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End-capped carbosiloxane dendrimers with cholesterol and pyridine derivatives

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Abstract

Dendritic carbosilanes containing 48 and 96 cholesterol as well as pyridine derivatives on the periphery have been prepared. Low generations in the inner shell were prepared by the use of iterative hydrosilation with dichloromethyl silane and alcoholysis with allyl alcohol. The cholesterol and pyridine derivatives containing dendrimers were prepared by the reaction of the Si–Cl group on parent dendrimers (G4-48Cl and G5-96Cl) and HOR (HOR = cholesterol, *p*-pyridinepropanol, and *p*-pyridinealdoxime) in the presence of TMEDA. © 2001 Published by Elsevier Science B.V.

Keywords: Dendrimer; Carbosilane; Siloxane; Cholesterol; Pyridine

1. Introduction

Dendrimers are macromolecules containing a regular, unified and highly branched structure and precisely determined characteristic end groups [1–4]. Much attention has currently been devoted to the dendrimers because of their importance in many fields such as material science and applications that are suggested in the literature including their uses for nanomaterials [5–7]. Peripherally modified dendrimers can be easily approached for characteristic patterns. Therefore, dendritic research has lately been shifted from finding the simple construction of unified macromolecules to materials with specific functions with exhibit supramolecular properties [8–10]. Recently, carbosilane as well as carbosiloxane dendrimers have appeared as one of important families of dendritic macromolecules [10,11].

Synthetic methods of silicone-containing dendrimers were reported by a great number of researchers who used the repeating procedures, such as hydrosilation and alkenylation [12–15]. As an extension of our previous work [16–18] we now try to find the preparative method of peripheral generations with cholesterol and pyridine derivatives. The reaction of the Si–Cl bonded dendritic generation with HOR (HOR = *p*-pyridinepropanol, *p*-pyridinealdoxime and cholesterol) in the presence of TMEDA (1,1,2,2-tetramethylethyenediamine) revealed Si–O–R bonded dendrimers with very high yields. The amine derivatives in the terminal make an estimate of strong basic properties that can act as a metal holder in nanoscale compounds [19–21] and the cholesterol-containing dendrimers are expected to possess liquid crystalline properties [22]. In this paper, we will report the synthetic method and the identification, which can be obtained by the ^{1}H- and ^{13}C-NMR, UV and IR spectroscopy, SEC measurement as well as elemental analysis.

2. Results and discussion

The preparation of low generations in the inner shell of parent dendrimers was established with siloxane tetramer as the core, while growth branches ($-SiMe(OCH_2CH{=}CH_2)_2$) were prepared by the use of catalytic hydrosilation and alcoholysis [17,23,24]. The G*n*P-*m*Cl generations ($n = 4$, $m = 48$; $n = 5$, $m = 96$) containing chlorodimethylsilyl (Me_2SiCl) groups on peripheral layers were prepared by the catalytic hydrosilation of the third generation (G3-48Allyl) as well as the

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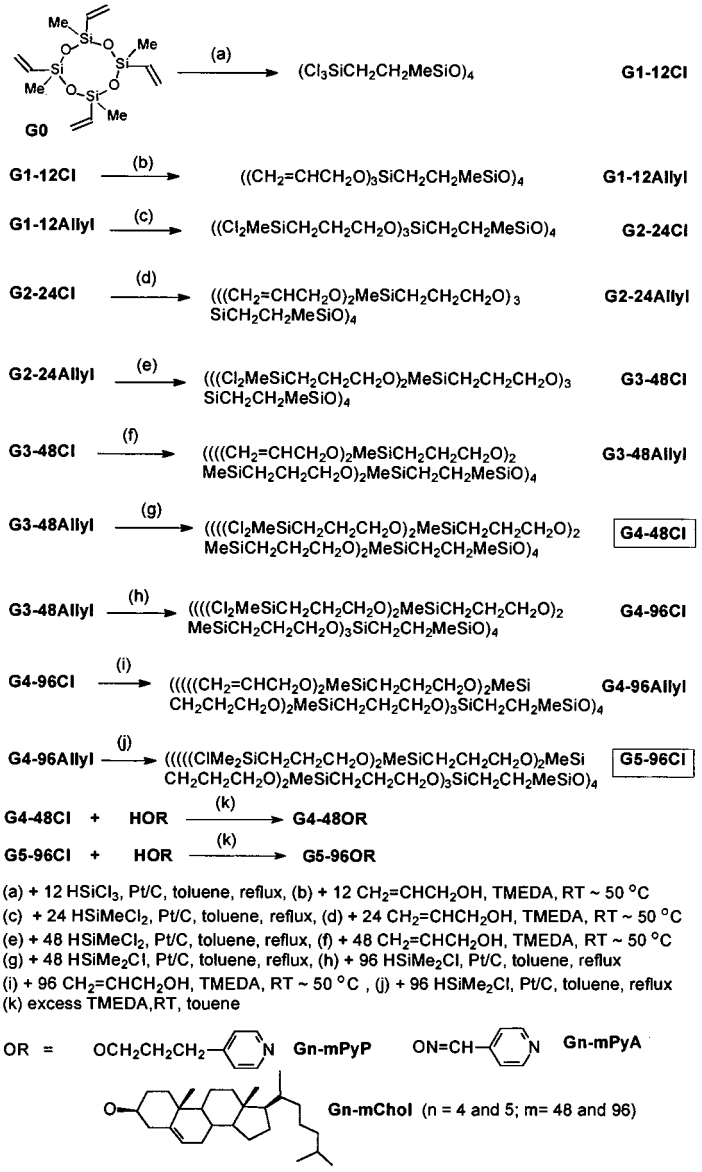
fourth generation (G4-96Allyl) with dimethylchlorosilane, Me_2SiHCl, under refluxed condition in toluene (Scheme 1). The yields of the hydrosilation procedure for all generations were almost quantitative, but the use of THF as a reaction solvent did not serve our purpose because THF could polymerize under the long refluxing condition with a hydrosilation catalyst. The identification of the hydrosilation process of parent dendrimers was obtained by the NMR spectroscopy, which clearly revealed an exchange from allyloxysilyl group containing dendritic branches to propaneoxysilyl branches. The major product in carrying out the hydrosilation process of allyloxy groups in dendritic terminals and chlorosilanes such as dichloromethylsilane and chlorodimethylsilane revealed all β-formed branches ($-OCH_2CH_2CH_2SiMe_nCl_{3-n}$; $n = 1, 2$). The formation of allylsilyloxy bonds from chlorosilyl groups in G*n*-*m*Cl dendritic generations is reported in the previous paper [17].

The reaction of silylated G*n*P-*m*Cl generations in parent dendrimers ($n = 4$, $m = 48$; $n = 5$, $m = 96$) with *p*-pyridinepropanol and *p*-pyridinealdoxyme in excess TMEDA in toluene was converted to the corresponding Si–O–C bonds containing dendrimers on the outermost periphery. The treatment of G4-48Cl with 48 equivalents of *p*-pyridinepropanol in the presence of TMEDA in toluene gave an almost quantitative yield, which was monitored by NMR spectroscopy. But after chromatograpic treatment with alumina there remained 67% yield of G4-48PyP. Under the same condition, the



Scheme 1. Synthetic methods for G*n*-*m*PyP, G*n*-*m*PyA and G*n*-*m*Chol.

treatment of G5-96Cl with 96 equivalents of *p*-pyridinepropanol gave the same result. Naturally, this means the unified dendritic molecule on the periphery is formed on the fifth generation with the 96 *p*-pyridinepropanoxy groups (G5-96PyP) under mild conditions. The terminated dendrimers, G4-48PyA with 48 and G5-96PyA with the rest of 96 *p*-pyridinealdoxime in the terminal layer, were prepared by the use of the same procedure as the previous synthetic method of G*n*-*m*PyP, which gave almost quantitative yields. G4-48Chol and G5-96Chol were prepared by the reaction of parent dendrimers such as G4-48Cl and G5-96Cl with dried cholesterol in the presence of TMEDA in toluene at room temperature. The terminated dendrimers produced almost quantitative yields.

The identification of G4-48PyP, G5-96PyP, G4-48PyA, G5-96PyA, G4-48Chol, and G5-96Chol was obtained by the use of NMR, SEC, UV as well as elemental analysis (Fig. 1). MALDITOF mass spectroscopic treatment of all fourth and fifth generations did not reveal any spectrum. From the UV spectroscopic measurement of G*n*-*m*PyP, G*n*-*m*PyA, G*n*-*m*Chol type dendrimers, the unified character of dendritic macromolecules has been observed. The tendency of the molar absorbitivities (ε_{max}) of the terminated dendrimers was proportional to the number of pyridine groups (Table 1) [25].

Size exclusion chromatography provided additional information on the unified character of each generation. The polydispersity remained almost unchanged in value in going from G4-48PyA, G4-48PyP and G4-48Chol to G5-96PyA, G5-96PyP and G5-96Chol, respectively (M_w/M_n estimated at 1.1–1.2; see Table 1). Therefore, by the use of SEC and UV attachments, the mole mass of the terminated dendrimers (G*n*-*m*PyA, G*n*-*m*PyP and G*n*-*m*Chol) was grossly estimated to structural perfection.

3. Experimental

All reactions were carried out under a dried N_2 atmosphere and THF was dried by sodium benzophenone ketyl, while toluene was dried from 4 Å molecular sieves. The NMR spectra were recorded on a Bruker AC-200 Spectrometer. The UV spectra were measured by an HP 8452A Diode Array UV–Visible Spectrophotometer (HP). THF was used as the solvent, all data were referred to polystyrene standards, and a combination of columns 10^5, 10^4, and 10^3 was employed. The IR spectra were measured by FES 55 (Bruker). The Pusan and Taejon Branches of the Korean Basic Science Institute (KBSI) performed the elemental analysis. The synthetic methods of the lower generations such as G*n* and G*n*P-*m*Cl ($n=1$; $m=12$, $n=2$; $m=2$ etc.) type dendrimers were recorded in previous reports [17].

3.1. G4-48Cl

Molecular weight: 11 385. A mixture of 5.8 g (0.87 mmol) of G3-48A, 5.7 g (60.03 mmol) of $HSiMe_2Cl$, and 0.10 g of a hydrosilation catalyst (Pt/C, 10% content on activated carbon) was refluxed for 24 h in toluene. The catalyst was filtered off and the volatile components were removed under vacuum, leaving 8.7 g (0.77 mmol, 89%) of G4-48Cl, as a colorless oil. Further purification was not available because of sensitivity to moisture. ^{1}H-NMR ($CDCl_3$): $\delta = 0.12$ (s, MeSi (G0), 12H), 0.23 (s, MeSi (G2), 36H), 0.20 (s, MeSi (G3), 72H), 0.41 (s, MeSi (G4), 288H), 0.47–0.72 (m, CH_2 (G0, G1)), 64H), 0.72–0.88 (m, CH_2 (G2), 96H), 1.47–1.77 (m, CH_2 (G3), 192H), 3.56–3.74 (m, OCH_2 (G1–G3), 168H). ^{13}C-NMR ($CDCI_3$): $\delta = 0.43$ (MeSi (G0)), 0.91 (MeSi (G2)), 4.96 (MeSi (G3)), 1.63 (MeSi (G4)), 9.00 (CH_2 (G0)), 9.53, 26.15 (CH_2 (G1–G2)), 14.90, 26.35 (CH_2 (G3)), 64.58 (OCH_2 (G3)), 65.03 (OCH_2 (G1)), 65.31 (OCH_2 (G2)).

3.2. G4-96Cl

Molecular weight: 12 365. The same procedure as that for G4-48Cl was used in the reaction of 8.7 g (1.27 mmol) of G3-48A and 10.25 g (89.10 mmol) of $HSiMeCl_2$, and 0.10 g of a hydrosilation catalyst was refluxed for 24 h in toluene. Yield: 14.00 g (1.13 mmol, 90%) of colorless oil. ^{1}H-NMR ($CDCl_3$): $\delta = 0.07$ (s, MeSi (G0), 12H), 0.18 (s, MeSi (G2), 36H), 0.12 (s, MeSi (G3), 72H), 0.70 (s, MeSi (G4), 144H), 0.41–0.77 (m, CH_2 (G0, G1), 64H), 1.08–1.28 (m, CH_2 (G2), 96H), 1.49–1.85 (m, CH_2 (G3), 192H), 3.54–3.79 (m, OCH_2(G1–G3), 168H). ^{13}C-NMR ($CDCl_3$): $\delta = -1.52$ (MeSi (G0)), 0.39 (MeSi (G2)), -5.02 (MeSi (G3)), 5.16 (MeSi (G4)), 9.02 (CH_2 (G0)), 17.66, 25.95 (CH_2 (G1, G2)), 17.76, 25.95 (CH_2 (G3)), 64.46 (OCH_2 (G1)), 65.18 (OCH_2 (G2)), 63.74 (OCH_2 (G3)).

3.3. G4-96A

Molecular weight: 14 432. A mixed solution of allyl alcohol (8.0 g, 137.62 mmol) and 16.0 g (137 mmol) of TMEDA in toluene (200 ml) was slowly added to G4-96Cl (14.0 g, 1.13 mmol) in toluene (100 ml). After the addition was over, the reaction mixture was warmed to 50 °C for 1 h and the TMEDA·HCl salt was filtered. The volatile components were removed under vacuum, leaving 16 g of a colorless wax. This was chromatographed on silica gel with $CHCl_3$. Yield: 11.21 g (0.77 mmol, 68%) of a colorless wax. ^{1}H-NMR ($CDCl_3$): $\delta = 0.05$ (s, MeSi (G0), 12H), 0.10 (s, MeSi (G2), 36H), 0.08 (s, MeSi (G3), 72H), 0.13 (s, MeSi (G4), 144H), 0.49–0.70, 1.46–1.71 (m, CH_2 (G0–G3), 352H), 3.55–3.69 (m, OCH_2 (G1–G3), 168H), 4.14–4.28 (m, OCH_2 (G4), 192H), 5.04–5.31 (m, =CH_2,

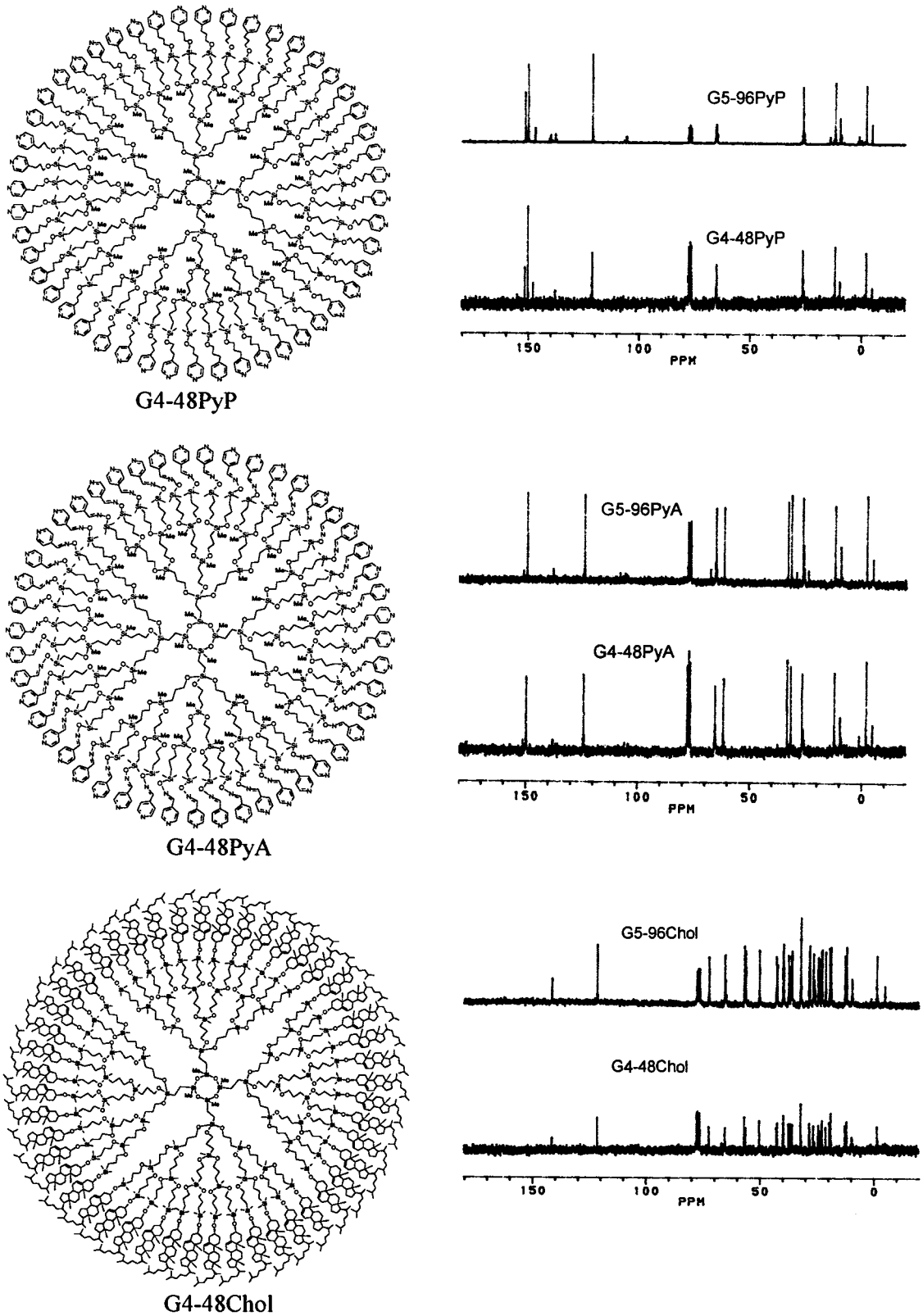


Fig. 1. Planar schematic view of fourth generations and the ^{13}C-NMR spectra of Gn-mPyP, Gn-mPyA and Gn-mChol ($n = 4$; $m = 48$ and $n = 5$; $m = 96$).

192H), 5.79-6.00 (m, HC=, 96H). ^{13}C-NMR ($CDCl_3$): $\delta = 1.42$ (MeSi (G0)), -1.54 (MeSi (G2–G3)), -4.87 (MeSi (G4)), 9.00 (CH_2 (G0)), 25.90, 9.68 (CH_2 (G1–G3)), 63.44 (OCH_2 (G3)), 64.88 (OCH_2 (G2)), 65.32 (OCH_2 (G1)), 114.59, 136.79 (CH=CH_2). GPC: $M_w/M_n = 1.19$, $R_t = 21.0$ min.

3.4. G5-96Cl

Molecular weight: 23 524. The same hydrosilation procedure as for the preparation of G4-48Cl was used. 10.3 g (0.71 mmol) of G4-96Cl, 8.2 g (71.3 mmol) of $HSiMe_2Cl$, and 0.10g of Pt/C in 50 ml toluene were refluxed for 24 h. Yield: 15.08 g (0.64 mmol, 90%) of a colorless wax. ^{1}H-NMR ($CDCl_3$): $\delta = 0.17$ (s, MeSi (G0), 12H), 0.10 (s, MeSi (G2–G4), 252H), 0.40 (s, MeSi (G5), 576H), 0.46–0.75 (m, CH_2 (G0-G2), 160H), 0.75–0.86 (m, CH_2 (G3), 192H), 1.45–1.77 (m, CH_2 (G4), 384H), 3.56–3.77 (m, OCH_2 (G0–G4), 360H). ^{13}C-NMR ($CDCl_3$): $\delta = -1.36$ (MeSi (G0)), -4.90 (MeSi (G2, G3)), 4.99 (MeSi (G4)), -1.60 (MeSi (G5)), 9.00 (CH_2(G0)), 9.50, 25.99 ($SiCH_2$ (G1–G3)), 14.86, 26.11 (CH_2 (G4)), 64.54 (OCH_2 (G4)), 64.76 (OCH_2(G3)), 64.96 (OCH_2(G1)), 65.31 (OCH_2(G2)).

3.5. G4-48PyP

Molecular weight: 16 219. A mixed solution of *p*-pyridinepropanol (0.47 g, 3.42 mmol) and 0.78 g (6.62 mmol) of TMEDA in toluene (50 ml) was slowly added to G4-48Cl (0.68 g, 0.06 mmol) in toluene. After the addition was over, the reaction mixture was warmed to 50 °C for 1 h and the TMEDA·HCl salt was filtered and washed with pentane. The volatile components were removed under reduced pressure, leaving 1.45 g of a colorless wax. This was chromatographed on alumina with THF. Yield: 0.71 g (0.04 mmol, 67%) of a yellow wax. ^{1}H-NMR ($CDCl_3$): $\delta = 0.06$ (s, MeSi (G0–G3), 120H), 0.08 (s, MeSi (G4), 288H), 0.41–0.72, 1.43–1.72 (m, CH_2 (G0–G3), 352H), 3.52-3.79 (m, OCH_2 (G0–G4), 264H), 1.97–1.72, 2.54–2.79 (m, CH_2 (propanoxy), 192H), 7.06–7.18, 8.43–8.59 (m, pyridine, 192H). ^{13}C-NMR ($CDCl_3$): $\delta = -4.98$ (MeSi (G0–G3)), -2.20 (MeSi (G4)), 9.00 (CH_2 (G0)), 9.51, 25.93 (CH_2 (G1–G2)), 11.98, 26.35 (CH_2 (G3)), 65.20 (OCH_2 (G0–G3)), 61.46 (OCH_2 (G4)), 31.44, 33.00 (CH_2 (propanoxy)), 123.91, 149.67, 152.29 (pyridine). GPC: $M_w/M_n = 1.02$, $R_t = 26.84$ min. Anal. Found: C, 56.32; H, 8.41. Calc. for $C_{780}H_{1408}0_{136}Si_{92}N_{48}$: C, 57.75; H, 8.74%. FT-IR (KBr, cm^{-1}): $\nu_{pyridine}$ 1606. UV–Vis: λ_{max} (nm) (ε_{max}, $M^{-1}cm^{-1}$) (EtOH): 256 (0.10×10^6).

3.6. G4-48PyA

Molecular weight: 15 497. The same procedure as the preparative method of G4-48PyP was used in the reaction of 0.68 g (0.06 mmol) of G4-48Cl and 0.86 g (5.85 mmol) of TMEDA, and 0.38 g (3.11 mmol) of *p*-pyridinealdoxime in 50 ml toluene were added slowly. The product was chromatographed on alumina with $CHCl_3$. Yield: 0.56 g (0.04 mmol, 80%) of colorless gel type liquid. ^{1}H-NMR ($CDCl_3$): $\delta = 0.07$ (s, MeSi (G0–G3)), 120H), 0.24 (s, MeSi (G4), 288H), 0.40–0.65, 0.65–0.81, 1.43–1.75 (m, CH_2 (G0–G3), 352H), 3.50–3.81 (m, OCH_2 (G0–G3), 168H), 8.06–8.18 (s, N=CH, 48H), 7.40–7.52, 8.59–8.69 (m, pyridine, 192H). ^{13}C-NMR ($CDCl_3$): $\delta = -5.02$ (MeSi (G0–G3)), -2.36 (MeSi (G4)), 8.98 (CH_2 (G0)), 9.49, 25.98 (CH_2 (G1–G3)), 11.61, 26.09 (CH_2 (G4)), 65.21 (OCH_2 (G1, G2)), 64.81 (OCH_2 (G3)), 64.98 (OCH_2 (G4)), 121.01 (N=CH), 146.99, 150.08, 151.40 (pyridine). FT-IR (KBr, cm^{-1}): $\nu_{C=N,oxime}$ 1665. UV–Vis: λ_{max} (nm) (ε_{max}, $M^{-1}cm^{-1}$) (EtOH): 253 (0.55×10^6). GPC: $M_w/M_n = 1.10$, $R_t = 21.42$ min. Anal. Found: C, 51.52; H, 7.48. Calc. for $C_{684}H_{1168}O_{136}Si_{92}N_{96}$: C, 53.01; H, 7.59%.

3.7. G4-48Chol

Molecular weight: 28 194. The same procedure as for the preparative method of G4-48PyP was used in the reaction of 5.84 g (0.047 mmol) of G4-48Cl, and 0.91 g (2.37 mmol) of cholesterol in 50 ml toluene was added slowly. The product was chromatographed on silica gel with $CHCl_3$. Yield: 0.71 g (0.0025 mmol, 83%) of a

Table 1
GPC and UV spectroscopic data

Dendrimers	M_w	Polydispersity index, PDI	Retention time, R_t (min)	UV spectroscopic data	
				λ_{max} (nm)	$\varepsilon_{max} \times 106$ ($M^{-1}\,cm^{-1}$)
G4-48PyP	16 219	1.02	26.84	256	0.10
G5-96PyP	33 192	1.28	26.28	256	0.18
G4-48PyA	15 497	1.10	21.42	253	0.55
G5-96PyA	31 747	1.11	20.70	254	1.01
G4-48Chol	28 194	1.12	20.54	210	0.085
G5-96Chol	57 142	1.08	19.12	210	0.177

colorless wax. ^{1}H-NMR ($CDCl_3$): $\delta = 0.06$ (s, MeSi (G0–G3), 120H), 0.10 (s, MeSi (G4), 288H), 0.47–0.65, 1.43–1.72 (m, CH_2, 352H), 3.51–3.73 (s, OCH_2 (G1–G3), 168H), 0.65–2.34 (m, cholesteroxy rest, 2160H). ^{13}C-NMR ($CDCl_3$): $\delta = -4.90$ (MeSi (G0–G3)), -1.44 (MeSi (G4)), 12.56, 26.47 (CH_2 (G3)), 9.51, 25.96 (CH_2 (G1, G2)), 9.04 (CH_2 (G0)), 65.27 (OCH_2 (G3)), 65.28 (OCH_2 (G2)), 65.54 (OCH_2 (G1)), 11.8, 18.7, 19.3, 21.0, 22.5, 22.8, 23.8, 24.2, 25.6, 28.0, 28.2, 31.9, 32.0, 35.7, 36.1, 36.5, 37.3, 39.5, 39.7, 42.3, 42.7, 50.1, 56.1, 56.7, 72.3, 121.3, 141.2 (cholesteroxy rest). IR (KBr, cm^{-1}): $\nu_{C=C}$ 1634. UV–Vis: λ_{max} (nm) (ε_{max}, $M^{-1}cm^{-1}$) (THF): 210 (-8.51×10^4). GPC: $M_w/M_n = 1.12$, $R_t =$ 20.54 min. Anal. Found: C, 71.42; H, 11.27. Calc. for ($C_{1692}H_{3088}O_{136}Si_{92}$): C, 72.08; H, 11.03%.

3.8. G5-96PyP

Molecular weight: 33 192. The same procedure as the preparative method of G4-48PyP was used in the reaction of 2.26 g (0.096 mmol) of G5-96C1, 1.37 g (9.98 mmol) of *p*-pyridine propanol and 1.92 g (16.56 mmol) of TMEDA in 100 ml toluene. The product was chromatographed on alumina with the THF–diethylamine (5:1) mixed solvent. Yield: 1.60 g (0.048 mmol, 50%) of yellow gel type liquid. ^{1}H-NMR ($CDCl_3$): $\delta = 0.07$ (s, MeSi (G0–G4), 264H), 0.14 (s, MeSi (G5), 576H), 0.32–0.70, 1.41–1.71 (m, CH_2(G0–G4), 736H), 3.42–3.75 (m, OCH_2 (G0–G5), 552H), 1.71–1.93, 2.57–2.74 (m, CH_2, 384H), 7.02–7.15, 8.40–8.55 (m, pyridine, 384H). ^{13}C-NMR ($CDCl_3$): $\delta = -4.96$ (MeSi (G0–G4)), -2.19 (MeSi (G5)), 9.00 (CH_2 (GO)), 9.53, 26.24 (CH_2(G1–G3)), 11.99, 26.35 (CH_2 (G4)), 67.57 (OCH_2 (G1–G4)), 61.47 (OCH_2 (G5)), 31.44, 33.00 (CH_2), 123.91, 149.54, 151.21 (pyridine). FT-IR (KBr, cm^{-1}): $\nu_{pyridine}$ 1608. UV–Vis: λ_{max} (nm) (ε_{max}, $M^{-1}cm^{-1}$) (EtOH): 256 (1.80×10^5). GPC: $M_w/M_n = 1.28$, $R_t =$ 26.28 min. Anal. Found: C, 56.55; H, 8.64. Calc. for $C_{1596}H_{2896}O_{280}Si_{188}N_{96}$: C, 57.75; H, 8.79%.

3.9. G5-96PyA

Molecular weight: 31 747. The same procedure as the preparative method of G4-48PyA was used in the reaction of 2.26 g (0.1 mmol) of G5-96C1, 1.11 g (9.08 mmol) of *p*-pyridinealdoxime and 1.92 g (16.5 mmol) of TMEDA in 100 ml toluene. The product was chromatographed on silica gel with $CHCl_3$. Yield: 2.39 g (0.075 mmol, 78%) of yellow gel type liquid. ^{1}H-NMR ($CDCl_3$): $\delta = 0.07$ (s, MeSi (G0–G4), 264H), 0.24 (s, MeSi (G5), 576H), 0.43–0.65, 0.65–0.86, 1.45–1.86 (m, CH_2 (G0–G4), 384H), 3.47–3.84 (m, OCH_2 (G1–G4), 360H), 8.52–8.63 (m, N=CH, 96H), 7.36–7.50, 8.05–8.13 (m, pyridine, 384H). ^{13}C-NMR ($CDCl_3$): $\delta = -4.98$ (MeSi (G0–G4)), -2.32 (MeSi (G5)), 9.00 (CH_2 (G0)), 9.50, 25.99 (CH_2 (G1–G3)), 11.61, 26.09 (CH_2 (G4)), 64.99 (OCH_2 (G1–G5)), 120.94 (N=CH), 147.03, 150.06, 151.40 (pyridine). IR (KBr, cm^{-1}): $\nu_{C=N,\ oxime}$ 1664. UV–Vis: λ_{max} (nm) (ε_{max}, $M^{-1}cm^{-1}$) (EtOH): 254 (1.08×10^6). GPC: $M_w/M_n = 1.11$, $R_t = 20.70$ min. Anal. Found: C, 52.23; H, 7.79. Calc. for $C_{1404}H_{2416}O_{280}Si_{188}N_{192}$: C, 53.11; H, 7.67%.

3.10. G5-96Chol

Molecular weight: 57 142. The same procedure as the preparative method of G4-48Chol was used in the reaction of 1.51 g (0.064 mmol) of G4-48C1, 2.40 g (6.21 mmol) of cholesterol and 1.41 g (12.1 mmol) of TMEDA in 100 ml toluene. The product was chromatographed on silica gel with $CHCl_3$. Yield: 2.48 g (0.043 mmol, 67%) of yellow gel type liquid. ^{1}H-NMR ($CDCl_3$): $\delta = 0.09$ (s, MeSi (G0–G5), 840H), 0.40–0.62, 0.62–0.86, 1.45–1.86 (m, CH_2 (G0–G4), 384H), 4.45–4.72 (m, OCH_2 (G1–G4), 360H), 0.62–2.40 (m, cholesterol rest, 4320H). ^{13}C-NMR ($CDCl_3$): $\delta = -4.90$ (MeSi (G0–G4)), -1.39 (MeSi (G5)), 9.13 (CH_2 (G0)), 9.50, 26.06 (CH_2 (G1–G3)), 12.59, 26.48 (CH_2 (G4)), 65.29 (OCH_2 (G1–G4)), 11.8, 18.7, 19.4, 21.0, 22.5, 22.8, 25.7, 26.0, 28.0, 28.2, 31.9, 32.0, 35.8, 36.2, 36.5, 37.3, 3.5, 42.3, 42.7, 50.1, 56.1, 56.7, 65.1, 65.2, 112.3, 121.3, 141.2 (cholesterol rest)). IR (KBr, cm^{-1}): $\nu_{C=C}$ 1664. UV–Vis: λ_{max} (nm) (ε_{max}, $M^{-1}cm^{-1}$) (THF): 210 (1.77×10^5). GPC: $M_w/M_n = 1.08$, $R_t = 19.21$ min. Anal. Found: C, 70.15; H, 11.03. Calc. for $C_{3420}H_{6256}O_{280}Si_{188}$: C, 71.88; H, 11.03%.

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