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Powdered MQ Resin−Platinum Complexes and Their Use as Silicone-Soluble Hydrosilylation Cure Catalysts

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Moldable poly(dimethylsiloxane) formulations are described which use so-called MQ resins as reinforcing fillers. These formulations employ platinum-catalyzed hydrosilylation as the cure reaction. The platinum catalysts were prepared by spray-drying solutions composed of vinyl-containing MQ resins with platinum(0) complexes of divinyltetramethydisiloxane ($M^{vi}M^{vi}$) ligands. The spray-dried mixture was a white, free-flowing powder containing 1% by weight platinum where the platinum was Pt(0) and coordinated to the vinyls of the MQ resin. Molecular MQ resin−platinum complexes, free of platinum crystallites were obtained by spray-drying but not by vacuum oven treatment of solutions of resin and $Pt(M^{vi}M^{vi})_x$ complexes. Oven-prepared, platinum-containing resins contained platinum crystallites. The platinum-containing spray-dried MQ resin was a useful catalyst to cure moldable poly(dimethylsiloxane) formulations. Higher catalytic activity was observed for resin without platinum crystallites. Analyses of the platinum-containing MQ resins included ^{195}Pt NMR and TEM.

Introduction

One important application of poly(dimethylsiloxane) (PDMS) materials is liquid injection molding (LIM) products. A typical LIM system employs platinum-catalyzed hydrosilyation reactions to rapidly cure liquid precursors into a silicone elastomer with good tensile properties. The uncured liquid components contain two principle types of siloxane polymers and a filler. One of the PDMS polymers has pendent Si−vinyl functionality, while the other polymer contains Si−H functional groups. A filler, frequently a silicone resin, is added to improve the physical properties of the cured silicone elastomer. Platinum catalysts and hydrosilylation inhibitors are used to catalyze the curing reaction at a controlled rate.

A useful shorthand nomenclature for describing the polymers and fillers is the M, D, T, Q nomenclature.[1] An example of a LIM formulation using this shorthand is a combination of

$M^{vi}D_xM^{vi}$: a Si−vinyl endcapped PDMS

$MD^{H}_{x}D_{y}M$: a $SiMe_3$ endcapped dimethyl, methylhydrogen siloxane copolymer

MQ resin: a reinforcing filler

MQ resin is a low molecular weight three-dimensional silicone polymer. In this paper we will refer to “resin” to mean this three-dimensional polymer. These resins act as reinforcing fillers and thus impart tremendous improvements to the physical properties of the cured LIM material. An additional benefit of using MQ resins as reinforcing fillers is their high degree of miscibility in PDMS. This results in low initial viscosities of the uncured LIM inputs. In addition to MQ resin, functionalized resins can be employed such as $M_xM^{vi}_yQ$ or $M_xD^{vi}_yQ$ wherein the filler resin can react with the SiH-containing cross-linker and become part of the cured network.

The most common commercial MQ resin has a molecular formula of $[M_{0.65}Q^{OH}_{0.15}Q_{0.85}]_{30}$.[2] This formula corresponds to a hyperbranched resin structure which has an average molecular weight (M_n) of about 3500 g/mol. Most MQ resins also contain a high level of OH groups, typically about 2% by weight OH groups per weight of the total resin. These OH groups reside on silicate Q sites which are not fully condensed. A unique property of these materials is their high degree of solublity in organic solvents and poly(dimethylsiloxanes), despite their three-dimensional structure. Thus, MQ resins are used as soluble reinforcing fillers in optically clear silicone compositions.

MQ resins are synthesized using sol−gel processes.[3] The most frequently used process is the hydrolysis and condensation of Me_3SiCl in the presence of aqueous sodium silicate.[4] MQ resins can also be synthesized by cohydrolysis and condensation of $Si(OEt)_4$ and Me_3SiCl.[5] These sol−gel processes can by modified by substituting

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chlorosilanes containing SiH or Si–vinyl groups for some of the Me_3SiCl to produce hydrosilylation reactive MM^HQ or $MM^{vi}Q$ resins. Alternatively, OH groups on MQ resins can be silylated with silazanes such as tetramethyldisilazane or tetramethyldivinylsilazane[6] or chlorosilanes to give similar hydrosilylation reactive MM^HQ or $MM^{vi}Q$ resins.

Spray-drying is a well-known industrial process for rapidly removing solvents, usually water, from a variety of materials such food products, fertilizers, plastics, or pharmaceuticals.[7] Due to the extremely low residence time, spray-drying is particularly useful for drying thermally sensitive materials. One good example is spray-dried, powdered milk.[7] Spray-drying is also used to prepare powders having controllable particle sizes.[7] Powdered silicone MQ resins having a number average particle size of 20 μm have recently been prepared by spray-drying solutions of MQ resin in toluene or xylene.[8] The resulting powder can be dispersed into silicone polymers by simple hand mixing. Conversely, when MQ resins are dried by more conventional means, a sticky solid is obtained which cannot be readily dispersed into a silicone polymer LIM mixture without the aid of solvent.[9]

Spray-drying of MQ resins has been achieved by combining a solution of MQ resin in an aromatic solvent with an inert heated gas in a two-fluid nozzle.[8,9] Due to the use of the flammable aromatic solvent, the heated gas was nitrogen to avoid an explosion. The dryer inlet temperature was 250 °C, the outlet temperature was 142 °C, and the dried resin contained 1.2% residual solvent.[9]

In 1959 Speier described the first soluble platinum hydrosilylation catalyst composed of H_2PtCl_6 in 2-propanol.[10] Subsequently silicone-soluble platinum catalysts have been introduced with a general structure $Pt(M^{vi}M^{vi})_x$.[11] The common features of the platinum catalysts in use industrially for the last 20 years are the Pt(0) oxidation state and the coordination environment about platinum composed of vinylsilicon groups. These platinum(0) olefin compounds have several distinguishing spectroscopic features including characteristic ^{195}Pt chemical shifts and diagnostic ^{1}H and ^{13}C NMR shifts and couplings. Previous attempts to use solid platinum compounds as catalysts has shown that the solid compounds are either poorly miscible with poly(methylsiloxane) polymers and have poor catalytic activity or both. A typical heterogeneous catalyst such as Pt/C is poorly miscible in poly(methylsiloxane) polymers.[12,13] Furthermore, LIM systems cure at a very slow rate in the presence of Pt/C compared to $Pt(M^{vi}M^{vi})_x$. There are many other examples of solid platinum compounds employed as hydrosilylation catalysts. These compounds have poor activity for cure of LIM materials at ambient temperature and include $CpPtMe_3$,[14] $PtCl_2(CH_3CN)_2$,[15] Pt–styrene complex,[16] Pt–N complexes,[17] Pt–phosphine–vinyl complexes,[18] and Pt–poly(dimethylsiloxane) ammonium compounds.[19] In addition solid Pt–alkyne complexes are not stable in air.[20]

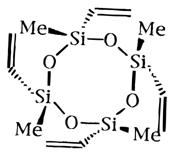
This paper describes our efforts to make free flowing solid powdered $M_xM^{vi}{}_yQ$ resins containing platinum and the use of these resins as silicone LIM cure catalysts.[21]

Results and Discussion

Preparation of Pt–Resin Complexes: Initial Attempts. A $M_{0.74}M^{vi}{}_{0.07}Q$ resin (1.5% by weight vinyl functionality) was prepared as a 60% solids mixture in xylene. The resin was synthesized by silylating the OH groups on a standard, commercial MQ resin with an excess of divinyltetramethyldisilazane. The MQ resin was prepared by reaction of Me_3SiCl with an aqueous sodium silicate hydrosol in the presence of 2-propanol and toluene solvent.[4]

Two attempts were made to make a Pt–resin-complex using a standard vacuum oven to remove solvents. In one case $Pt(D_4{}^{vi})_x$, **1**, was employed as the platinum source. Complex **1** was prepared according to eq 1.[11,22]

$$H_2PtCl_6 + D_4{}^{vi} \xrightarrow[2.\ NaHCO_3]{1.\ 100°C} \underset{\underline{1}}{(Pt(D_4{}^{vi})_x)} + \text{higher cyclics} \quad (1)$$



$D_4{}^{vi}$

Complex **1** was combined with the solution $M_{0.74}M^{vi}{}_{0.07}Q$ resin in toluene. Attempts to dry the mixture of **1** with resin in a vacuum oven gave unusable sticky masses. The high boiling point of $D_4{}^{vi}$ and the higher cyclics formed upon production of **1** in eq 1, precluded the use of spray drying to remove all volatiles.

Platinum-Spray-Dried Resin. Complex **2** was prepared as shown in eq 2. This xylene solution contained about 5.5% platinum by weight. When a

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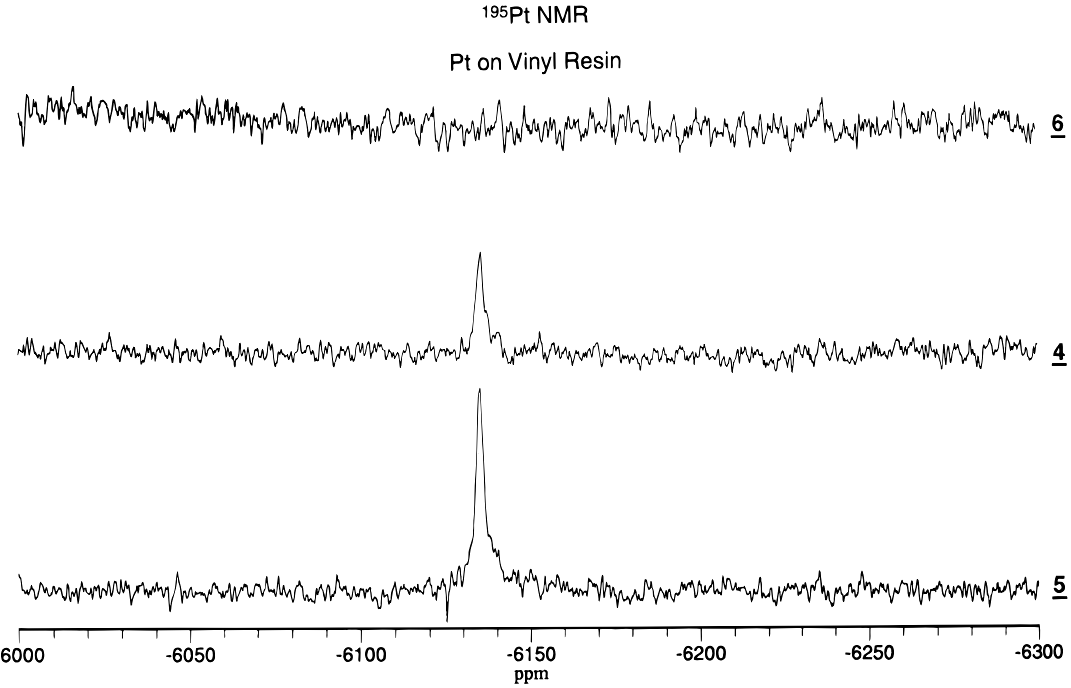

Figure 1. ^{195}Pt NMR spectra for **4** (middle), **5** (bottom), and **6** (top).

Table 1. Platinum Compositions[a]

no.	complex	description	% Pt
1	$Pt(D_4{}^{vi})_x$	Pt in a mixture of cyclic vinyl oligomers	1.8
2	$Pt(M^{vi}M^{vi})_x$	in xylene	5.65
4	resin **3** + **2**	vacuum oven-treated, gray	0.82
5	resin **3** + **2**	spray dried, white	1.05
6	resin **3** + **2**	vacuum oven-treated **5**, brown	1.06

[a] Resin = $M_{0.74}M^{vi}{}_{0.07}Q$.

$$H_2PtCl_6 + xsM^{vi}M^{vi} \xrightarrow[\substack{2.\ NaHCO_3\\ 3.\ Vacuum\ strip,\ add\ xylene}]{1.\ 100\ ^{\circ}C} \underset{\mathbf{2}}{(Pt(M^{vi}M^{vi})_x)} + \begin{matrix} M^{vi}D_xM^{vi} \\ x = 0-9 \\ \bar{x} = 2 \end{matrix} \quad (2)$$

mixture of **2** and the toluene solution of $M_{0.74}M^{vi}{}_{0.07}Q$ resin, **3**, were combined and dried by heating in a vacuum oven at 130 °C for 2 h, a gray, free-flowing powder was obtained, **4** (1% Pt based on solids). If instead the mixture of **2** and **3** were spray-dried, then a white, free-flowing powder was obtained, **5**. Finally sample **5** was treated in a vacuum oven at 150 °C for 2h to give a brown powder, **6** (**5** lost 3% of its weight due to the vacuum treatment, Table 1).

Analysis of Pt Complexes. The ^{195}Pt NMR spectrum for the two liquid platinum mixtures, **1** and **2**, show upfield resonances at about −6100 ppm. ^{195}Pt NMR analysis was carried out for complexes **4**–**6**. As shown in Figure 1, a sharp resonance at around −6100 ppm was observed for **5**, while no resonance was observed for **6** and a broad, less intense resonance was observed for **4** in this region. All three samples were prepared in equal concentrations, and the NMR spectra were run for the same number of transients. These results suggest that **4**–**6** contain different amounts of the molecular Pt(0)–vinyl compound $Pt(M_{0.74}M^{vi}{}_{0.07}Q)_x$. The −6100 ppm ^{195}Pt NMR resonance was indicative of such a molecular compound. The ^{13}C NMR spectrum for **5** showed resonances at 57.1 and 67.1 ppm consistent with vinyl bound to platinum.[11]

Complexes **4**, **5**, and **6** were also analyzed by high-resolution electron microscopy (HREM). Complex **6**, (Figure 2), shows 30–40 Å diameter particles which were shown by energy-dispersive spectroscopy (EDS) to contain platinum. In addition, diffraction fringes were observed for these particles whose spacings were consistent with the 200 spacing in crystalline platinum. These platinum crystallites were reminiscent of those reported previously as end-product from platinum-catalyzed hydrosilylation reactions.[23] Agglomeration was not observed in previous TEM studies of evaporated solutions from platinum-catalyzed hydrosilylation reactions[24] and agglomeration of platinum particles was not observed in the present study either. HREM analysis for **4** also showed the presence of the platinum crystallites, but there were significantly fewer of these particles per unit area than there were for sample **6**. These results, taken with the ^{195}Pt NMR data, suggest that **6** is composed entirely of crystallites and devoid of molecular compounds, while **4** contained a mixture of crystallites and molecular compound. Image analysis of the HREM photographs showed that for **4** there was an average of about 1800 particles/m^2, while for **6** there were about 16 000 particles/m^2. Because there is a lower overall concentration of molecular compounds in **4** versus **5**, the intensity of the ^{195}Pt NMR signal was lower for **4** than that observed for **5**. EDS analysis of **5** did indicate that platinum was present throughout the sample, but no platinum clusters were observed. These results were consistent with **5** being entirely composed of molecular compounds of Pt(0) containing silicon–vinyl ligands derived from the $M_{0.74}M^{vi}{}_{0.07}Q$ resin.

Catalytic Activities of Platinum Materials. The relative activity of the platinum materials described in

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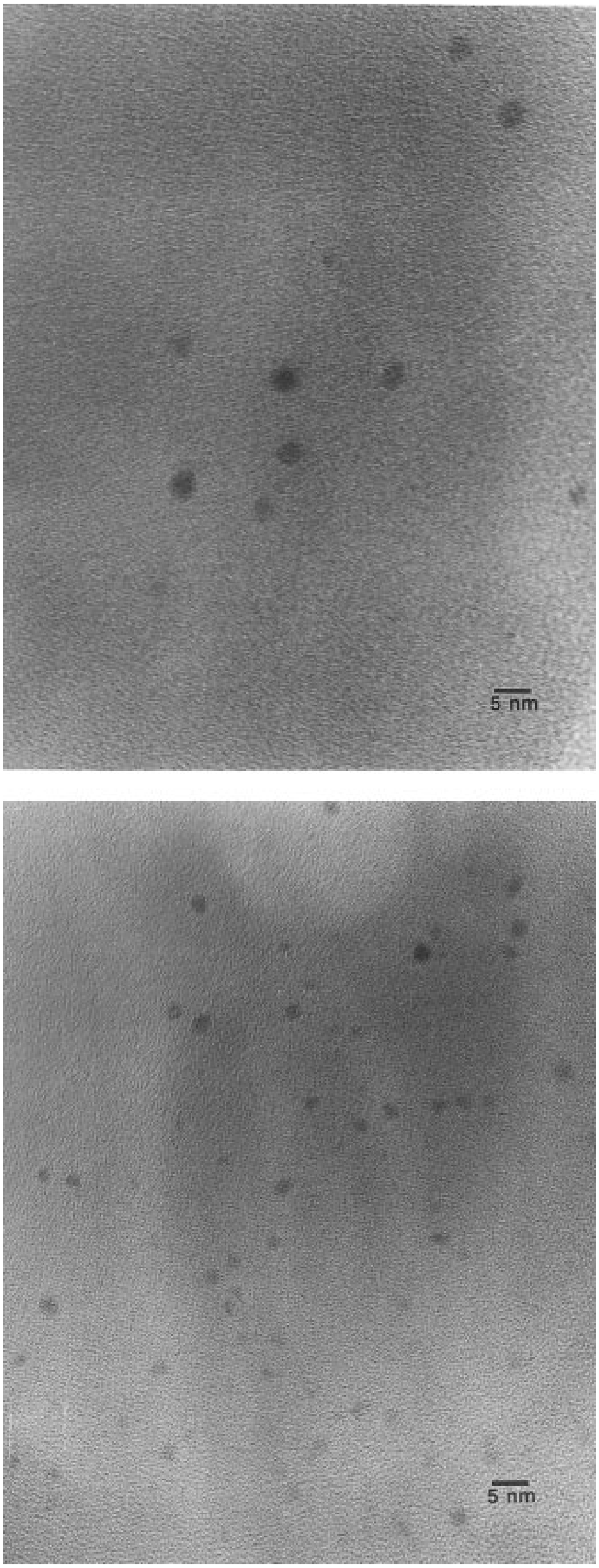

Figure 2. HREM of **4** (top) and **6** (bottom).

Table 2. Cure Rate of Platinum Materials

catalyst	slope	normalized rel rate
1	2.98×10^{-2}	1.85
2	1.8×10^{-2}	1.11
4	1.77×10^{-3}	0.11
5	1.61×10^{-2}	1.0
6	4.94×10^{-3}	0.31

Table 3. Hardness as a Function of Time for Curable LIM

	hardness Shore A durometer		
catalyst	4 h	22 h	comment
1		33	control
4	0	0	not cured 4 h, tacky at 22 h
5	8.5	23	mostly cured at 4 h
6	0	4	tacky at 4 h, partially cured at 22 h

Table 1 were measured using a curable LIM system. The cure system was composed of an A and B component. The A component was composed of 75% by weight of $M^{vi}D_xM^{vi}$ polymer (viscosity = 80 000 cps) and 25% by weight $M_{0.65}D^{vi}_{0.07}Q$ resin. The B component was composed 68.7% by weight of $M^{vi}D_xM^{vi}$ polymer, 22.9% by weight $M_{0.65}D^{vi}_{0.07}Q$ resin and 8.4% by weight $[M^{H}_2Q]_x$ resin. In a typical experiment platinum material was combined with the A component and then equal weights of this A + Pt mixture was combined with the B component to initiate the reaction. The combined A + Pt + B mixture was degassed, and then the viscosity was measured as a function of time in a cone and plate viscometer at constant temperature. A plot of the natural logarithm of viscosity vs time gave a straight line whose slope was used as the apparent rate of cure. Table 2 gives the results from these experiments. As shown in Table 2 the activity of **5** was about the same as **2**.

Catalyst **1** was the most active. Presumably the difference in activity between and **1** and **2** reflects the difference in chelating ability between $M^{vi}M^{vi}$ and D_4^{vi}; D_4^{vi} is more difficult to replace by oncoming substrate vinyls than $M^{vi}M^{vi}$. Material **6** had about a third the activity of **5**. Note that **6** was composed of platinum crystallites. The least active complex was **4**, and this was composed of a mixture of molecular Pt(0) complexes and of platinum crystallites. These results are consistent with complex **5** having a platinum environment equivalent to platinum in **1**; D_4^{vi} and $MM^{vi}Q$ have roughly equivalent ligating ability to platinum. These results also are consistent with Pt(0) olefin complex being the most active catalyst for hydrosilylation.[23]

A second, curable LIM formulation was prepared to measure relative activity by using the hardness of the cured LIM material to compare relative degree of cure as a function of which platinum material was present. In this case 94.6% by weight of the same A component described above was mixed with the appropriate platinum material (30 ppm Pt by weight). Then 4.4% by weight of the B component composed of $[M^{H}_2Q]_x$ resin was added. After addition of the B component, cure time (if any) was noted and the hardness was measured by Shore A durometer. A control sample using complex **1** gave a Shore A durometer reading of 33 after 22 h at ambient temperature. Table 3 shows the relative cure rates for the other platinum materials. These results again show that complex **5** had roughly the same activity as **1**.

Conclusions

This report describes a unique, solid material containing platinum. This material was composed of free-flowing white powder with 1% by weight platinum in the form of a Pt(0) olefin complex. The solid was mostly composed of $MM^{vi}Q$ resin. These resins were completely miscible in silicone and had roughly the same catalytic activity for hydrosilylation as a commercially available platinum catalyst composed of D_4^{vi} ligands. These results show that $MM^{vi}Q$ and D_4^{vi} had about the same ligating ability to platinum. Spray drying provided the only method for producing these platinum-containing resins. Standard oven-drying techniques resulted in the formation of darkened products which contained platinum crystallites and had inferior catalytic activity compared to the spray-dried platinum-containing resin. These results further emphasize the unique ability of spray drying to provide solid materials that are thermally sensitive. The resultant platinum-containing $MM^{vi}Q$ resin has retained its pure white color without loss of catalytic activity with storage at ambient temperature in air for over 2 years.

Experimental Section

General Methods. ^{13}C NMR spectra were recorded on a GE QE-300 instrument at 75.48 MHz. ^{29}Si and ^{195}Pt NMR spectra were recorded on a GE GN-Omega 500 NMR instrument at 76.77 and 107.512 MHz, respectively. ^{29}Si NMR spectra were all recorded with 1% $Cr(acac)_3$ relaxation reagent and employed gated decoupling. The platinum NMR used Na_2PtCl_6 at 0 ppm as an external standard. Platinum analyses were performed by inductively coupled plasma with $Pt(acac)_2$ as a spike standard. HREM analyses were carried out using a JEOL Model 2010 EM operated at 200 kV on a copper-coated carbon grid. Energy-dispersive spectroscopy was performed using a LINK Model XEL instrument. Particle analyses were carried out by normalizing equal field areas to obtain statistics. The images were contrast enhanced to better delineate the particles. Viscosity measurements were made with a Brookfield Model DV-II digital cone and plate viscometer using a No. 52 cone. The viscometer's bottom block was connected in series to a constant-temperature bath. Hardness of cured formulations was measured with a Shore Durometer Hardness Type A device.

Preparation of $M_{0.74}M^{vi}_{0.07}Q$ Resin. The first stage of the synthesis of this Si−vinyl substituted MQ resin was the preparation of a silanol-substituted MQ resin. This procedure is a variation of the method described by Daudt and Tyler.[4] An aqueous sodium silicate solution (91 g, 27% by weight SiO_2) was mixed thoroughly with water (130 g). This mixture was added with stirring to HCl (81 g, of a 16.5% by weight solution) in cooled water over a period of 7 min. Shortly after this addition was complete (within 45 s), 2-propanol (87 g) was added over a period of 5 min. After a delay time of 1 min, a mixture of Me_3SiCl (31 g) and toluene (3.4 g) was next added over a 20 min time period. The reaction mixture was heated to reflux for 1.5 h. The remaining amount of toluene solvent (40.5 g) was then added with stirring. The reaction mixture was next transferred to an addition funnel in which the organic and aqueous layers were separated. The aqueous layer was discarded as waste, and the resin-containing organic layer was washed with water. The water/2-propanol/toluene azeotrope was distilled from the mixture yielding a 60% by weight solution of a silanol containing MQ resin with an M/Q ratio of 0.6−0.7 and an OH content of 2% by weight.

This silanol-substituted MQ resin was silylated with divinyltetramethyldisilazane ($[Me_2ViSi]_2NH$) as follows: A mixture of 1,3-divinyltetramethyldisilazane (900 g) and the 60% MQ resin by weight in toluene solution prepared above (2400 g) were refluxed under nitrogen for 15 h. Infrared analysis showed that the majority of the silanol groups on the MQ resin were absent, indicating that M^{vi} groups had been introduced onto the resin. The reaction mixture was then spray dried using a Niro portable spray dryer equipped with a two-fluid nozzle. The drying gas was hot nitrogen. The rate of drying was 63 kg/h and the inlet/outlet temperatures were 244 °C/118 °C. A ^{29}Si NMR analysis of the resulting free-flowing resin powder indicated it had a composition of $M_{0.74}M^{vi}_{0.07}Q$ as determined by integration of the three observed resonances.

Preparation of $MM^{vi}Q$−Pt Complex, 5. The above $MM^{vi}Q$ resin (400 g) was dissolved in 267 g of toluene. This solution was combined with 80.3 g of a xylene solution of a platinum complex (5% Pt by weight) obtained by reacting H_2PtCl_6 with 1,3-divinyltetramethyldisiloxane according to the method of Karstedt.[11] The reaction mixture was then spray dried using a Niro portable spray dryer equipped with a two-fluid nozzle. The drying gas was nitrogen. The rate of drying was 75 kg/h, and the inlet temperature was 180 °C. Elemental analysis, ^{195}Pt NMR, and TEM analysis of the resulting white, free-flowing powder is described above.

Relative Rate Study. A heat-curable silicone was prepared by mixing vinyl-terminated poly(dimethylsiloxane) fluid (80 000 cps, 72 g), vinyl polymer $M_{0.65}D^{vi}_{0.07}Q$ (23.8 g), M^H_2Q (4.2 g) and platinum catalyst (see Tables 1 and 2). For example **5** (0.048 g, 10 ppm) was used, and the rate of cure was measured by measuring viscosity changes vs time at 30 °C.

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