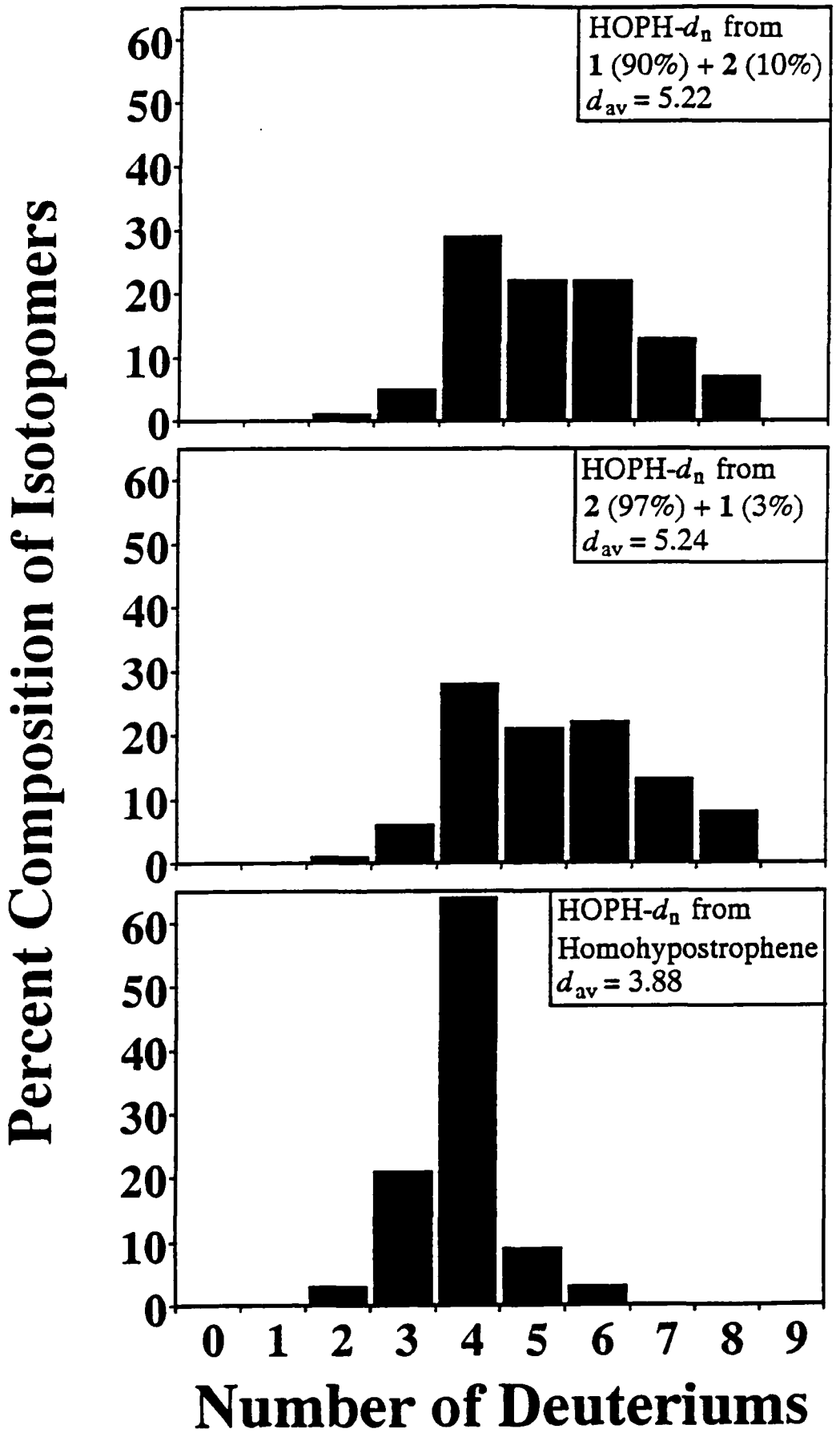

1 (90%) + 2 (10%)
D2
Pt
(2.1 D)
(3.1 D)
H2O
(iv)
2 (97%) + 1 (3%)
D2
Pt
(2.1 D)
(3.1 D)
H2O
(iii)
D2
Pt
(3.9 D)
(0.0 D)
(ii)
H2
Pt
Hd
Ha
Hb
Hc
He
a
b
c
d
e
(i)
2.1
2.0
1.9
1.8
1.7
1.6
1.5
1.4
PPM

moieties of $DOPtR_2$ on the surface of the catalyst proceeds with retention of configuration, and are consistent with our assumption that the adsorption of the norbornyl moieties from **1** and **2** proceeds with retention of configuration.

Mass Spectral Data for the Homohypostrophanes. Figure 7 provides the mass spectral data for the homohypostrophanes produced in the reductions by D_2 of homohypostrophene and mixtures of **1** and **2**. In all cases the major isotopomer produced is HOPH-d_4. The reduction of homohypostrophene produces HOPH-d_4 relatively cleanly (> 60%). Reductions of **1** and **2**, however, produce significant quantities of other isotopomers, HOPH-d_n (n = 5-8).

The broader distribution of isotopomers of HOPH produced from the reductions of **1** and **2** relative to that produced from the reduction of homohypostrophene probably results from steric destabilization of the *endo*, *endo* bound surface diolefin, or the *endo*-surface alkyl. This additional strain energy probably allows the rate of other processes (e.g. isomerization of *endo*-HOP* to *exo*-HOP*) to become competitive with the rate of reductive elimination from the surface.

Figure 7. Isotopic distributions determined from the mass spectra of the tetracyclo[6.3.0.0^{4,11}.0^{5,9}]undecanes (homohypostrophanes) produced in the reductions by D_2 of homohypostrophene (bottom), a mixture of 97% **2** & 3% **1** (middle), and a mixture of 90% **1** & 10% **2** (top). The percent composition shown for each isotopomer is probably accurate to ± 5%.


HOPH-d_n from
1 (90%) + 2 (10%)
d_{av} = 5.22
HOPH-d_n from
2 (97%) + 1 (3%)
d_{av} = 5.24
HOPH-d_n from
Homohypostrophene
d_{av} = 3.88
Percent Composition of Isotopomers
0
10
20
30
40
50
60
0 1 2 3 4 5 6 7 8 9
Number of Deuteriums

Conclusions

The major conclusions from this work are:

1. *The stereochemistry of the reduction of C* bonds by H* (D*) proceeds with retention of configuration.* This conclusion is based on the observation that the reduction of **1** with D_2 incorporates deuterium into the *exo* position of norbornane, and that the reduction of **2** with D_2 incorporates deuterium predominantly into the *endo* position of norbornane. Our proposed mechanisms for the reductions of exo-*2-norbornyl** and endo-*2-norbornyl** moieties (Schemes III and IV) argue that final reductive elimination of C* bonds from the surface proceeds with absolute retention of configuration. These arguments rely, however, on the correctness of the assumption that the adsorptions of **1** and **2** to form 2-norbornyl* moieties proceeds without loss of the stereochemistry of bonding between the 2-norbornyl moieties and platinum(II). Support for this assumption is detailed in the **Results and Discussion** section of this paper.

2. *The reduction of* exo-*2-norbornyl* moieties is relatively clean; the reduction probably proceeds via simple reductive elimination from the surface (major pathway,* ca *70%), and β-H elimination and incorporation of deuterium prior to reductive elimination from the surface (minor pathway,* ca *20%).* The results which support this conclusion are (i) the incorporation of excess deuterium into the *exo* position of norbornane in the reduction by D_2 of a sample containing 90% **1** and 10% **2**, and (ii) the distribution of isotopomers of norbornane produced from this reduction.

3. *The reduction of* endo-*2-norbornyl* moieties is less straightforward; the reduction probably proceeds via simple reductive elimination from the surface*

(major pathway. ca *65%), and α-H activation and epimerization to* exo-*2-norbornyl* moieties followed by the reduction of these species as described in conclusion 2 (minor pathway,* ca *35%).* This conclusion is supported by (i) the incorporation of excess deuterium into the *exo* position of norbornane in the reduction by D_2 of a sample containing 97% **2** and 3% **1**, and (ii) the distribution of isotopomers of norbornane produced in this reaction.

The differences in reactivity between *exo** moieties and *endo** moieties probably result from greater steric destabilization of *endo** than of *exo**. Repulsions between the surface of platinum and the *endo* hydrogens of the *endo** moieties are responsible for this additional destabilization.

4. *The reduction of $DOPtR_2$ complexes occurs via initial adsorption of the platinum atom in the organometallic complex.* Support for this conclusion is the observation that the reductions by D_2 of **1** and **2** incorporate deuterium predominantly into the *endo* positions of homohypostrophane, but the reduction of homohypostrophene by D_2 incorporates deuterium exclusively into the *exo* positions of homohypostrophane.

Supplementary Material. We provide the following for **1** and **2:** procedures for the determination of structure, summary of the crystallographic data, atomic coordinates and equivalent isotropic displacement parameters for non-hydrogen atoms, complete tables of bond distances and angles, anisotropic displacement parameters for non-hydrogen atoms, coordinates for hydrogen atoms, observed and calculated structure factors, packing diagrams, and UV absorption spectra (44 pages). Ordering information is given on any current masthead page.

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(74) There is much support in the literature for the existence of αα-di-sigma-absorbed species. See for example: Kesmodel, L.L.; Dubois, L. H.; Somorjai, G. A. *Chem. Phys. Lett.* **1978**, *56*, 267-271; Kesmodel, L.L.; Dubois, L. H.; Somorjai, G. A. *J. Chem. Phys.* **1979**, *70*, 2180-2188; Albert, M. R.; Sneddon, L. G.; Eberhardt, W.; Greuter, F.; Gustafsson, T.; Plummer, E. W. *Surf. Sci.* **1982**, *120*, 19-37; references 6 and 10.

(75) We have have indicated that the epimerization of *endo** to *exo** occurs via αα-di-sigma-absorbed species. The epimerization might occur, however, via sigma-adsorbed olefins. See for example: Zaera, F.; Hall, R. B. *J. Phys. Chem.* **1987**, *91*, 4318-4323; Naito, S.; Mitsutoshi, T. *J. Catal.* **1986**, *102*, 377-385; Mintsa-Eya, V.; Hilaire, L.; Choplin, A.; Tourode, R.; Gault, F. G. *J. Catal.* **1983**, *82*, 267-278; references 5 and 19.

(76) We did not detect norbornene during reductions of **2**; this observation does not, however, rule out the possibility that desorption of norbornene occurs during the reduction.

(77) We phrase the arguments in terms of kinetics; thermodynamics might, however, be responsible for the observations: we believe that *exo** is lower in potential energy than *endo**; this difference in potential energy could influence the incorporation of deuterium even if these species are in equilibrium.

(78) The changes in the chemical shifts of HOPH upon incorporation of deuterium are not due to concentration effects: a mixture (*ca* 1:1) of HOPH-d_0 and HOPH-d_4 (from the reduction by D_2 of homohypostrophene) gave chemical shifts consistent with those shown in Figure 7.

Figure 6. ^1H NMR spectra ($CDCl_3$, 500 MHz) of the tetracyclo[6.3.0.0^{4,11}.0^{5,9}]-undecanes (homohypostrophanes) from: (i) the reduction by H_2 of homohypostrophene,[68] the reduction by D_2 of (ii) homohypostrophene, (iii) samples containing 97% **2** and 3% **1**, and (iv) samples containing 90% **1** and 10% **2**. The changes in chemical shifts between (i)-(iv) are probably due to isotope effects.[78]

Figure 7. Isotopic distributions determined from the mass spectra of the tetracyclo[6.3.0.0^{4,11}.0^{5,9}]undecanes (homohypostrophanes) produced in the reductions by D_2 of homohypostrophene (bottom), a mixture of 97% **2** & 3% **1** (middle), and a mixture of 90% **1** & 10% **2** (top). The percent composition shown for each isotopomer is probably accurate to ± 5%.

Scheme I. Proposed Analogy Between Surface Alkyls Derived from Norbornene (left) and Those Derived from (Homohypostophene)neopentyl(*exo*-2-norbornyl)platinum(II) (right). HOP = Homohypostrophene; Np = Neopentyl.

Scheme II. Synthesis of Homohypostrophene.

Scheme III. Proposed Reactions of the Norbornyl* Moieties Generated in the Reduction by D_2 of a Mixture of 90% **1** and 10% **2**. Hydrogen atoms have been omitted for clarity.

Scheme IV. Proposed Reactions of the Norbornyl* Moieties Generated in the Reduction by D_2 of a Mixture of 97% **2** and 3% **1**. Hydrogen atoms have been omitted for clarity.

Captions for Figures and Schemes

Figure 1. ORTEP drawing (30% probability level, showing atomic labelling scheme) for (homohypostrophene)neopentyl(*exo*-2-norbornyl)platinum(II) **1**. The molecule crystallizes in a non-centrosymmetric space group; the absolute configuration (*R*) was determined crystallographically. The data for this structure were collected at 0 °C.

Figure 2. ORTEP drawing (30% probability level, showing atomic labelling scheme) for (homohypostrophene)neopentyl(*endo*-2-norbornyl)platinum(II) **2**. The molecule crystallizes in a centrosymmetric space group; the *S* enantiomer is shown. The data for this structure were collected at -58 °C.

Figure 3. ^{1}H NMR spectra (C_6D_6, 500 MHz) of (i) norbornane-d_0, and the norbornanes from the reduction by D_2 of samples containing (ii) 97% **2** and 3% **1**, and (iii) 90% **1** and 10% **2**. ^{2}H NMR spectra (C_6D_6, 46.03 MHz) of the norbornanes from the reductions by D_2 of samples containing (iv) 97% **2** and 3% **1**, and (v) 90% **1** and 10% **2**.

Figure 4. Isotopic distributions determined from the mass spectra of the norbornanes produced from the reductions by D_2 of norbornene (bottom), a mixture of 97% **2** and 3% **1** (middle), and a mixture of 90% **1** and 10% **2** (top). The percent composition shown for each isotopomer is probably accurate to ± 5%.

Figure 5. Mass spectra of neopentane-d_0 (bottom), and the neopentanes produced from the reductions by D_2 of a mixture of 97% **2** and 3% **1** (middle), and 90% **1** and 10% **2** (top).

Supplementary Material

for

The Reduction of C* Bonds Proceeds with Retention of Configuration: Stereochemical Investigation of the Heterogeneous Reduction by Dideuterium of (Homohypostrophene)neopentyl(2-norbornyl)platinum(II) Complexes on Platinum Black.

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Supplementary Material. We provide the following for **1** and **2**: procedures for the determination of structure, summary of the crystallographic data, atomic coordinates and equivalent isotropic displacement parameters for non-hydrogen atoms, complete tables of bond distances and angles, anisotropic displacement parameters for non-hydrogen atoms, coordinates for hydrogen atoms, observed and calculated structure factors, packing diagrams, and UV absorption spectra (44 pages).

X-ray Crystallography: General. We obtained the data using a Nicolet R3m/V four-circle diffractometer equipped with a LT-1 low-temperature device. Collection of the data was controlled with the Nicolet P3 program.[1] We checked the symmetry of the unit cells with the program XCELL, and processed the raw data with the program XDISK. We used the program XEMP to perform empirical absorption corrections, and solved the structures by use of the SHELXTL-PLUS[2] package of programs. Drawings were produced using the Nicolet program XP.

X-ray Crystallography for (Homohypostrophene)neopentyl-(*exo*norbornyl)platinum(II) *1*. We grew crystalline plates of **1** by low temperature crystallization from hexane/ethanol. The plates were transparent except for a cloudy line along one of the diagonals. We cut one of the plates (0.1 x 0.2 x 0 .3 mm) along the diagonal, and mounted it in air as follows. We attached the crystal to a 0.30 mm glass fiber with a minimum amount of epoxy glue. We glued the fiber to a 1/8" diameter brass pin using epoxy, and attached the pin to the goniometer head. We transferred the goniometer head to the diffractometer where the crystal was bathed by a cold nitrogen stream (0 (1) °C).

We used the data from a random search of reciprical space to index the unit cell. A lattice determination using both the P3 program and XCELL suggested a primitive orthorhombic cell. Examination of the axial photographs confirmed this assignment. We obtained the final unit cell parameters by performing a least squares refinement of 48 selected reflections, including four Friedel pairs, in the range $15° < 2\theta < 30°$.

We collected a total of 4005 reflections in the range $4° < 2\theta < 48°$ ($-h, -1, l$ to h, k, l). Of these, 3001 were unique reflections, and 1791 with $F_o > 6\sigma(F_o)$ were used in the structure solution. We measured the intensities of three check

reflections, (1, -2, -8), (-3, -3, 1) and (0, -5, 1), after every 60 reflections. These check reflections showed that the crystal did not decay during the 52 hours of exposure.

Systematic absences uniquely determined the space group to be $P2_12_12_1$; successful solution in this space group confirmed its choice. We located the platinum atom from direct methods, and used standard difference map techniques to find the remaining non-hydrogen atoms. After all of the non-hydrogen atoms were located and refined anisotropically, a difference map revealed approximately one-half of the hydrogen atom positions. The hydrogen atoms were placed in calculated positions (Uiso(H) = 1.2Uiso(C); dC-H = 0.96 Å) for refinement. We performed the refinement to convergence ($\Delta/\sigma(max) < 0.010$) with this model. The weighting scheme was $w = [\sigma^2(F) + gF^2]^{-1}$ ($g = 1.5 \times 10^{-4}$). The final difference map contained three peaks (1.61, 1.44 and 1.41 eÅ-3) located near the platinum atom. All other peaks were less than 0.67 eÅ-3.

The molecule crystallizes chirally, with one unique crystallographically independent molecule. We determined the correctness of the enantiomorph by inversion of configuration. With the program XEMP, we applied a semi-empirical absorption correction using scans near $\chi = 270$, and scans from 5 reflections in the range $14° < 2\theta < 31°$. Refinement yielded a structure with no non-positive definite atoms.

Table Ia. Summary of the crystallographic data for **1**

Color of crystal	yellow
Empirical formula	$C_{23}H_{34}Pt$
Crystal dimensions, mm	0.10 x 0.20 x 0.30
Space group	$P2_12_12_1$ (No. 19)
Cell dimensions	
a, Å	9.980 (4)
b, Å	11.419 (4)
c, Å	16.912 (3)
Temperature, °C	0 (1)
Wavelength, Å	0.71073
Z (molecules/cell)	4
Volume, Å^3	1927 (1)
d_{calcd}, g cm^{-3}	1.74
Linear absorption coefficient, cm^{-1}	73.6
Scan type	$\theta - 2\theta$
Scan speed, deg/min	3 - 30
Scan width, deg (+ dispersion)	2.0
Background / scan ratio	0.50
2θ range, deg	4 - 48
Data collected	$h, \pm k, l$
$F(000)$	1000
Parameters refined	217
Total number of reflections collected	4005
Number of unique reflections	3001
R_{int}	0.0524
number with $F_o > 6.00\sigma(F_o)$	1791
R(F)	0.0548
R_w(F)	0.0547
"Goodness of fit" for last cycle	1.099
Largest Δ/σ for last cycle	0.002
Final difference map (maximum)	1.61, 1.44 and 1.41 eÅ^{-3} near Pt
(remainder)	<0.67 eÅ^{-3}

[a] $R = \Sigma|(Fo-Fc)|/\Sigma Fo$

[b] $R_w = \Sigma(w^{1/2}|(Fo-Fc)|/\Sigma(w^{1/2}Fo)$, $w = [\sigma^2(F) + gF^2]^{-1}$

Table Ib. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for **1**[a]

atom	x	y	z	U(eq)
Pt	6775(1)	10325(1)	5380(1)	44(1)
C(1)	10982(20)	8135(23)	5535(15)	52(9)
C(2)	10388(22)	8914(25)	4924(16)	50(10)
C(3)	9802(22)	8365(30)	6093(14)	55(11)
C(4)	8899(20)	8428(21)	4789(13)	32(8)
C(5)	8484(19)	8043(25)	5600(13)	48(10)
C(6)	9563(21)	9672(29)	6170(13)	46(8)
C(7)	10016(19)	10074(22)	5316(17)	49(10)
C(8)	8770(20)	10481(23)	4801(12)	37(6)
C(9)	8148(23)	9511(18)	4475(11)	36(7)
C(10)	7403(24)	8759(24)	6044(13)	37(8)
C(11)	7997(22)	9668(32)	6342(13)	59(10)
C(12)	5145(25)	10811(41)	6107(15)	119(21)
C(13)	4513(35)	9969(43)	6689(22)	130(23)
C(14)	3150(43)	10521(58)	6832(30)	204(34)
C(15)	3392(34)	11517(45)	7320(20)	121(20)
C(16)	4950(61)	11544(47)	7432(20)	142(27)
C(17)	5551(47)	11806(36)	6736(20)	118(20)
C(18)	5088(38)	10313(51)	7428(22)	127(23)
C(19)	5923(22)	11407(23)	4532(17)	51(10)
C(20)	4785(28)	11055(27)	3964(17)	59(11)
C(21)	5196(30)	10022(23)	3435(14)	68(14)
C(22)	4496(37)	12091(31)	3433(17)	89(16)
C(23)	3457(24)	10758(31)	4368(17)	89(15)

[a] Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

Table Ic. Complete listing of bond lengths (Å) for **1**

Pt-C(8)	2.226 (20)	C(6)-C(11)	1.590 (30)
Pt-C(9)	2.256 (21)	C(7)-C(8)	1.589 (30)
Pt-C(10)	2.202 (25)	C(8)-C(9)	1.384 (32)
Pt-C(11)	2.167 (24)	C(10)-C(11)	1.297 (41)
Pt-C(12)	2.112 (27)	C(12)-C(13)	1.514 (55)
Pt-C(19)	2.075 (27)	C(12)-C(17)	1.609 (54)
C(1)-C(2)	1.487 (37)	C(13)-C(14)	1.519 (60)
C(1)-C(3)	1.533 (32)	C(13)-C(18)	1.430 (54)
C(2)-C(4)	1.602 (31)	C(14)-C(15)	1.426 (76)
C(2)-C(7)	1.527 (38)	C(15)-C(16)	1.566 (69)
C(3)-C(5)	1.601 (31)	C(16)-C(17)	1.355 (56)
C(3)-C(6)	1.517 (47)	C(16)-C(18)	1.412 (79)
C(4)-C(5)	1.500 (32)	C(19)-C(20)	1.542 (38)
C(4)-C(9)	1.541 (31)	C(20)-C(21)	1.536 (39)
C(5)-C(10)	1.548 (33)	C(20)-C(22)	1.513 (45)
C(6)-C(7)	1.581 (37)	C(20)-C(23)	1.529 (38)

Table Id. Complete listing of bond angles (°) for **1**

C(8)-Pt-C(9)	36.0(8)	C(3)-C(5)-C(10)	101.5(19)
C(8)-Pt-C(10)	92.0(9)	C(4)-C(5)-C(10)	118.8(21)
C(9)-Pt-C(10)	80.7(8)	C(3)-C(6)-C(7)	99.3(19)
C(8)-Pt-C(11)	81.7(8)	C(3)-C(6)-C(11)	99.6(23)
C(9)-Pt-C(11)	91.4(9)	C(7)-C(6)-C(11)	116.7(18)
C(10)-Pt-C(11)	34.5(11)	C(2)-C(7)-C(6)	102.4(21)
C(8)-Pt-C(12)	157.5(13)	C(2)-C(7)-C(8)	101.9(20)
C(9)-Pt-C(12)	166.1(12)	C(6)-C(7)-C(8)	111.2(16)
C(10)-Pt-C(12)	97.8(12)	Pt-C(8)-C(7)	115.8(14)
C(11)-Pt-C(12)	95.0(10)	Pt-C(8)-C(9)	73.2(13)
C(8)-Pt-C(19)	90.8(9)	C(7)-C(8)-C(9)	109.6(21)
C(9)-Pt-C(19)	91.4(9)	Pt-C(9)-C(4)	113.1(13)
C(10)-Pt-C(19)	162.2(10)	Pt-C(9)-C(8)	70.8(12)
C(11)-Pt-C(19)	162.8(11)	C(4)-C(9)-C(8)	106.6(18)
C(12)-Pt-C(19)	86.0(12)	Pt-C(10)-C(5)	112.4(15)
C(2)-C(1)-C(3)	91.1(18)	Pt-C(10)-C(11)	71.2(17)
C(1)-C(2)-C(4)	105.2(20)	C(5)-C(10)-C(11)	107.0(21)
C(1)-C(2)-C(7)	108.3(22)	Pt-C(11)-C(6)	114.5(16)
C(4)-C(2)-C(7)	97.9(17)	Pt-C(11)-C(10)	74.2(15)
C(1)-C(3)-C(5)	105.7(18)	C(6)-C(11)-C(10)	112.4(25)
C(1)-C(3)-C(6)	110.0(22)	Pt-C(12)-C(13)	122.2(30)
C(5)-C(3)-C(6)	98.1(19)	Pt-C(12)-C(17)	112.1(22)
C(2)-C(4)-C(5)	103.2(18)	C(13)-C(12)-C(17)	97.1(26)
C(2)-C(4)-C(9)	102.8(18)	C(12)-C(13)-C(14)	102.3(37)
C(5)-C(4)-C(9)	114.6(18)	C(12)-C(13)-C(18)	103.1(35)
C(3)-C(5)-C(4)	100.4(17)	C(14)-C(13)-C(18)	96.1(35)
C(13)-C(14)-C(15)	105.7(36)	Pt-C(19)-C(20)	125.3(19)
C(14)-C(15)-C(16)	104.7(35)	C(19)-C(20)-C(21)	111.5(22)
C(15)-C(16)-C(17)	109.8(36)	C(19)-C(20)-C(22)	107.8(25)
C(15)-C(16)-C(18)	94.4(41)	C(21)-C(20)-C(22)	107.8(23)
C(17)-C(16)-C(18)	99.9(38)	C(19)-C(20)-C(23)	114.7(23)
C(12)-C(17)-C(16)	107.9(37)	C(21)-C(20)-C(23)	108.7(25)
C(13)-C(18)-C(16)	103.7(38)	C(22)-C(20)-C(23)	105.9(25)

Table Ie. Anisotropic displacement parameters ($Å^2 \times 10^3$) for **1**[a]

atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Pt	26(1)	75(1)	30(1)	8(1)	0(1)	-5(1)
C(1)	25(11)	63(13)	69(19)	15(12)	23(13)	-22(15)
C(2)	19(11)	64(21)	67(18)	1(14)	15(12)	-26(17)
C(3)	27(13)	113(29)	24(13)	38(16)	-6(12)	-5(15)
C(4)	25(10)	28(15)	42(15)	12(10)	8(11)	-8(12)
C(5)	18(13)	72(21)	55(16)	-4(11)	0(11)	7(13)
C(6)	39(12)	53(18)	46(13)	3(17)	-16(11)	-10(18)
C(7)	22(10)	48(24)	78(17)	2(10)	14(14)	5(18)
C(8)	36(10)	41(9)	34(13)	-3(12)	14(9)	-15(13)
C(9)	45(11)	26(13)	37(11)	20(16)	-3(13)	-20(12)
C(10)	46(13)	33(17)	32(12)	-1(13)	3(12)	-3(13)
C(11)	38(14)	101(24)	38(12)	23(20)	4(12)	-17(17)
C(12)	32(15)	297(61)	28(14)	43(24)	-19(14)	-45(25)
C(13)	92(25)	177(57)	122(31)	-22(30)	95(26)	-30(33)
C(14)	80(27)	387(91)	144(37)	-3(55)	28(31)	-106(52)
C(15)	51(22)	236(53)	75(22)	83(31)	-13(20)	-39(28)
C(16)	249(65)	128(43)	49(22)	-85(46)	40(31)	-62(26)
C(17)	154(38)	130(40)	70(25)	-19(32)	33(25)	-47(26)
C(18)	96(28)	200(56)	85(26)	-19(36)	-31(22)	-69(35)
C(19)	41(13)	43(18)	70(18)	18(12)	14(16)	36(17)
C(20)	61(18)	50(21)	67(19)	0(17)	-11(17)	3(19)
C(21)	100(22)	62(34)	41(13)	18(18)	-35(15)	-20(15)
C(22)	106(28)	100(32)	61(20)	19(24)	-32(21)	8(21)
C(23)	35(17)	143(35)	88(21)	-19(17)	24(15)	-32(20)

[a] The anisotropic displacement exponent takes the form: $-2\pi^2(h^2a^{*2}U_{11} + ... + 2hka^*b^*U_{12})$.

Table If. Coordinates of hydrogen atoms (x 10^4) for **1**

atom	x	y	z
H(1A)	11827	8407	5734
H(1B)	11042	7333	5368
H(2A)	10916	8993	4452
H(3A)	9875	7968	6592
H(4A)	8893	7796	4415
H(5A)	8295	7219	5620
H(6A)	10082	10015	6588
H(7A)	10737	10630	5329
H(8A)	8907	11141	4458
H(9A)	7882	9523	3929
H(10A)	6743	8355	6354
H(11A)	7732	9878	6869
H(12A)	4463	11115	5764
H(13A)	4424	9163	6539
H(14A)	2752	10784	6347
H(14B)	2506	10046	7106
H(15A)	3171	12238	7058
H(15B)	2942	11491	7821
H(16A)	5287	11929	7896
H(17A)	6506	11865	6795
H(17B)	5199	12550	6574
H(18A)	4551	10015	7854
H(18B)	5993	10039	7485
H(19A)	5628	12088	4816
H(19B)	6653	11660	4204
H(21A)	6016	10204	3164
H(21B)	4508	9853	3055
H(21C)	5331	9352	3769
H(22A)	4225	12770	3728
H(22B)	3801	11881	3067
H(22C)	5304	12262	3147
H(23A)	3151	11392	4693
H(23B)	3612	10080	4691
H(23C)	2790	10580	3977

H	K	L	10FO	10FC	10S	H	K	L	10FO	10FC	10S	H	K	L	10FO	10FC	10S	H	K	L	10FO	10FC	10S	H	K	L	10FO	10FC	10S
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4	0	0	259	240	37	8	5	0	455	419	29	1	12	0	213	130	-72	6	2	1	630	637	23	-4	5	1	417	393	26
6	0	0	2121	2125	27	9	5	0	116	96	-85	2	12	0	326	231	-70	7	2	1	547	529	30	-3	5	1	793	797	48
8	0	0	1181	1157	24	10	5	0	684	691	24	3	12	0	104	180	-74	8	2	1	695	705	22	-2	5	1	802	855	59
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2	1	0	402	399	14	2	6	0	103	47	-70	1	0	1	472	440	8	11	2	1	164	211	-83	1	5	1	382	408	19
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5	4	0	1387	1354	52	2	10	0	324	309	-65	-7	2	1	523	534	43	4	4	1	1271	1180	28	-2	7	1	750	736	23
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-2	11	1	189	166	-92	-11	2	2	329	318	-66	0	4	2	906	972	25	-6	7	2	372	416	51	0	10	2	353	311	46

H	K	L	10FO	10FC	10S	H	K	L	10FO	10FC	10S	H	K	L	10FO	10FC	10S	H	K	L	10FO	10FC	10S	H	K	L	10FO	10FC	10S
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2	10	2	356	301	-62	6	1	3	448	456	34	-5	4	3	770	770	24	10	6	3	240	227	-145	-4	10	3	144	270	-144
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3	7	15	101	111	-101	0	3	16	423	394	49	3	0	17	846	824	27	4	4	17	266	259	-135	0	4	18	226	182	-102
-1	8	15	477	449	47	1	3	16	496	509	28	4	0	17	509	558	42	-3	5	17	296	209	-165	1	4	18	392	396	-66
0	8	15	111	221	-79	2	3	16	424	441	45	5	0	17	499	527	49	-1	5	17	456	512	39	2	4	18	377	318	59
1	8	15	504	435	74	3	3	16	257	221	-77	-5	1	17	401	360	55	0	5	17	443	429	52	1	0	19	324	312	52
0	0	16	778	799	25	4	3	16	399	408	58	-4	1	17	699	696	34	1	5	17	494	519	34	2	0	19	152	58	-152
1	0	16	580	601	22	5	3	16	381	415	-78	-3	1	17	561	518	38	2	5	17	280	342	-83	-2	1	19	587	568	41
2	0	16	480	465	37	-5	4	16	600	437	81	-2	1	17	595	573	35	3	5	17	231	220	-129	.	1	19	223	151	-90
3	0	16	89	89	-89	-4	4	16	273	294	-204	-1	1	17	265	251	-107	-1	6	17	309	345	-75	0	1	19	117	93	-88
4	0	16	236	151	-92	-3	4	16	576	467	54	0	1	17	92	150	-65	0	6	17	572	562	34	1	1	19	250	135	-86
5	0	16	334	324	-64	-2	4	16	395	270	-71	1	1	17	164	255	-164	1	6	17	455	377	-105	2	1	19	617	575	37
6	0	16	445	543	71	-1	4	16	398	376	42	2	1	17	622	577	32	0	0	18	95	209	-95	-1	2	19	325	289	-67
-6	1	16	432	483	62	0	4	16	444	433	33	3	1	17	503	524	39	1	0	18	723	729	47	0	2	19	262	290	-71
-5	1	16	432	460	49	1	4	16	425	389	43	4	1	17	678	693	37	2	0	18	98	288	-98	1	2	19	188	299	-188
-3	1	16	201	172	-118																								

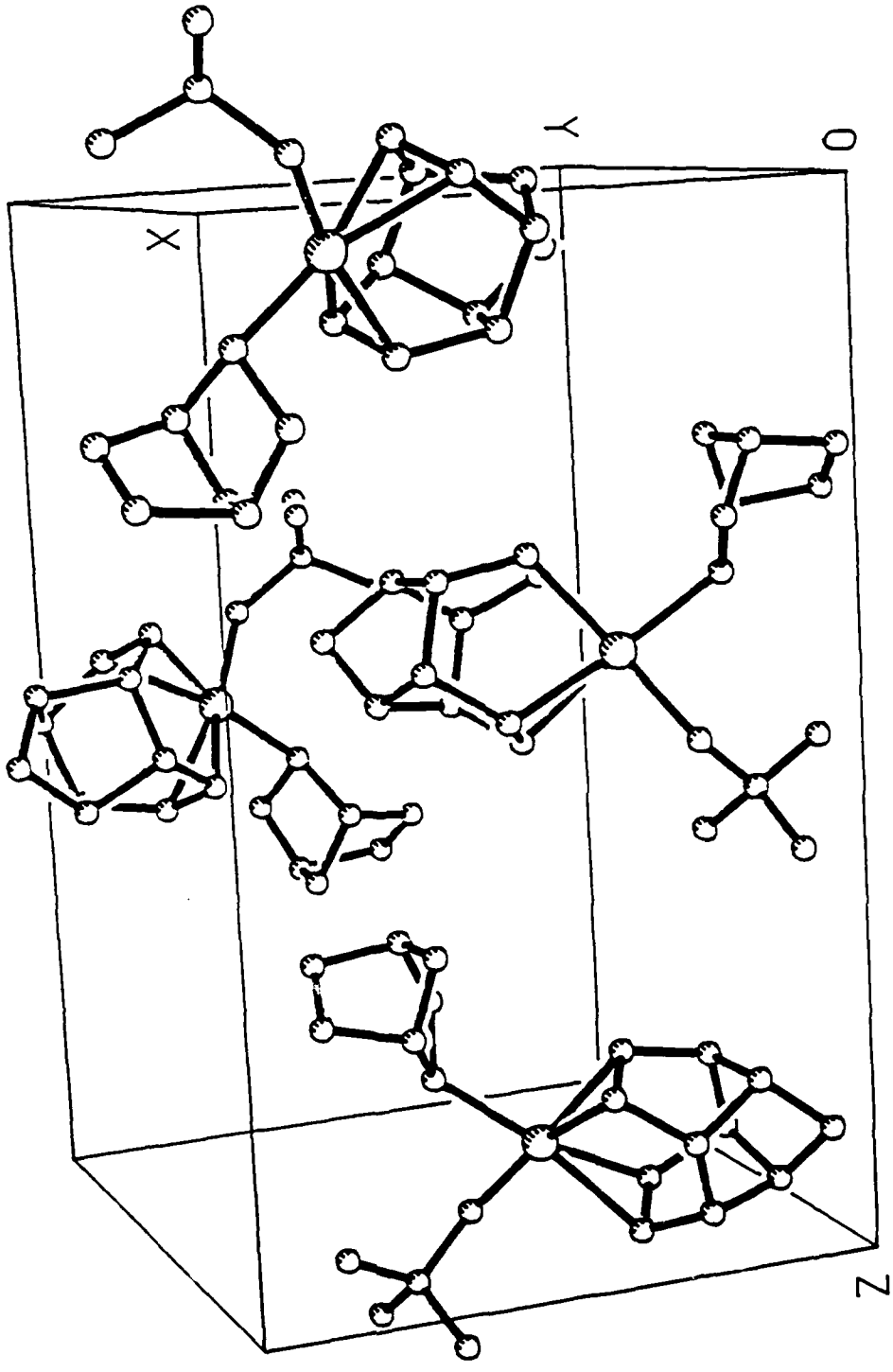

Figure 1a. Packing diagram for **1**.

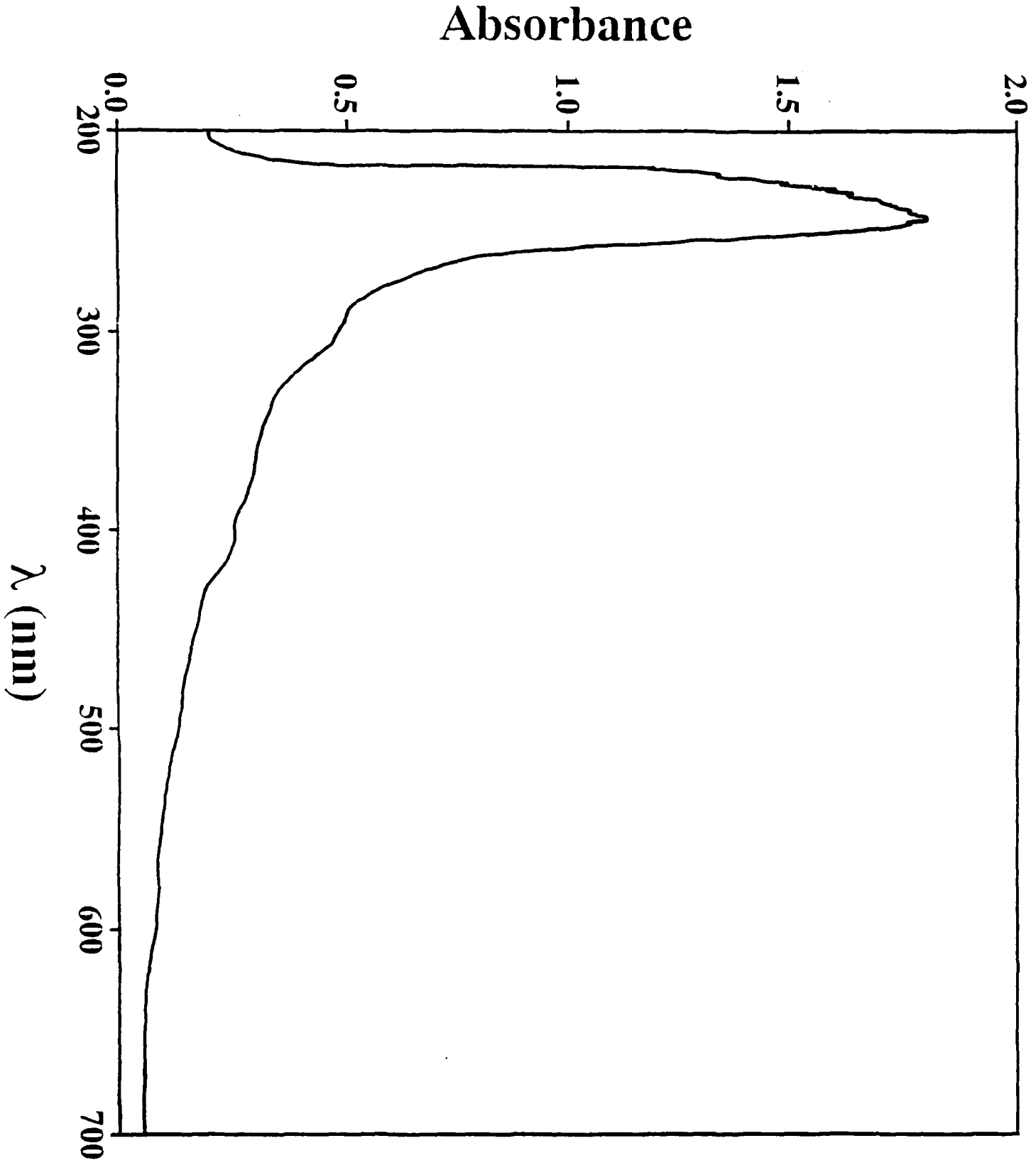

Figure 1b. The UV absorption spectrum of a mixture of 90% **1** and 10% **2** in *n*-hexane.

X-ray Crystallography for (Homohypostrophene)neopentyl-(*endo*norbornyl)platinum(II) *2*. We grew x-ray quality crystals of **2** by low temperature recrystallization from diethyl ether/methanol, and mounted them in air as follows. We attached a crystal of dimensions 0.10 x 0.10 x 0.30 mm to a 0.30 mm glass fiber with a minimum amount of silicon grease. We glued the fiber to a 1/8" diameter brass pin using epoxy, and attached the pin to the goniometer head. We transferred the goniometer head to the diffractometer where the crystal was bathed by a cold nitrogen stream (-58 (1) °C).

We used the data from a random search of reciprical space to index the unit cell. A lattice determination using both the P3 program and XCELL suggested a primitive monoclinic cell. Examination of the axial photographs confirmed this assignment. We obtained the final unit cell parameters by performing a least squares refinement of 47 selected reflections, including ten Friedel pairs, in the range $15° < 2\theta < 30°$.

We collected a total of 3641 reflections in the range $4° < 2\theta < 48°$ (*-h*, -1, *l* to *h*, *k*, *l*). Of these, 2888 were unique reflections, and 1951 with $F_O > 6\sigma(F_O)$ were used in the structure solution. We measured the intensities of three check reflections, (-2, 8, 3), (0, 2, 6) and (6, -1, -6), after every 60 reflections. These check reflections showed that the crystal did not decay during the 59 hours of exposure.

Systematic absences uniquely determined the space group to be $P2_1/n$. Successful solution in this space group confirmed its choice. We located the platinum atom from a Patterson synthesis, and used standard difference map techniques to find the remaining non-hydrogen atoms. After all of the non-hydrogen atoms were located and refined anisotropically, a difference map

revealed approximately two-thirds of the hydrogen atom positions. The hydrogen atoms were placed in calculated positions ($U_{iso}(H) = 1.2U_{iso}(C)$; $d_{C-H} = 0.96$ Å) for refinement. We performed the refinement to convergence ($\Delta/\sigma(max) < 0.001$) with this model. The weighting scheme was $w = [\sigma^2(F) + gF^2]^{-1}$ ($g = 3.4 \times 10^{-4}$). The final difference map contained two peaks (1.55 and 1.28 eÅ^{-3}) located near the platinum atom. All other peaks were less than 0.88 eÅ^{-3}.

The molecule crystallizes as a racemate, with one unique crystallographically independent molecule. We attempted a number of semi-empirical absorption corrections. Corrections employing only scans near $\chi = 270$, using either ABSCOR (public domain) or XEMP (Siemens), yielded one non-positive definite thermal ellipse. In the end, we used XEMP to apply an absorption correction which was based on scans from 6 reflections in the range $12° < 2\theta < 36°$ and equivalent data (intensity > 25σ). Refinement yielded a structure with no non-positive definite atoms.

Table IIa. Summary of the crystallographic data for **2**

Color of crystal	yellow
Empirical formula	$C_{23}H_{34}Pt$
Crystal dimensions, mm	0.10 x 0.10 x 0.30
Space group	$P2_1/n$ (No. 8)
Cell dimensions	
a, Å	10.679 (5)
b, Å	17.085 (7)
c, Å	11.514 (4)
β, deg	116.77 (3)
Temperature, °C	-58 (1)
Wavelength, Å	0.71073
Z (molecules/cell)	4
Volume, Å^3	1875.4 (14)
d_{calcd}, g cm^{-3}	1.79
Linear absorption coefficient, cm^{-1}	75.5
Scan type	$\theta - 2\theta$
Scan speed, deg/min	3 - 30
Scan width, deg (+ dispersion)	1.60
Background / scan ratio	0.50
2θ range, deg	4 - 48
Data collected	$h, k, \pm l$
$F(000)$	1000
Parameters refined	217
Total number of reflections collected	3641
Number of unique reflections	2888
R_{int}	0.0370
Number with $F_o > 6.00\sigma(F_o)$	1951
$R(F)$	0.0415
$R_w(F)$	0.0394
"Goodness of fit" for last cycle	1.50
Largest Δ/σ for last cycle	0.001
Final difference map (maximum)	1.55 and 1.28 eÅ^{-3} near Pt
(remainder)	<0.88 eÅ^{-3}

[a] $R = \Sigma|(Fo-Fc)|/\Sigma Fo$

[b] $R_w = \Sigma(w^{1/2}|(Fo-Fc)|/\Sigma(w^{1/2}Fo)$, $w = [\sigma^2(F) + gF^2]^{-1}$

Table IIb. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for 2[a]

atom	x	y	z	U(eq)
Pt	6333(1)	2112(1)	1750(1)	20(1)
C(1)	10599(15)	2240(8)	1269(12)	30(6)
C(2)	9440(13)	1632(8)	874(11)	25(5)
C(3)	9933(14)	2770(7)	1853(11)	23(5)
C(4)	9261(14)	1456(8)	2123(11)	23(5)
C(5)	8033(14)	2068(8)	126(11)	25(5)
C(6)	8418(14)	2899(9)	826(10)	26(5)
C(7)	9658(13)	2280(7)	2830(10)	21(5)
C(8)	7779(14)	1209(8)	1580(11)	24(6)
C(9)	7022(14)	1583(8)	375(11)	27(6)
C(10)	7667(13)	3086(7)	1666(10)	17(5)
C(11)	8389(14)	2711(8)	2815(12)	28(6)
C(12)	5133(14)	2860(9)	2296(10)	28(5)
C(13)	3778(15)	3207(8)	1166(12)	27(6)
C(14)	4190(17)	3776(9)	347(11)	39(6)
C(15)	4713(18)	4512(9)	1201(12)	39(7)
C(16)	4649(17)	4244(9)	2485(12)	40(7)
C(17)	5771(15)	3607(9)	3100(11)	33(6)
C(18)	3297(15)	3768(9)	1899(12)	34(6)
C(19)	4678(14)	1316(9)	1224(11)	29(6)
C(20)	4591(15)	685(8)	2167(12)	29(6)
C(21)	5723(15)	64(9)	2546(13)	37(6)
C(22)	3155(16)	277(10)	1490(14)	46(7)
C(23)	4709(19)	1045(9)	3430(12)	45(8)

[a] Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

Table IIc. Complete listing of bond lengths (Å) for **2**

Pt-C(8)	2.253 (15)	C(6)-C(10)	1.543 (22)
Pt-C(9)	2.217 (16)	C(7)-C(11)	1.536 (21)
Pt-C(10)	2.220 (13)	C(8)-C(9)	1.405 (16)
Pt-C(11)	2.224 (13)	C(10)-C(11)	1.354 (16)
Pt-C(12)	2.096 (16)	C(12)-C(13)	1.563 (16)
Pt-C(19)	2.092 (15)	C(12)-C(17)	1.544 (19)
C(1)-C(2)	1.520 (20)	C(13)-C(14)	1.550 (23)
C(1)-C(3)	1.486 (22)	C(13)-C(18)	1.512 (23)
C(2)-C(4)	1.563 (21)	C(14)-C(15)	1.539 (19)
C(2)-C(5)	1.546 (18)	C(15)-C(16)	1.579 (23)
C(3)-C(6)	1.530 (16)	C(16)-C(17)	1.535 (21)
C(3)-C(7)	1.532 (20)	C(16)-C(18)	1.524 (22)
C(4)-C(7)	1.585 (17)	C(19)-C(20)	1.563 (21)
C(4)-C(8)	1.478 (19)	C(20)-C(21)	1.516 (21)
C(5)-C(6)	1.593 (19)	C(20)-C(22)	1.539 (20)
C(5)-C(9)	1.488 (23)	C(20)-C(23)	1.531 (21)

Table IId. Complete listing of bond angles (°) for **2**

C(8)-Pt-C(9)	36.6(4)	C(2)-C(5)-C(9)	103.4(11)
C(8)-Pt-C(10)	91.8(5)	C(6)-C(5)-C(9)	116.3(13)
C(9)-Pt-C(10)	81.3(5)	C(3)-C(6)-C(5)	101.8(10)
C(8)-Pt-C(11)	79.5(5)	C(3)-C(6)-C(10)	102.3(9)
C(9)-Pt-C(11)	91.1(5)	C(5)-C(6)-C(10)	114.4(12)
C(10)-Pt-C(11)	35.5(4)	C(3)-C(7)-C(4)	101.8(10)
C(8)-Pt-C(12)	168.2(5)	C(3)-C(7)-C(11)	100.9(10)
C(9)-Pt-C(12)	155.1(4)	C(4)-C(7)-C(11)	112.9(10)
C(10)-Pt-C(12)	92.7(6)	Pt-C(8)-C(4)	114.4(9)
C(11)-Pt-C(12)	98.0(5)	Pt-C(8)-C(9)	70.3(9)
C(8)-Pt-C(19)	93.0(6)	C(4)-C(8)-C(9)	108.6(13)
C(9)-Pt-C(19)	92.1(6)	Pt-C(9)-C(5)	114.4(9)
C(10)-Pt-C(19)	161.7(4)	Pt-C(9)-C(8)	73.1(9)
C(11)-Pt-C(19)	162.6(5)	C(5)-C(9)-C(8)	107.4(11)
C(12)-Pt-C(19)	86.1(6)	Pt-C(10)-C(6)	113.4(8)
C(2)-C(1)-C(3)	93.5(12)	Pt-C(10)-C(11)	72.4(8)
C(1)-C(2)-C(4)	106.0(10)	C(6)-C(10)-C(11)	106.6(12)
C(1)-C(2)-C(5)	107.1(11)	Pt-C(11)-C(7)	115.6(8)
C(4)-C(2)-C(5)	96.5(12)	Pt-C(11)-C(10)	72.1(7)
C(1)-C(3)-C(6)	107.0(10)	C(7)-C(11)-C(10)	110.4(13)
C(1)-C(3)-C(7)	106.8(11)	Pt-C(12)-C(13)	116.3(9)
C(6)-C(3)-C(7)	99.2(11)	Pt-C(12)-C(17)	121.3(11)
C(2)-C(4)-C(7)	100.7(11)	C(13)-C(12)-C(17)	100.3(11)
C(2)-C(4)-C(8)	102.5(9)	C(12)-C(13)-C(14)	109.5(13)
C(7)-C(4)-C(8)	117.3(12)	C(12)-C(13)-C(18)	101.3(10)
C(2)-C(5)-C(6)	100.5(9)	C(14)-C(13)-C(18)	101.7(12)
C(13)-C(14)-C(15)	104.2(12)	Pt-C(19)-C(20)	123.7(7)
C(14)-C(15)-C(16)	101.8(12)	C(19)-C(20)-C(21)	113.2(14)
C(15)-C(16)-C(17)	106.5(14)	C(19)-C(20)-C(22)	108.5(10)
C(15)-C(16)-C(18)	99.9(10)	C(21)-C(20)-C(22)	108.2(12)
C(17)-C(16)-C(18)	102.4(12)	C(19)-C(20)-C(23)	112.2(12)
C(12)-C(17)-C(16)	105.5(10)	C(21)-C(20)-C(23)	107.1(10)
C(13)-C(18)-C(16)	95.3(13)	C(22)-C(20)-C(23)	107.4(15)

Table IIe. Anisotropic displacement parameters ($Å^2 \times 10^3$) for 2[a]

atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Pt	20(1)	20(1)	19(1)	0(1)	9(1)	-2(1)
C(1)	33(8)	27(8)	37(6)	8(6)	22(6)	8(7)
C(2)	16(7)	31(8)	33(6)	-7(6)	15(6)	-2(6)
C(3)	24(7)	20(8)	28(6)	-3(5)	13(6)	-6(6)
C(4)	24(8)	14(7)	28(6)	6(5)	10(6)	14(6)
C(5)	30(8)	22(7)	23(6)	2(6)	12(6)	-3(7)
C(6)	34(8)	20(7)	29(6)	4(6)	20(6)	-2(8)
C(7)	14(7)	23(8)	21(5)	-2(5)	3(5)	2(6)
C(8)	33(9)	11(7)	34(7)	-11(5)	21(7)	1(6)
C(9)	29(8)	26(8)	27(6)	-9(5)	12(6)	-2(7)
C(10)	13(7)	8(8)	29(6)	-2(5)	8(6)	-2(5)
C(11)	26(8)	33(9)	27(6)	-12(5)	13(6)	-14(6)
C(12)	39(8)	29(8)	22(6)	8(6)	20(6)	-4(8)
C(13)	21(8)	23(7)	36(7)	-12(6)	12(6)	-7(6)
C(14)	54(11)	33(8)	24(6)	0(6)	14(7)	18(9)
C(15)	55(11)	26(8)	39(7)	16(6)	24(8)	10(8)
C(16)	61(12)	29(9)	34(7)	3(6)	25(8)	6(8)
C(17)	36(9)	38(9)	27(6)	-1(6)	15(6)	2(8)
C(18)	31(9)	32(9)	40(7)	1(7)	17(7)	2(8)
C(19)	26(8)	42(9)	27(6)	18(6)	20(6)	18(8)
C(20)	21(8)	26(8)	31(7)	3(6)	4(6)	-6(7)
C(21)	27(9)	35(9)	45(8)	4(7)	13(7)	-6(7)
C(22)	35(10)	50(11)	57(9)	-3(8)	25(8)	-17(9)
C(23)	69(13)	34(9)	38(8)	11(6)	29(8)	4(9)

[a] The anisotropic displacement exponent takes the form: $-2\pi^2(h^2a^{*2}U_{11} + ... + 2hka^*b^*U_{12})$.

Table IIf. Coordinates of hydrogen atoms (x 10^4) for **2**

atom	x	y	z
H(1A)	10648	2464	526
H(1B)	11504	2056	1890
H(2A)	9560	1177	445
H(3A)	10440	3246	2203
H(4A)	9876	1046	2637
H(5A)	7786	2103	-784
H(6A)	8332	3321	244
H(7A)	10440	2264	3681
H(8A)	7583	667	1642
H(9A)	6349	1280	-334
H(10A)	7369	3613	1694
H(11A)	8512	2993	3581
H(12A)	4833	2541	2811
H(13A)	3088	2820	677
H(14A)	3399	3911	-459
H(14B)	4908	3549	169
H(15A)	4152	4967	811
H(15B)	5669	4623	1397
H(16A)	4672	4661	3052
H(17A)	6630	3747	3075
H(17B)	5963	3521	3989
H(18A)	3122	3517	2558
H(18B)	2490	4067	1334
H(19A)	4624	1031	484
H(19B)	3829	1616	931
H(21A)	6642	290	2984
H(21B)	5592	-303	3114
H(21C)	5628	-202	1776
H(22A)	3074	-111	2054
H(22B)	2425	659	1275
H(22C)	3070	32	708
H(23A)	5595	1305	3892
H(23B)	3963	1419	3191
H(23C)	4610	650	3977

H	K	L	10FO	10FC	10S	H	K	L	10FO	10FC	10S	H	K	L	10FO	10FC	10S	H	K	L	10FO	10FC	10S	H	K	L	10FO	10FC	10S
2	0	0	873	899	14	7	6	0	682	-626	33	5	12	0	232	144	-81	-8	1	1	373	175	50	9	3	1	805	-781	31
4	0	0	1687	-1724	19	8	6	0	212	-234	-111	6	12	0	493	-531	43	-7	1	1	1796	1705	19	10	3	1	695	698	36
6	0	0	764	692	23	9	6	0	1193	1156	28	7	12	0	107	129	-214	-6	1	1	554	-497	23	-11	4	1	115	107	-230
8	0	0	1033	1052	29	10	6	0	110	95	-220	8	12	0	1012	-936	28	-5	1	1	296	-177	37	-10	4	1	958	-847	33
10	0	0	743	-664	39	1	7	0	1559	1506	16	1	13	0	234	38	-82	-4	1	1	377	-342	26	-9	4	1	985	-900	32
1	1	0	269	-224	11	2	7	0	252	-274	-47	3	13	0	95	155	-188	-2	1	1	194	227	22	-8	4	1	882	653	32
2	1	0	1522	-1544	12	3	7	0	1993	-1950	20	4	13	0	248	-201	-72	0	1	1	460	428	7	-7	4	1	230	-218	-99
3	1	0	584	604	14	4	7	0	200	93	-62	5	13	0	97	-55	-193	1	1	1	3036	2937	8	-6	4	1	1154	1072	21
4	1	0	376	370	20	5	7	0	1105	-1084	21	6	13	0	1235	-1271	25	2	1	1	890	-852	12	-5	4	1	1362	1282	20
5	1	0	235	99	37	6	7	0	93	119	-184	7	13	0	206	-53	-109	3	1	1	1533	-1455	14	-4	4	1	1344	-1333	19
6	1	0	2021	1993	17	7	7	0	1397	1338	27	8	13	0	321	375	-75	4	1	1	159	6	-50	-3	4	1	571	-562	19
7	1	0	347	-404	40	8	7	0	377	-158	63	1	14	0	380	-351	52	5	1	1	1733	-1673	15	-1	4	1	1534	-1342	27
8	1	0	447	-379	39	9	7	0	256	313	-96	2	14	0	306	64	-65	6	1	1	552	540	20	0	4	1	1555	1508	18
9	1	0	192	-63	-103	10	7	0	242	-88	-101	3	14	0	94	-191	-188	7	1	1	1109	1078	19	1	4	1	708	715	20
10	1	0	867	-824	24	0	8	0	478	-498	30	4	14	0	1209	-1186	21	8	1	1	185	-85	-98	2	4	1	1233	1209	19
1	2	0	1495	1546	13	1	8	0	1346	-1365	30	5	14	0	373	462	56	9	1	1	867	812	21	3	4	1	1334	1255	17
3	2	0	612	671	19	2	8	0	395	258	32	6	14	0	275	352	-82	10	1	1	189	-213	-100	4	4	1	1950	-1885	19
4	2	0	1981	1964	21	3	8	0	816	-841	19	7	14	0	116	89	-234	-11	2	1	303	-178	-84	5	4	1	574	-612	25
5	2	0	805	-864	19	4	8	0	512	551	29	1	15	0	490	-466	33	-10	2	1	491	486	55	6	4	1	457	-386	36
6	2	0	726	-677	23	5	8	0	1440	1459	23	2	15	0	1308	-1243	21	-9	2	1	1446	1411	29	7	4	1	371	-467	-63
7	2	0	439	-323	44	6	8	0	269	-269	-69	3	15	0	425	365	44	-8	2	1	447	-350	58	8	4	1	919	939	32
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-3	9	11	81	123	-162	-1	1	12	359	-310	25	-8	4	12	542	508	32	-4	6	12	81	91	-162	-4	2	13	514	493	31
-2	9	11	461	-451	33	-9	2	12	313	-273	-57	-7	4	12	747	-744	21	-3	6	12	1017	-981	19	-6	3	13	692	701	22
-6	10	11	454	-485	36	-8	2	12	902	-886	22	-6	4	12	186	-261	-67	-2	6	12	85	-23	-168	-5	3	13	346	409	43
-5	10	11	179	58	-69	-7	2	12	315	335	45	-5	4	12	444	-422	30												

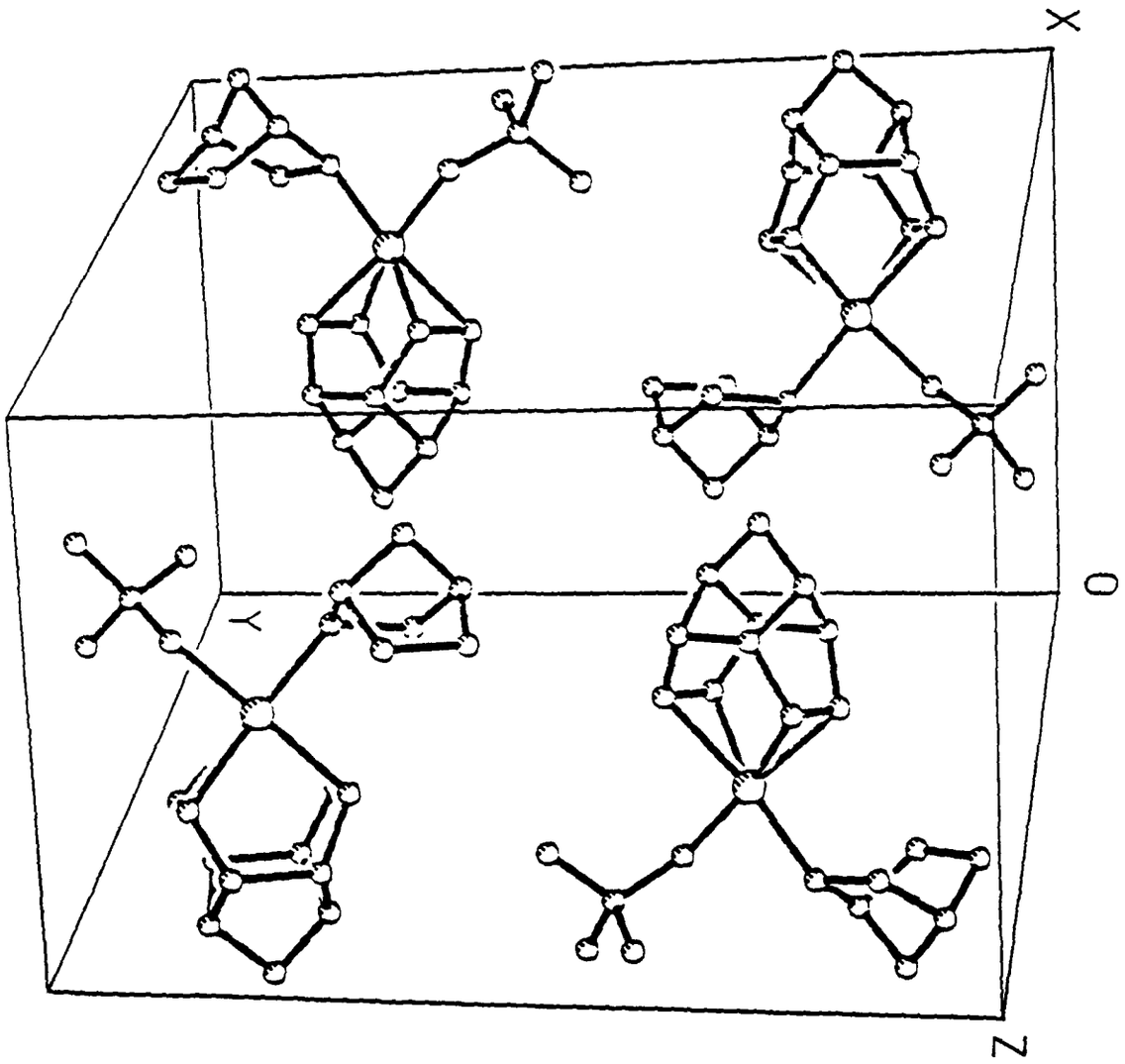

Figure 2a. Packing diagram for **2**.

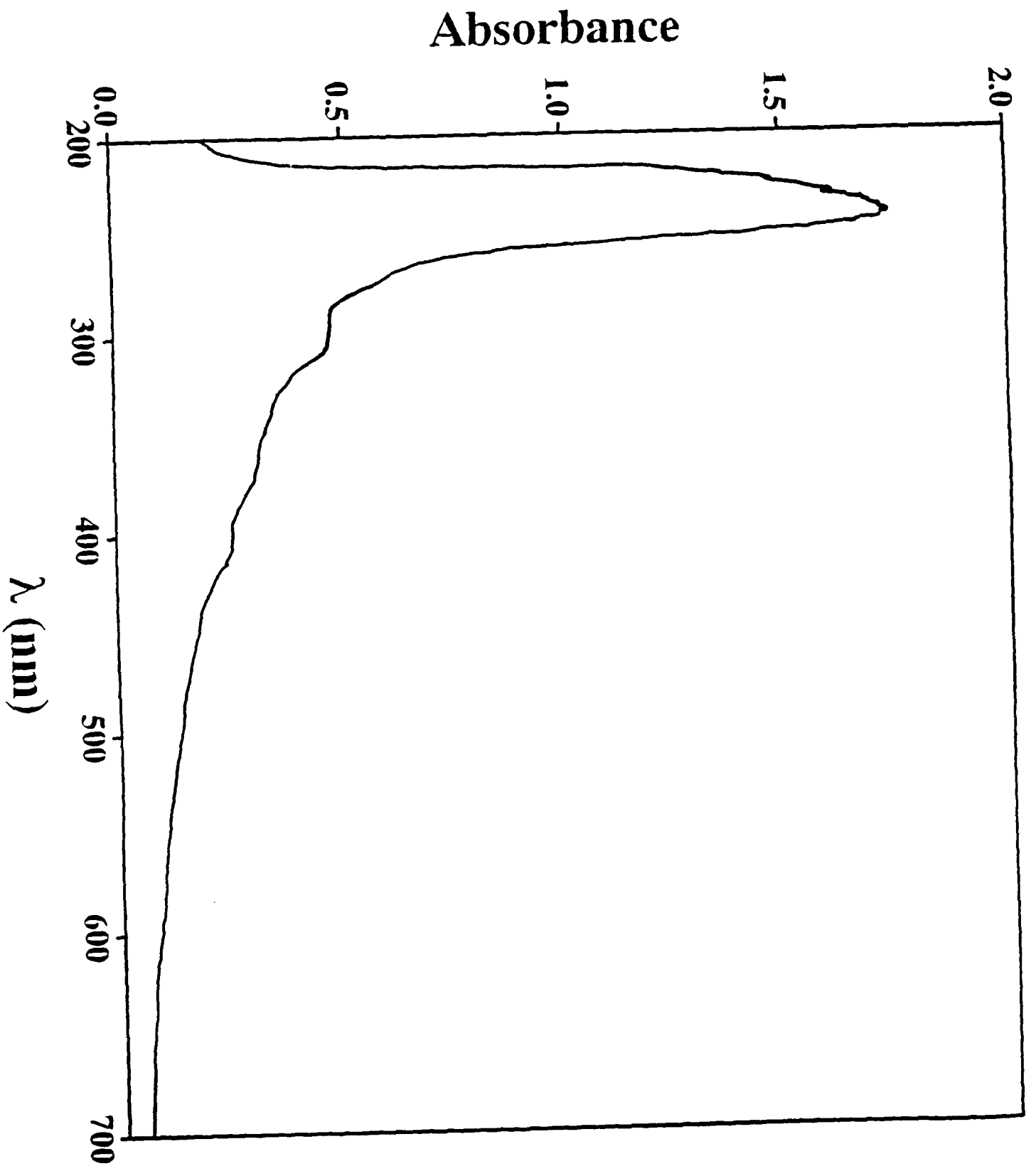

Figure 2b. The UV absorption spectrum of a mixture of 98% 2 and 2% 1 in *n*-hexane.

References.

(1) "P3/R3 Data Collection Manual" Nicolet Instrument Corp.: Madison, Wisconsin (1987).

(2) "SHELXTL - PLUS Users Manual" Nicolet Instrument Corp.: Madison, Wisconsin (1988).