Preparation of 7,8-Diacetyl-7,8-dicyanoquinodimethane and 7,8-Dibenzoyl-7,8-dicyanoquinodimethane and Their Polymerization Behavior

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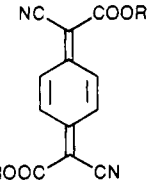
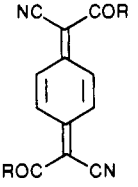
ABSTRACT: 7,8-Diacetyl-7,8-dicyanoquinodimethane (**2a**) and 7,8-dibenzoyl-7,8-dicyanoquinodimethane (**2b**) were prepared successfully as related compounds to 7,8-bis(butoxycarbonyl)-7,8-dicyanoquinodimethane (**1c**). First reduction potentials of **2a** and **2b** are +0.03 and −0.02 V, respectively, by cyclic voltammetry. When **2a** and **2b** were dissolved in polar solvents such as acetonitrile, acetone, tetrahydrofuran, *N*,*N*-dimethylformamide, and dimethyl sulfoxide, their polymerizations took place to give polymers with the molecular weight range of 2×10^3 to 2×10^5. **2a** and **2b** are readily homopolymerizable with radical and anionic initiators. Copolymerizations of the **2a**–styrene (St), **2b**–St, and **1c**–St systems with 2,2′-azobis(isobutyronitrile) in dichloromethane or chloroform at 50 °C took place in random fashion to obtain the monomer reactivity ratios as follows: r_1(**2a**) = 2.02 ± 0.2 and r_2(St) = 0.015 ± 0.01 for the **2a**–St system, r_1(**2b**) = 0.73 ± 0.2 and r_2(St) = 0.021 ± 0.015 for the **2b**–St system, and r_1(**1c**) = 2.59 ± 0.2 and r_2(St) = 0.030 ± 0.01 for the **1c**–St system.

Previously we reported that 7,8-bis(alkoxycarbonyl)-7,8-dicyanoquinodimethanes (**1a–c**) with two different substituents (cyano and alkoxycarbonyl groups) at the 7- and 8-positions were successfully prepared as stable compounds and that they are homopolymerizable with free radical and anionic initiators and copolymerizable with styrene (St) in a random fashion.[1,2] Their polymerization behaviors are widely different from those of 7,7,8,8-tetracyanoquinodimethane(TCNQ)[3] and 7,7,8,8-tetrakis(ethoxycarbonyl)quinodimethane[4] with identical substituents at the 7- and 8-positions, the latter compounds being scarcely or sparingly homopolymerizable and being copolymerizable with St in an alternating fashion. No quinodimethane compound with properties similar to those of the compound **1**, high homopolymerizability and random copolymerizability with St, has been known other than the compound **1**.

In this paper, we describe preparation and polymerization of 7,8-diacetyl-7,8-dicyanoquinodimethane (**2a**) and 7,8-dibenzoyl-7,8-dicyanoquinodimethane (**2b**) with acetyl and benzoyl groups, respectively, instead of alkoxycarbonyl groups, as related quinodimethane compounds to the compound **1**.



1a, R = Me
b, R = Et
c, R = Bu



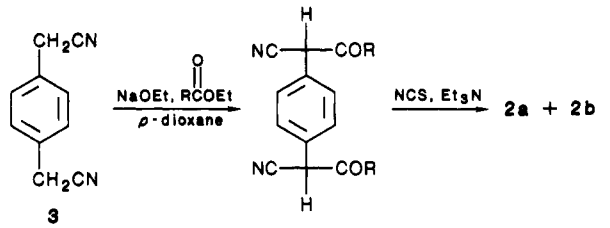
2a, R = Me
b, R = Ph

Experimental Section

2a and **2b** were prepared successfuly as the Scheme I.

Preparation of 4a. Sodium ethoxide (3.26 g, 48 mmol) was dissolved in 80 mL of dry *p*-dioxane and 3.0 g (19.2 mmol) of 7,8-dicyano-*p*-xylene, (**3**), and 3.89 g (44 mmol) of ethyl acetate was added to the solution. After it was refluxed under nitrogen for 2 h, the reaction mixture was placed under reduced pressure to remove volatile materials to yield dark brown solid, to which 400 mL of water was added to dissolve it. An amount of dilute hydrochloric acid enough to allow the resulting aqueous solution to be acidic was added to deposit yellow solid which was sucked off and dried under reduced pressure to obtain 4.3 g of crude **4a**. This was recrystallized from acetonitrile to give pale yellow powder of **4a** in 87% yield (4.0 g): mp 200–202 °C; IR (KBr) ν(CN) 2220, ν(C=O) 1640 cm^{-1}. Anal. Calcd for $C_{14}H_{12}N_2O_2$: C, 69.68; H, 5.50; N, 11.66. Found: C, 69.59; H, 5.50; N, 11.04.

Scheme I



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4a, R = Me
b, R = Ph

Preparation of 2a. The compound **4a** (1.0 g, 4.2 mmol) was suspended in 10 mL of acetonitrile under nitrogen. To the suspension kept at 0 °C was added 1.1 g (8.2 mmol) of *N*-chlorosuccinimide (NCS). After the mixture was stirred for 3 min, 2 drops of triethylamine was added to the mixture to allow the color change of the mixture from yellow to orange. The orange solid was filtered off and dried under reduced pressure. The solid obtained was dissolved again in 30 mL of dichloromethane and then the insoluble portion, unreacted **4a**, was removed by filtration. The filtrate was evaporated under reduced pressure to dryness to yield 200 mg of crude **2a**, which was recrystallized from dichloromethane to give crystalline orange plates of **2a** in 14% yield (140 mg): mp 180 °C dec; IR (KBr) ν(CN) 2200, ν(C=O) 1680, ν(C=C) 1530 cm^{-1}; ^{1}H NMR (60 MHz, $CDCl_3$) δ 2.63 (s, 6 H), 7.41 (dd, J = 10, 1.7 Hz, 2 H), 8.37 (dd, J = 10, 1.7 Hz, 2 H); UV ($CHCl_3$) 414 nm (ϵ 3.76×10^4). Anal. Calcd for $C_{14}H_{10}N_2O_2$: C, 70.57; H, 4.24; N, 11.76. Found: C, 70.43; H, 4.39; N, 11.41.

Preparation of 4b. The compound **4b** was prepared in 85% yield according to the procedure similar to that for **4a** except ethyl benzoate was used instead of ethyl acetate: mp 218–220 °C; IR (KBr) ν(CN) 2200, ν(C=O) 1700 cm^{-1}. Anal. Calcd for $C_{24}H_{16}N_2O_2$: C, 79.09; H, 4.43; N, 7.69. Found: C, 78.91; H, 4.20; N, 7.62.

Preparation of 2b. The compound **4b** (1.0 g, 2.7 mmol) was suspended in 100 mL of chloroform under nitrogen. To the suspension kept at 0 °C was added 0.72 g (5.2 mmol) of NCS. After stirring for 5 min, 2 drops of triethylamine was added, changing the yellow suspension to a red homogeneous solution, which was washed twice with ice water (100 mL × 2) and dried over anhydrous magnesium sulfate. The chloroform solution was placed under reduced pressure to evaporate the solvent until its volume became about 15 mL. The resulting solution was passed through the column (1.5 cm diameter × 30 cm high) packed with silica gel using chloroform as eluent. The first elution band portion was evaporated under reduced pressure to dryness to obtain 380 mg of crude **2b**, which was recrystallized from toluene to give crystalline orange plates of **2b** in 29% yield (290 mg): mp 183.0–183.5 °C; IR (KBr) ν(CN) 2200, ν(C=O) 1660, ν(C=C) 1600 and 1550 cm^{-1}; ^{1}H NMR (60 MHz, $CDCl_3$) δ 8.10–7.30 (m); UV

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Table I
First Reduction Potentials[a]

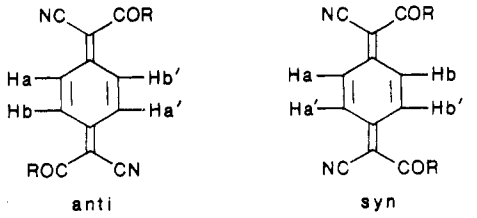
monomer	E_1, V[b]	monomer	E_1, V[b]
TCNQ	+0.19	**2b**	−0.02
2a	+0.03	**1c**	−0.10
PCA	+0.01		

[a] Solvent, dichloromethane containing $(Bu_4N)ClO_4$ (0.1 mol/L); reference electrode, Ag/AgCl. [b] Relative error, ± 0.01 V.

$(CHCl_3)$ 398 nm (ϵ 3.81 × 10^4). Anal. Calcd for $C_{24}H_{14}N_2O_2$: C, 79.54; H, 3.90; N, 7.73. Found: C, 78.09, H, 3.89; N, 7.65.

Results and Discussion

Configuration and Electron-Accepting Character. Both **2a** and **2b** should exhibit geometric isomerism in the syn and anti forms. It is conceivable in their ^1H NMR



spectra that hydrogen atoms (Ha and Hb) on the quinodimethane nucleus exhibit two double doublet peaks (Ha, double doublet (α- and β-coupling); Hb, double doublet (α- and β-coupling)) for the anti form and two double doublet peaks (Ha, double doublet (β- and γ-coupling); Hb, double doublet (β- and γ-coupling)) for the syn form. The values of α-, β-, and γ-coupling constants on aromatic compounds were reported to be 6–10 Hz, 1–3 Hz, and 0–1 Hz, respectively.[5] Two double doublet peaks at δ 7.41 and 8.37 in the ^1H NMR spectrum of **2a** were observed to be split due to coupling with the coupling constants of J_{ab} = 10 Hz and $J_{ab'}$ = 1.7 Hz, indicating the anti form of it.

Unfortunately, the ^1H NMR spectrum of **2b** showed a serious overlapping of hydrogen atoms on the quinodimethane nucleus with hydrogen atoms on the benzoyl group, so we are uncertain at present of its configuration.

Electron-accepting character of **2a** and **2b** was estimated as first reduction potentials, E_1 value, by cyclic voltammetry. Dichloromethane-containing tetrabutylammonium perchlorate (0.1 mol/L) and Ag/AgCl were used as solvent and reference electrode, respectively. The found values for **2a** and **2b** are listed in Table I, together with those of **1c**, TCNQ, and *p*-chloranil (PCA) for comparison. It is obvious that **2a** and **2b** are much stronger in electron-accepting character than **1c** and much less than TCNQ as expected from well-known Hammett values[6] of the electron-withdrawal character of butoxycarbonyl, acetyl, benzoyl, and cyano groups and they are incidentally as strong as PCA.

Polymerization Behavior. **2a** and **2b** were dissolved readily in toluene, benzene, dichloromethane, and chloroform to give red monomer solutions. When **2a** and **2b** were dissolved in polar solvent such as acetonitrile, acetone, tetrahydrofuran, *N*,*N*-dimethylformamide, and dimethyl sulfoxide, the red solutions of **2a** changed to colorless in 10 min and those of **2b** did gradually to pale yellow in 2 h, except for the tetrahydrofuran solution after the dissolution, indicating complete conversion of **2a** and **2b** monomers to their polymers except for **2b** in tetrahydrofuran. The red solution of **2b** in tetrahydrofuran remained almost unchanged in 4 h and it changed to yellow in the period more than 24 h. After standing for 2 h, portions of the resulting solutions were added to excess tetrahydrofuran and the tetrahydrofuran solutions were subjected to gel permeation chromatography to determine the molecular weight for the polymers of **2a** and **2b**. Table II summarizes the results of the polymerizations of **2a** and **2b** in the above-mentioned five kinds of polar solvents. The polymers of **2a** and **2b** obtained in the polar solvents are in the molecular weight range of 2 × 10^3 to 2 × 10^5, which are lower than those of polymers of **1c** obtained in the same solvents ((8 × 10^5) – (4 × 10^6)). It is obvious that **2a** and **2b** are readily homopolymerizable with such polar solvents, as well as **1c**.[1,2]

Table II
Spontaneous Homopolymerizations[a] of 2a and 2b in Various Polar Solvents at Room Temperature

solvent	ν(OD) shift,[b] MeOD	**2a**, $\bar{M}_n/10^4$	**2b**, $\bar{M}_n/10^4$
acetonitrile	49	15.0	2.6
acetone	64	6.8	7.0
tetrahydrofuran	93	20.0	2.1
N,*N*-dimethylformamide	107	0.22	3.4
dimethyl sulfoxide	141	0.48	6.3

[a] Monomer, 5 mg; solvent, 2 mL; time of polymerization, 2 h. [b] As solvent basicity, ν(OD) stretching frequency shift of methanol-*d* due to hydrogen bond formation with solvent was determined.

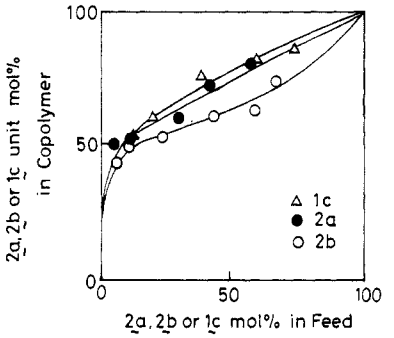


Figure 1. Composition diagram of the copolymerizations for the **2a**–St (●), **2b**–St (○), and **1c**–St (△) systems.

Table III summarizes the results of the polymerization of **2a** and **2b** with conventional anionic, cationic, and free radical initiators. It is obvious that anionic and free radical initiators are highly effective in homopolymerizations of **2a** and **2b** but the cationic one is not. Among proton sponge (pK_a = 12.36),[7] pyrrolidine (11.27),[7] triethylamine (11.01),[7] and pyridine (5.21)[7] as basic amine initiators, the former three compounds are able to conduct homopolymerizations of **2a** and **2b** but the last one is not, similarly to the case of **1c**. Triphenylphosphine is able to initiate the polymerization of **2a** but is not those of **2b** and **1c**, corresponding well with the fact that **2a** (E_1 = +0.03 V) is a stronger electron-accepting compound than **2b** (−0.02 V) and **1c** (−0.10 V). It is concluded, therefore, that **2a** and **2b** exhibit similar behavior in anionic polymerization with various initiators to that of **1c**.

Table IV summarizes the results of the copolymerizations of the **2a**–St, **2b**–St, and **1c**–St systems with 2,2′-azobis(isobutyronitrile) (AIBN) in chloroform or dichloromethane at 50 °C and Figure 1 shows their copolymerization composition curves. The copolymerizations were adequately treated with the Mayo–Lewis equation. It can be pointed out therefore that the copolymerizations are really in random fashion. The monomer reactivity ratios of the **1c**–St system were calculated according to the intersection[8] and Kelen–Tüdös methods[9] to be r_1(**1c**) = 2.59 ± 0.2 and r_2(St) = 0.030 ± 0.01. Those of the **2a**–St and **2b**–St systems were calculated, according to the integrated copolymerization equation[8] and the intersection

Table III
Homopolymerizations[a] of 2a and 2b Initiated by Various Catalysts at 0 °C

run	catalyst (I)	[2a or 2b]/[I]	solvent, mL	time	conv, %	$\bar{M}_n$[b]
			2a			
1	*n*-butyllithium	9.6	toluene, 30	150 (min)	61.3	5600
2	AIBN[c]	2.3	$CHCl_3$, 5	60	31.0	1200
3	BF_3·Et_2O	10.2	CH_2Cl_2, 20	90	no polymer	
4	triphenylphosphine	10.0	$CHCl_3$, 10	10	100	3700
5	proton sponge[d]	9.7	$CHCl_3$, 10	10	95.4	2100
6	pyrrolidine	10.5	$CHCl_3$, 10	5	93.5	3700
7	triethylamine	10.0	$CHCl_3$, 10	10	99.3	6800[e]
8	pyridine	10.0	$CHCl_3$, 10	75	no polymer	
			2b			
9	*n*-butyllithium	10.5	toluene, 30	37 (h)	10.2	6200
10	AIBN[c]	17.5	$CHCl_3$, 5	26	19.5	2800
11	BF_3·Et_2O	10.1	CH_2Cl_2, 10	17	no polymer	
12	triphenylphosphine	10.1	$CHCl_3$, 10	50	no polymer	
13	proton sponge[d]	9.6	$CHCl_3$, 10	18	85.9	880
14	pyrrolidine	10.0	$CHCl_3$, 10	24	8.3	3500
15	triethylamine	10.0	$CHCl_3$, 10	24	23.8	2600
16	pyridine	10.1	$CHCl_3$, 10	25	no polymer	

[a] **2a**, 0.126 mmol; **2b**, 0.083 mmol. Their polymerization procedures are similar to those in ref 2. [b] Determined by GPC. Tetrahydrofuran was the eluent. [c] Temperature of polymerization, 60 °C; AIBN, 2,2′-azobis(isobutyronitrile). [d] Proton sponge, 1,8-bis(dimethylamino)-naphthalene. [e] Soluble part in tetrahydrofuran.

Table IV
Copolymerizations[a] of the 2a–St, 2b–St, and 1c–St Systems at 50 °C

run	monomer acceptor, mg	feed St, mg	acceptor, mol %	time, h	solvent, mL	conv, %	anal. %H	anal. %C	anal. %N	copolymer comp, mol % acceptor	M_n[b]/10^3
					2a						
1	26.91	205.54	5.4	3.0	5	10.3	5.51	77.21	8.16	49.7	9.0
2	29.55	97.20	11.7	1.5	5	9.4	4.67	77.18	8.38	52.0	6.6
3	40.24	40.93	30.1	3.5	7	13.5	4.59	75.66	9.00	58.8	3.1
4	62.93	39.08	41.3	2.5	11	13.8	4.37	73.67	10.09	72.5	4.8
5	64.82	21.26	57.1	2.5	11	8.9	4.68	72.45	10.70	81.3	2.8
					2b						
6	55.55	222.25	6.1	18.5	5	11.5	4.57	82.90	5.58	42.7	6.8
7	47.08	114.07	10.6	19.5	5	13.8	4.59	82.31	5.97	49.4	8.6
8	115.25	102.74	24.4	19.0	5	15.2	4.57	82.25	6.13	52.4	7.4
9	114.32	43.43	43.1	17.5	5	14.7	4.51	81.32	6.55	61.5	7.6
10	242.55	48.83	58.8	13.5	5	14.1	4.21	81.51	6.64	63.6	6.6
11	223.00	30.97	67.4	12.5	5	10.5	4.07	80.79	7.01	73.7	3.1
					1c						
12	51.80	130.35	10.5	0.75	10	6.8	6.41	72.10	6.10	49.8	140
13	89.84	106.46	19.9	0.67	10	6.9	6.04	72.04	6.58	59.2	40
14	120.56	58.58	37.7	0.83	10	9.1	6.55	70.09	7.24	76.0	300
15	200.92	43.45	57.6	0.75	10	9.8	6.25	69.50	7.39	80.6	540
16	200.19	20.23	74.4	0.75	10	6.2	6.26	69.36	7.51	84.6	86

[a] Their polymerization procedures are similar to those in ref 2. Solvent: $CHCl_3$ containing 2 drops of acetic acid for the **2a**–St and **1c**–St systems and CH_2Cl_2 containing 2 drops of acetic acid for the **2b**–St systems. Initiator: 0.2 mg of AIBN for the **2a**–St system, 1 mg of AIBN for the **2b**–St system, and 2 mg of AIBN for the 1c–St system. [b] Determined by GPC. Tetrahydrofuran was the eluent.

methods[8] because of a little bit higher conversion for the differential form of the copolymerization equation, to be r_1(**2a**) = 2.02 ± 0.2 and r_2(St) = 0.015 ± 0.01 for the **2a**–St system and r_1(**2b**) = 0.73 ± 0.2 and r_2(St) = 0.021 ± 0.015 for the **2b**–St system. The products of the monomer reacitivity ratios, r_1r_2, for the **2a**–St, **2b**–St, and **1c**–St systems were calculated to be 0.030, 0.015, and 0.078, respectively, indicating that an alternating tendency increases in the following order: **1c**–St < **2a**–St < **2b**–St systems, being not in agreement with the electron-accepting character of those compounds (**2a** > **2b** > **1c**) (see Table I).

It can be pointed out that **2a**, **2b**, and **1c** exhibit similar polymerization behavior with anionic (even polar solvents) and free-radical initiators and that their copolymerizations with St show fairly different alternating tendency, suggesting that some factors other than a polarity and a general reactivity of a monomer influence significantly their copolymerizations. A study on these factors is now in progress.

Registry No. 2a, 110458-72-5; **2a** (homopolymer), 110458-75-8; (**2a**)(St) (copolymer), 110458-77-0; **2b**, 110458-74-7; **2b** (homopolymer), 110458-76-9; (**2b**)(St) (copolymer), 110458-78-1; **3**, 622-75-3; **4a**, 14557-29-0; **4b**, 110458-73-6; AIBN, 78-67-1; BuLi, 109-72-8; $MeCO_2Et$, 141-78-6; $PhCO_2Et$, 93-89-0; Et_3N, 121-44-8; $PhCH{=}CH_2$, 100-42-5; Ph_3P, 603-35-0; pyrrolidine, 123-75-1; 1,8-bis(dimethylamino)naphthalene, 20734-58-1.

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Poly(dimethylsiloxane-*co*-diphenylsiloxanes): Synthesis, Characterization, and Sequence Analysis

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ABSTRACT: Poly(dimethylsiloxane-*co*-diphenylsiloxane) copolymers have been prepared by ring-opening and step-growth condensation polymerizations. Composition of the copolymers has been determined by ^1H and ^{29}Si NMR spectroscopy. Sequence distribution up to the pentad level is observable in the ^{29}Si NMR spectrum. Copolymers have been characterized by GPC and DSC measurements. The glass transition temperatures increase with an increase in the proportion of diphenylsiloxane content.

Introduction

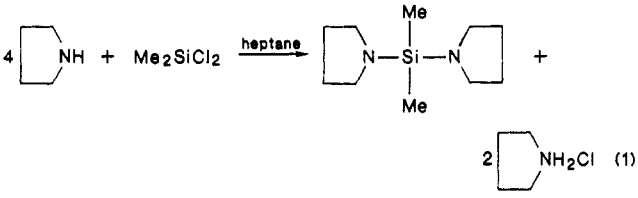
Silicone elastomers have long been known for their exceptional ability to exhibit and retain superior mechanical properties over a broad temperature range.[1] The main interest in these materials stems from the fact that they possess unique properties such as good low-temperature flexibility, excellent electrical properties, chemical inertness, water repellency, and biocompatibility not common in hydrocarbon polymers.[2,3] Various structural modifications have been investigated in order to modify some of the properties. For example reinforcement by silica fillers help in modifying the weak cross-linked network of conventional silicone rubber.[4] Segmented copolymers comprising hard and soft segments are an alternative approach to obtain polymers with specific end uses.[5-7] Synthesis of low and high molecular weight poly(dimethylsiloxane-*co*-diphenylsiloxanes) have also been reported.[8-10] It has been shown that introduction of diphenylsiloxane segment usually disrupts the low-temperature crystallization of polydimethylsiloxane and also increases the thermal and radiation stability.[1] There has been no systematic study of the structure-property relationship of these novel copolymers. The objective of this investigation is to synthesize and characterize high molecular weight poly(dimethylsiloxane-*co*-diphenylsiloxanes) and determine their sequence by ^{29}Si NMR spectroscopy. An attempt has also been made to correlate sequence distribution with the physical properties of the copolymers.

Experimental Section

Spectra. Proton and ^{29}Si NMR spectra were obtained on Varian XL-100 and XL-400 NMR spectrometers, respectively, at 100 MHz (^1H) and 79.46 MHz (^{29}Si). Chemical shifts are given relative to tetramethylsilane internal reference. Integrals on the proton spectra were obtained directly on concentrated solutions in chloroform-*d*. Ten milligrams (0.01 M) $Cr(acac)_3$ relaxation reagent was added to each 3 cm^3 solution of the samples before the ^{29}Si spectra was obtained in order to eliminate the nuclear Overhauser enhancement and reduce the relaxation times (T_1). Measurement of T_1 and nOe on a representative sample indicated all silicons in the polymer multiplets had comparable T_1 values of 5.0 s and that the nOe was 0.9. Quantitative ^{29}Si spectra were obtained by using 35° pulses and a 1.9-s recycle time to maximize the signal/noise. This partial saturation of the peaks will not effect integrals used to calculate the sequence distribution since all the polymeric absorptions had similar relaxation times. Also, the ratios of $SiPh_2O$:$SiMe_2O$ observed in the ^1H spectra match those observed in the ^{29}Si spectra.

Materials. Octamethylcyclotetrasiloxane (methyl tetramer, D_4), octaphenylcyclotetrasiloxane (phenyl tetramer, D_4^{Ph}), and diphenylsilanediol (DSD) were obtained from Petrarch Systems or Silar Laboratories. Bis(dimethylamino)dimethylsilane and 1,7-bis(dimethylamino)hexamethyltrisiloxane (Silar Lab.) were freshly distilled before use.

Synthesis. Bis(pyrrolidinyl)dimethylsilane. A dry 1-L three-necked, round-bottom flask was fitted with a reflux condenser, dropping funnel, mechanical stirrer, and an argon inlet. The apparatus was degassed by alternately evacuating and filling with argon. With argon flow through the system, freshly distilled dimethyldichlorosilane (90 g, 0.7 mol) and dry heptane (150 mL) were placed in the reaction vessel (eq 1). Dry pyrrolidine (199



g, 2.8 mol) was added dropwise over a period of 3 h with stirring and cooling in an ice bath. After completion of the addition, the reaction mixture was stirred overnight at ambient temperature. The amine hydrochloride precipitate was removed by filtration with positive argon pressure. The solvent was removed under vacuum. The residue was distilled at 85-87 °C (0.1 mm) in 95% yield: IR (neat) C-H, 3000, 2960, 2875 cm^{-1} (s, s, s); Si-Me, 1265 cm^{-1} (s), Si-N, 995 cm^{-1} (m).

Bis[phenyl(1-pyrrolidinylcarbonyl)amino]dimethylsilane[11,12] (Referred to as Diureidosilane). A 1-L, three-neck round-bottom flask was equipped with mechanical stirrer, an argon inlet, a reflux condenser, and a dropping funnel. The apparatus was alternately evacuated and filled with argon six to eight times. Freshly distilled dipyrrolidinyldimethylsilane (100 g, 0.5 mol) and 350 mL of dry ether were added, and the mixture was stirred in a 0-5 °C ice bath for approximately 10 min (eq 2). Dry phenyl isocyanate (119 g, 110 mol) was added to the chilled solution over a 1-h period. A white solid slowly separated during addition. Stirring was continued for an additional 3 h to ensure the completion of the reaction. The compound was then filtered under positive argon pressure and washed with dry pentane. The product was isolated in 90% yield: mp 125 °C lit.[11] mp 124-125 °C. Purity of the product was confirmed by elemental analysis. IR (KBr): C-H, 3000, 2960, 2875 cm^{-1} (s, s, s), Si-Me, 1260 cm^{-1} (s), N-H, 3475 cm^{-1} (s).

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0024-9297/87/2220-2654$01.50/0