

**Российская академия наук
Институт синтетических полимерных материалов им. Н.С. Ениколопова**

А.М. Музафаров

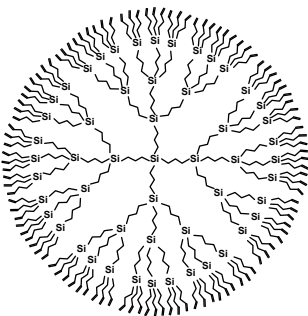
**"Дендримеры и другие макромолекулы-частицы как
объекты нанотехнологий и наноматериалов"**



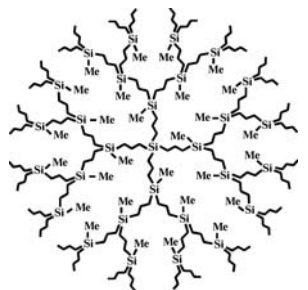
Макромолекулярные нанобъекты

Дендримеры

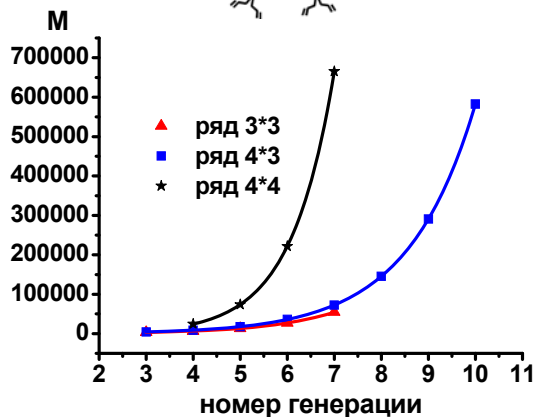
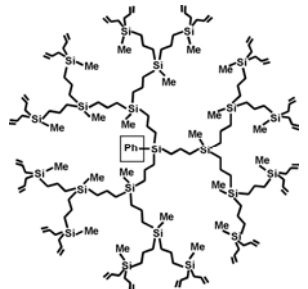
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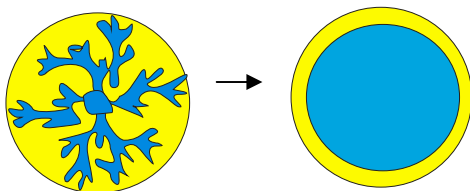
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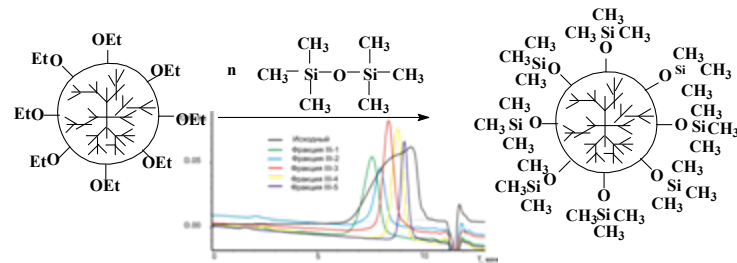
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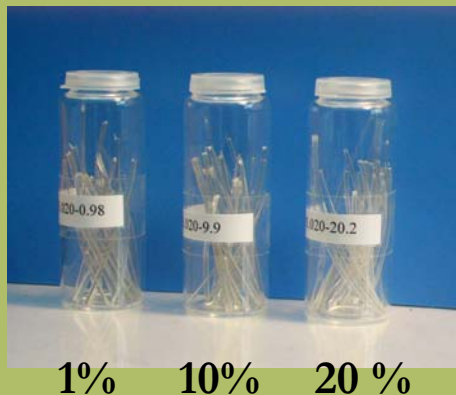
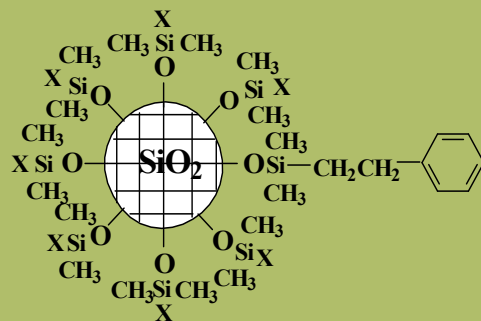
От макромолекулы к частице



Модельные гибридные кремнеземы

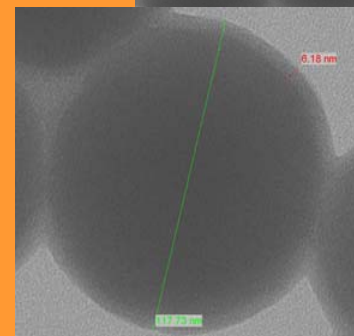
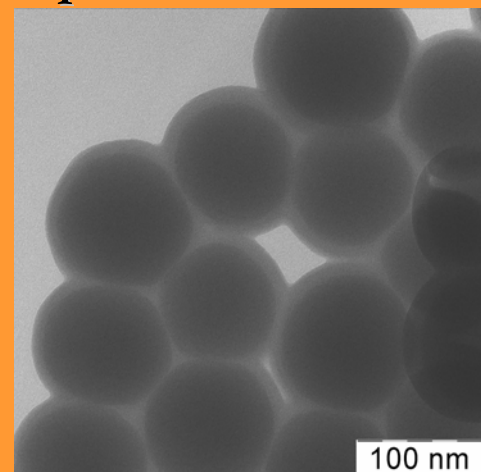


Полимерные нанокompозиты



1% 10% 20%

Кремнеземы с функциональным поверхностным слоем





Series Editor J. Matison
Volume Editors P. Dvornic · M. J. Owen

Silicon-Containing Dendritic Polymers

HIGHLIGHT

From the Discovery of Sodioxyorganoalkoxysila to the Organosilicon Dendrimers and Back



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ABSTRACT: Upon being discovered 20 years ago, sodioxyorganoalkoxysilanes became the key to the world of organoelement dendrimers. Even considering the great variety of objects that had appeared in this area during the last 20 years, the organosilicon dendrimers are still one of the most actual objects in this class. Above all, this is fair concerning the carbosilane systems. The high reactivity of the functional groups, the well controlled chemistry of their transformations, and the inertness of the molecular skeleton are the reason for making these systems highly actual in two main regards: as model objects for the deep research of the dendrimers' properties and as polyfunctional matrixes for numerous derivatives. In this review, we were mainly focusing on the importance of the former part. In the conclusion, we showed the motivation for further development of this area both in regard of synthesis of new carbosilane systems and further development of siloxane dendrimers. © 2008 Wiley Periodicals, Inc. *J Polym Sci Part A: Polym Chem* 46: 4935–4948, 2008

Keywords: dendrimers; glass transition; heteroatom-containing polymers; hyperbranched; networks; polysiloxanes



AZIZ M. MUZAFAROV

Professor A.M. Muzafarov graduated from Andrianov Chair of Moscow Institute of Fine Chemical Technology in 1973, obtained his Ph.D. in 1981 with Professor A.A. Zhdanov, on the subject of high functional organosilicon oligomers and thermostable polymers. He achieved his Dr. of Chemistry degree in 1997 on the subject of Organosilicon dendrimers and hyperbranched systems. In 1998 together with E.A. Rebrov, he received S.V. Lebedev Award of Academy Sciences of Russia for the investigation of organosilicon dendrimers and hyperbranched systems. From 1996 up till now he is the Head of the



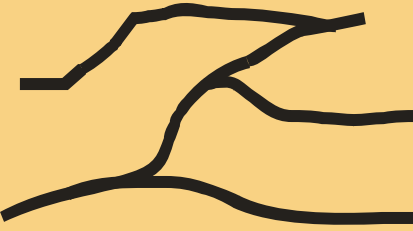
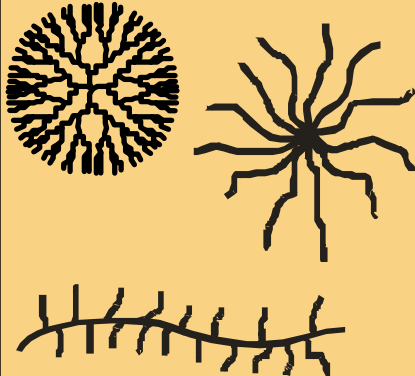
Correspondence to: A. M. Muzafarov (E-mail: a.m.muz@ipm.ru)
Journal of Polymer Science: Part A: Polymer Chemistry, Vol. 46, 4935–4948 (2008)
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Polymer Data Handbook

Second Edition

EDITED BY James E. Mark

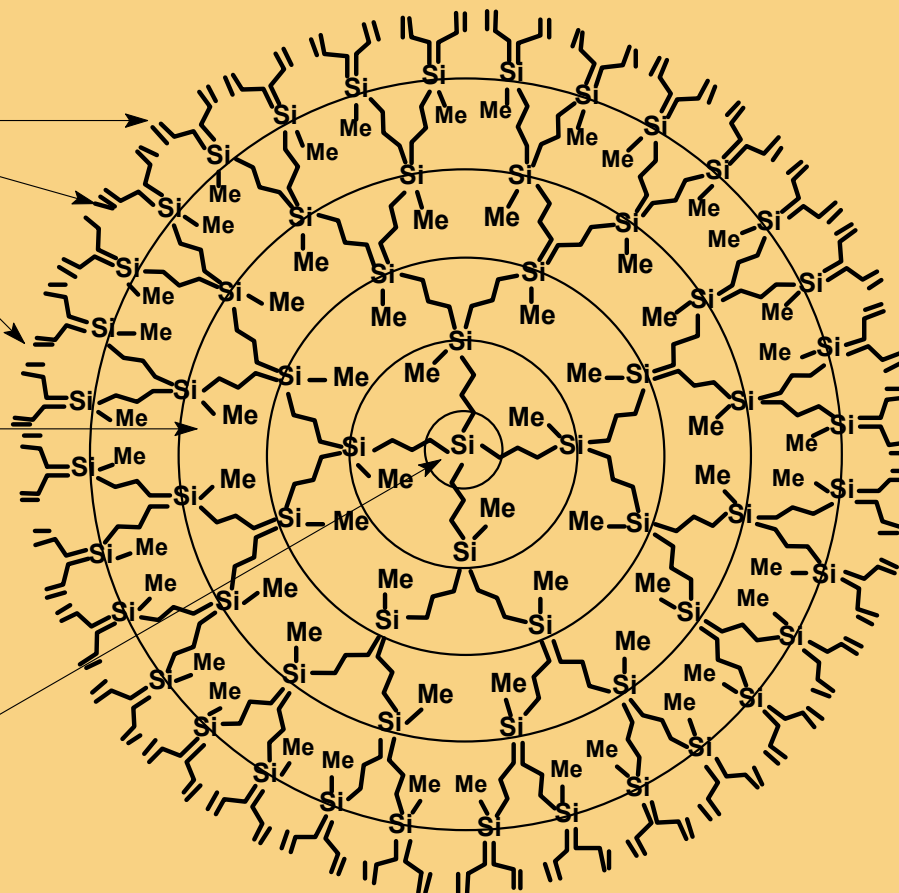
Классификация полимеров по структуре основной цепи

I линейные	II сетчатые	III разветвленные	IV макромолекулы- частицы
			
			<ul style="list-style-type: none">• Дендримеры• Сверхразветвленные полимеры• Молекулярные щетки• Многолучевые звезды• Нано- и микрогели

ПОВЕРХНОСТНЫЙ
СЛОЙ

ВНУТРЕННИЙ
МОЛЕКУЛЯРНЫЙ
СЛОЙ

ЯДРО



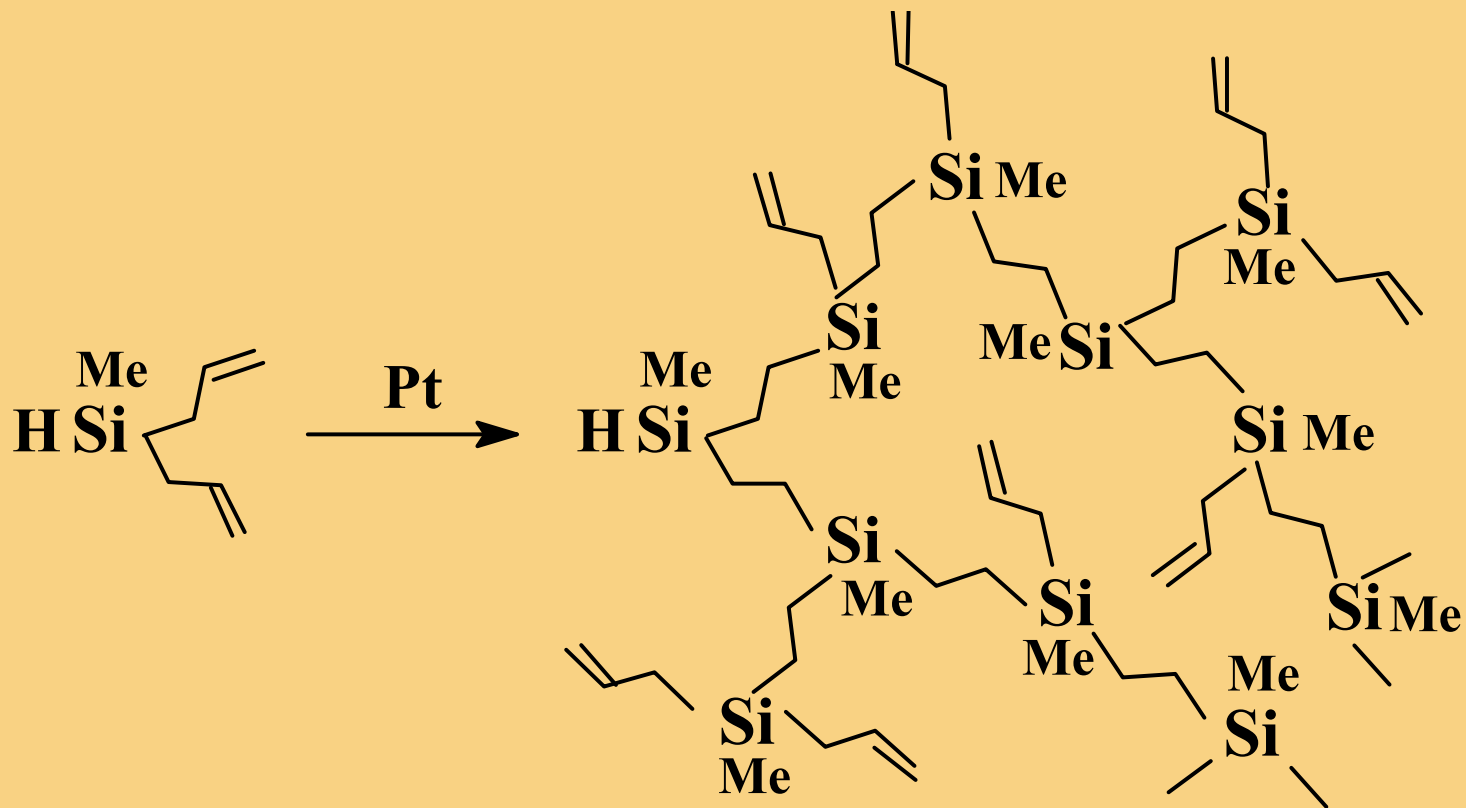
Граничная поверхность:

$$S=4\pi r^2$$

Количество функциональных групп:

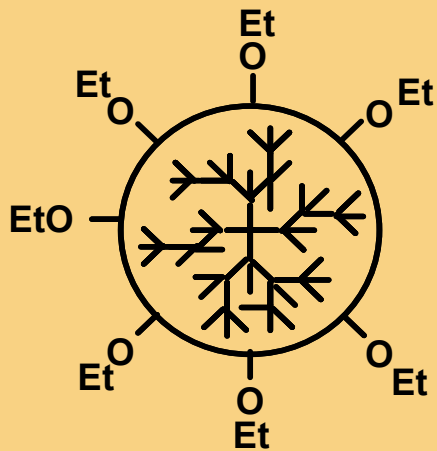
$$Z=N_c * N_b^G,$$

Сверхразветвленные полиаллилкарбосиланы

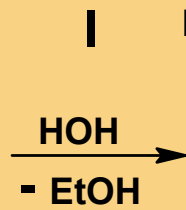


Inorganic dendritic structure

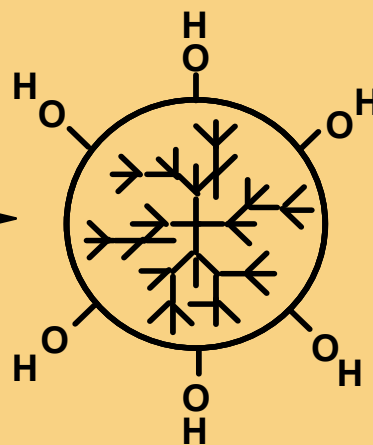
Hydrolysis



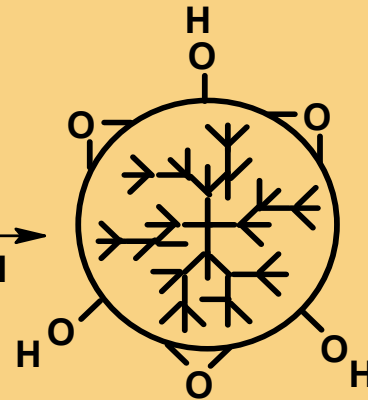
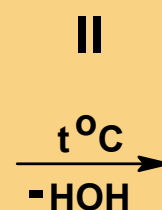
Hyperbranched
ethylsilicate



Polycondensation
(Polycyclization)



Product of hydrolysis



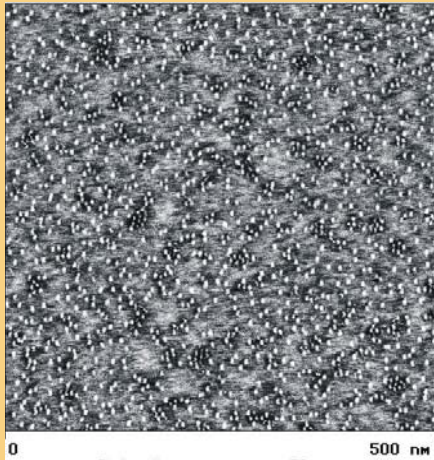
Silica-sol
soluble in water free THF

Solvent removing

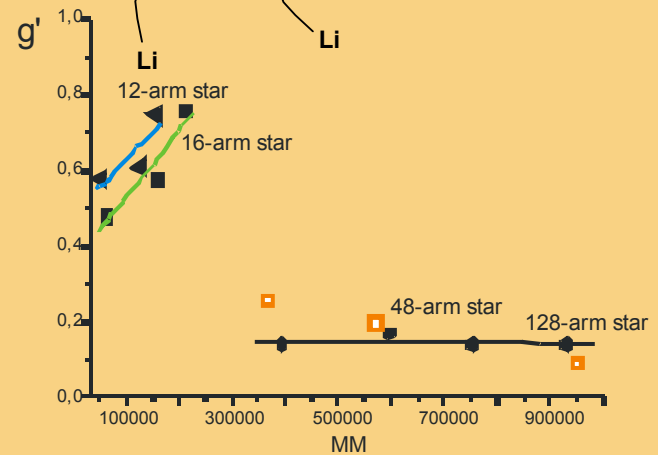
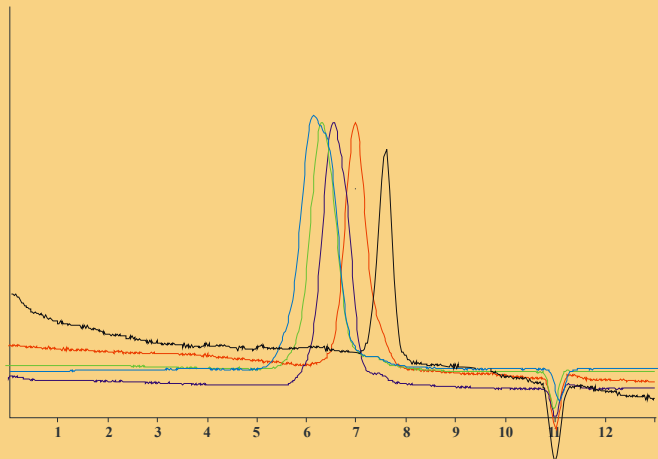
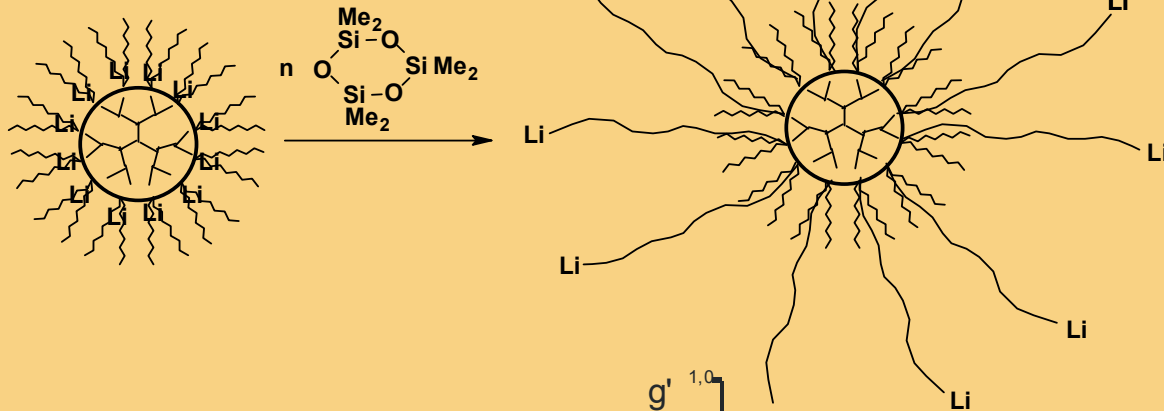
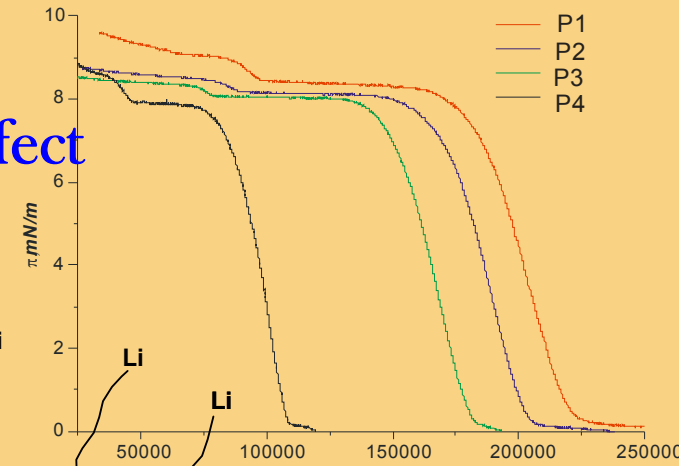


SILICA

Silica
particles



Molecular Membrane Effect



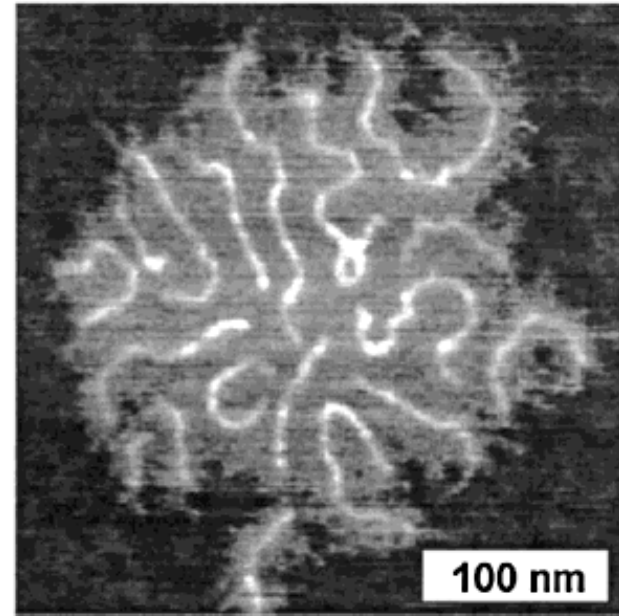
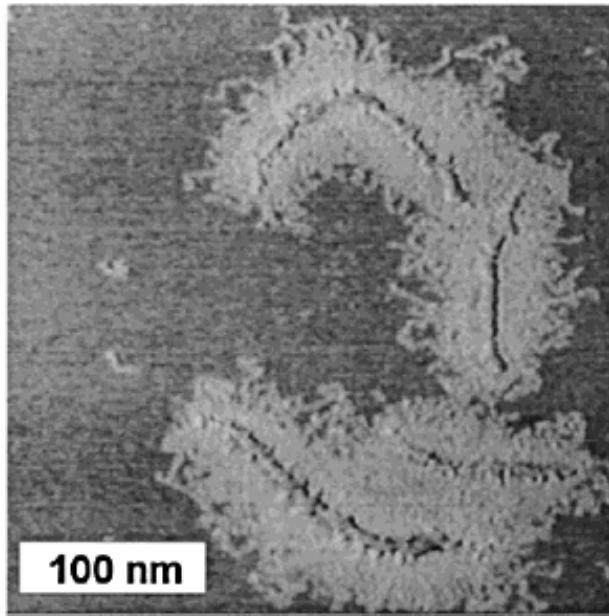
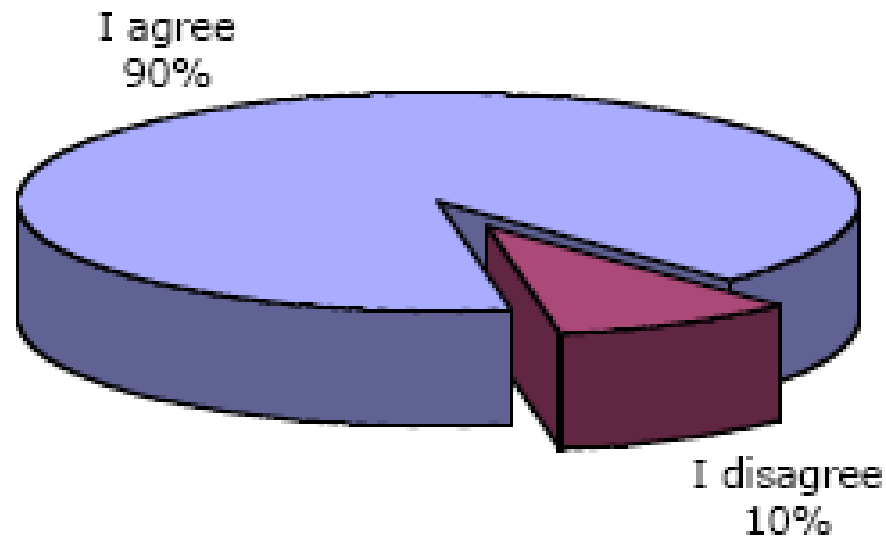
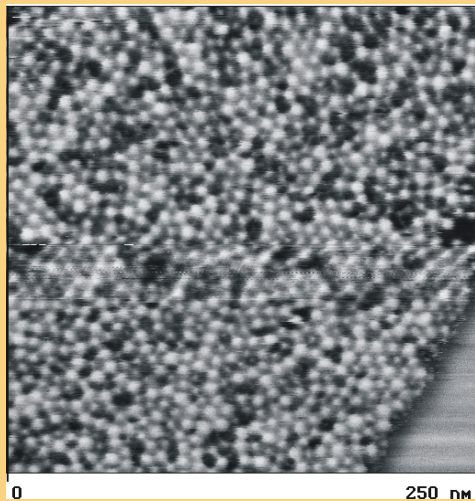


Figure 14. Molecular brushes with poly(*n*-butyl acrylate) side chains of different degrees of polymerization: (a) $n = 52$ and (b) $n = 10$.

Dendrimers can be considered the most versatile, compositionally & structurally controlled synthetic nanoscale building blocks available today

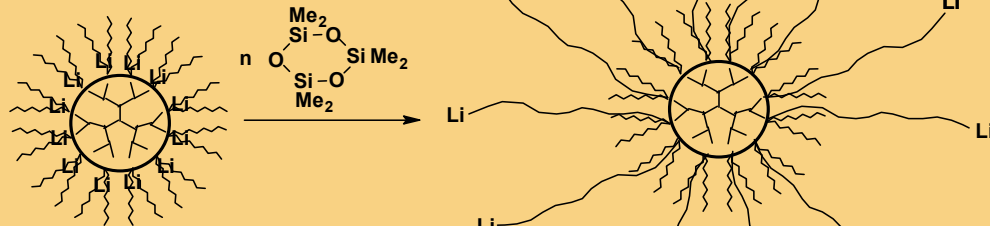




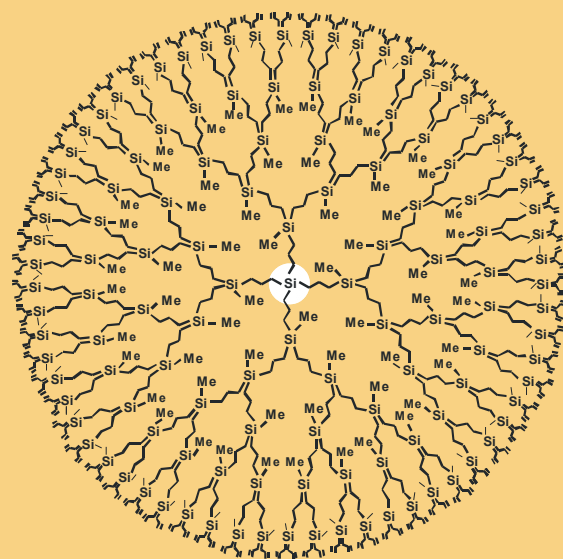
STM image of the 9-th generation of Carbosilane dendrimer on mica



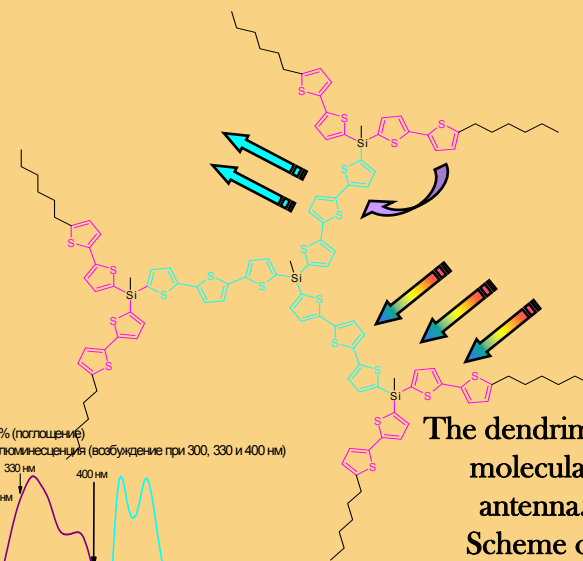
Hexane solution of carbosilane dendrimer
Filled by cobalt oxide(II)



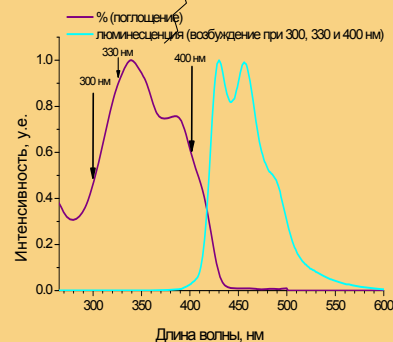
Multifunctional polyolithium (12-128)
Initiators of anionic polymerization



Carbosilane dendrimer



The dendrimer -
molecular
antenna.
Scheme of
photons
transformations,
left - absorption
and luminescence
spectra



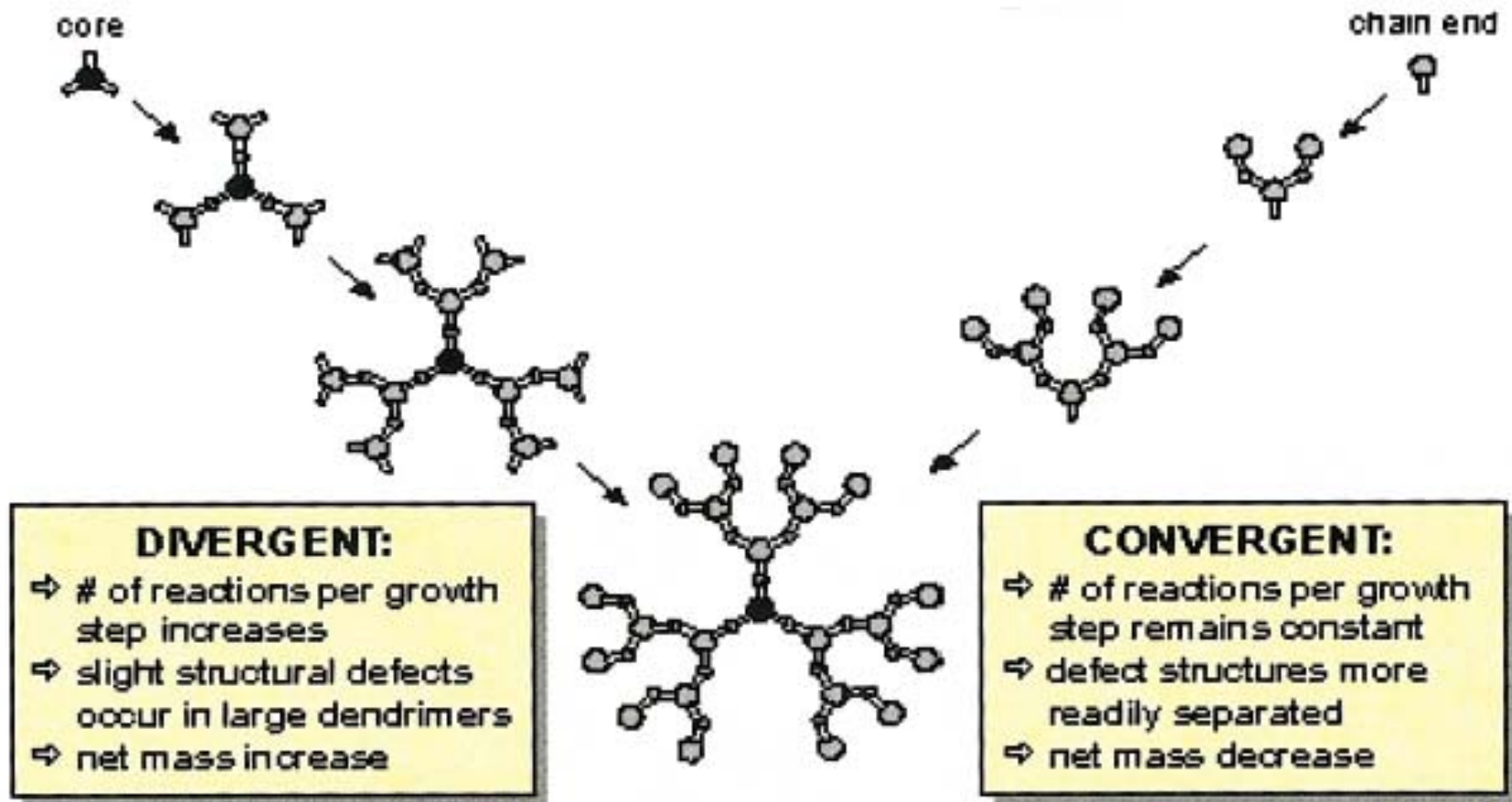
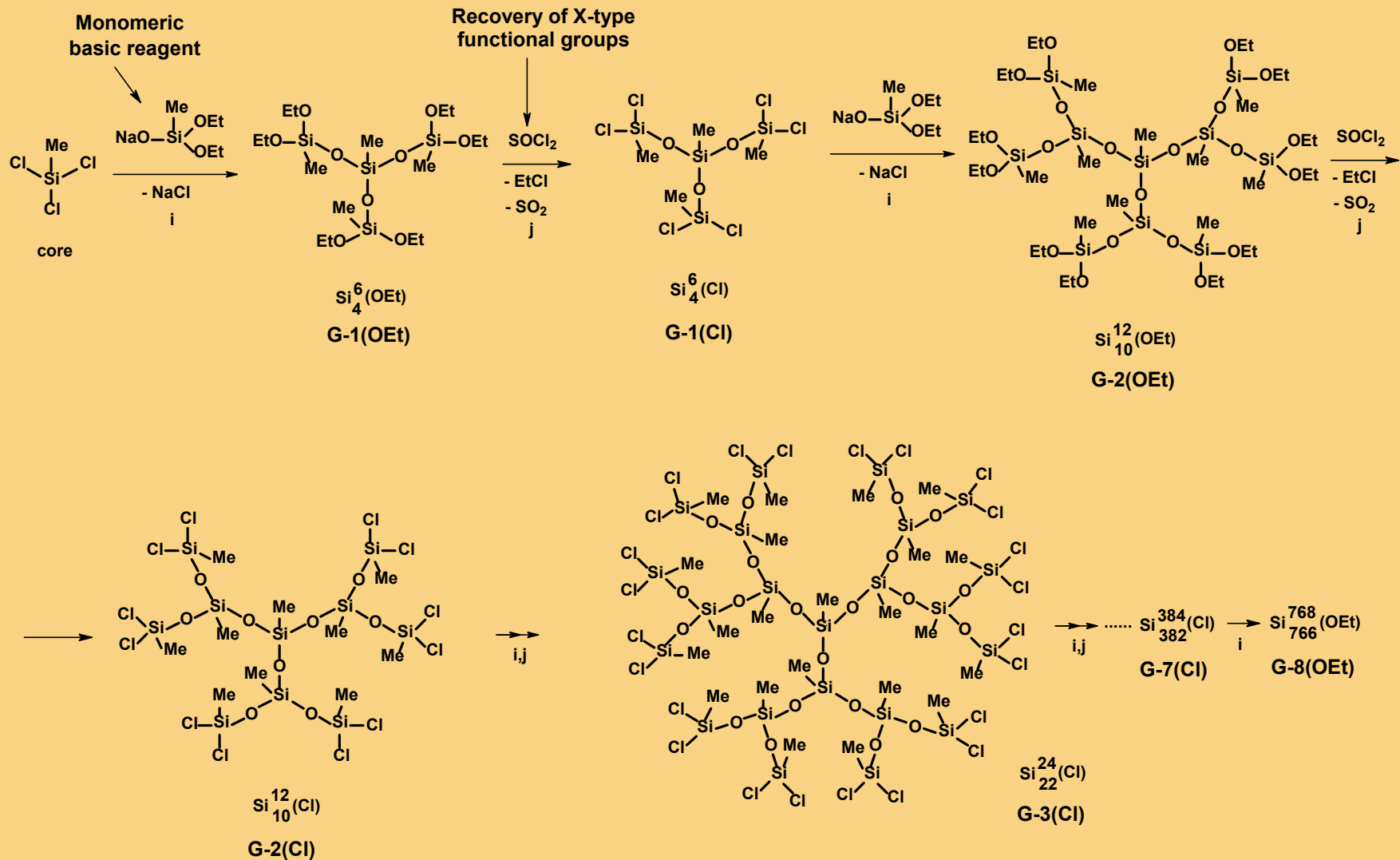


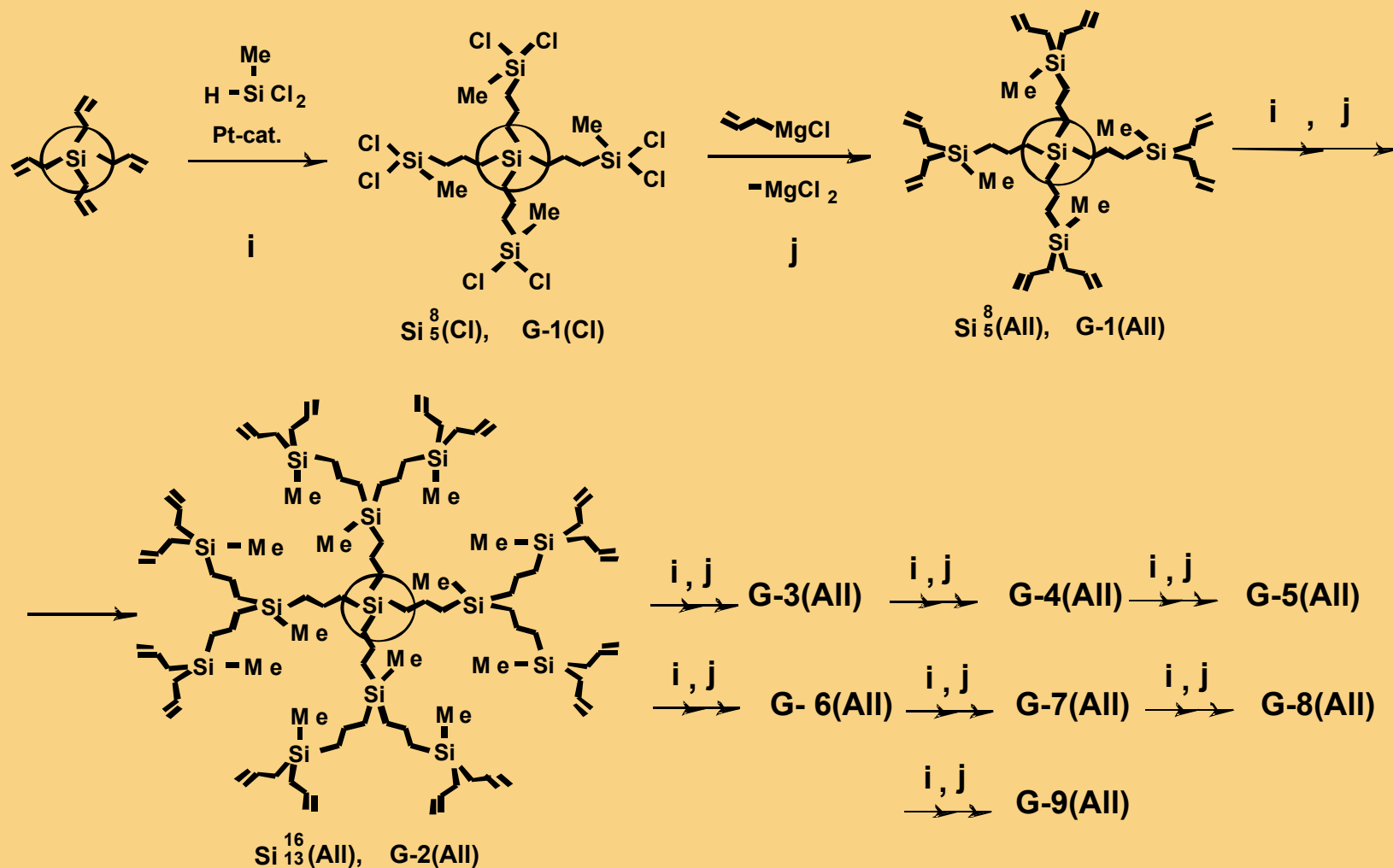
Figure 5. Representation of dendrimer growth by the divergent and convergent methods.



E.A.Rebrov, A.M.Muzafarov, V.S.Papkov, A.A.Zhdanov
Volume-groving polyorganosiloxanes

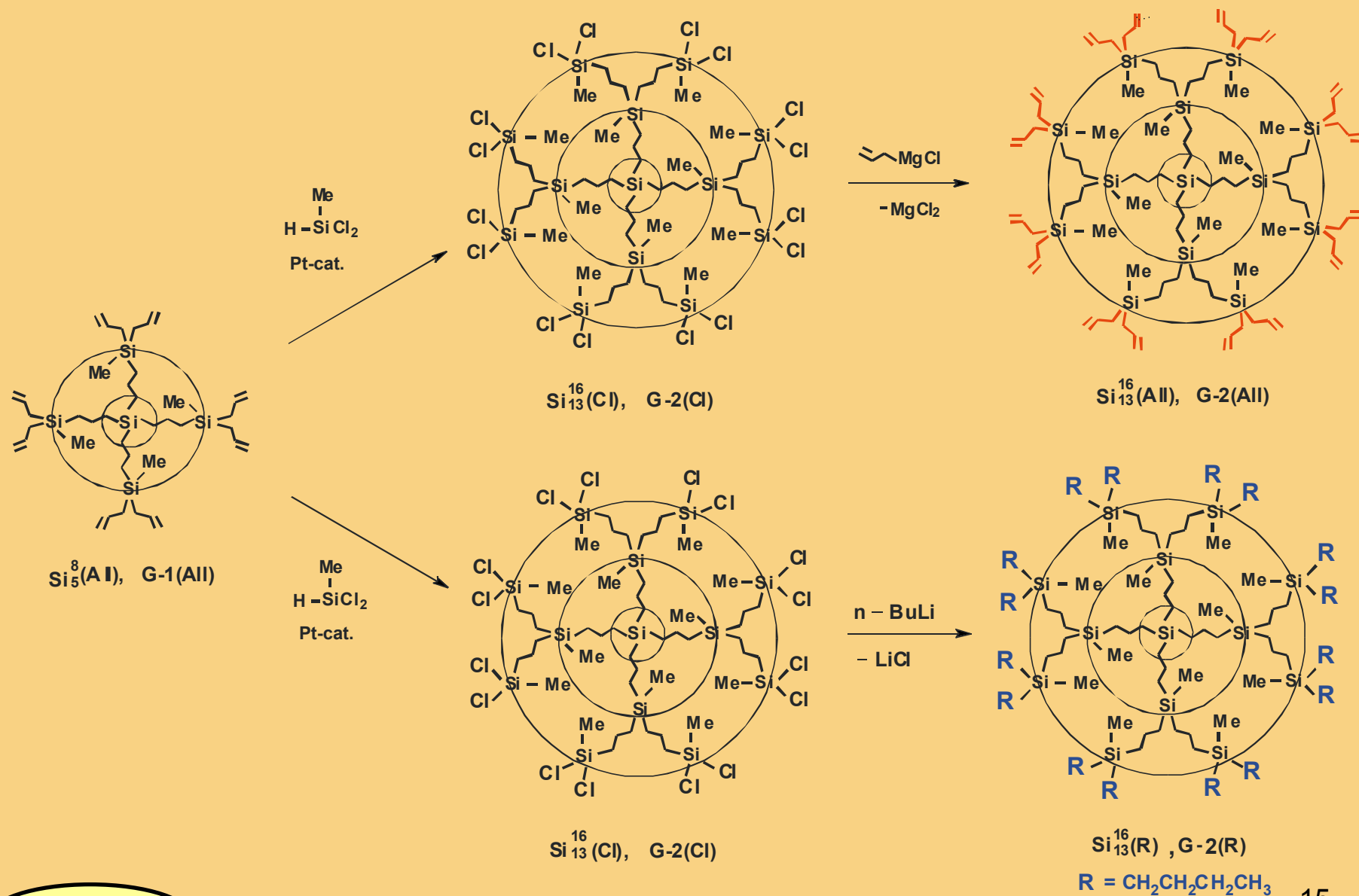
Doklady Chemistry, USSR, 1989, v. 309, №2, p. 376

Divergent scheme of carbosilane dendrimers synthesis

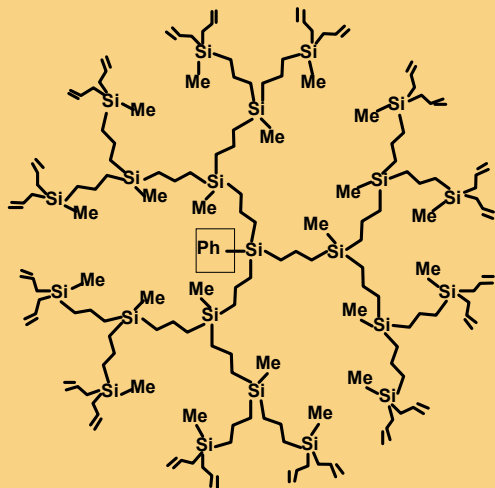


A.Muzafarov, O.B.Gorbatsevich, E.A. Rebrov, G.M.Ignat'eva, T.B.Chenskaya, V.D.Myakouchev,
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 Science, 1993, vol. 35A, No 11, p.1867.

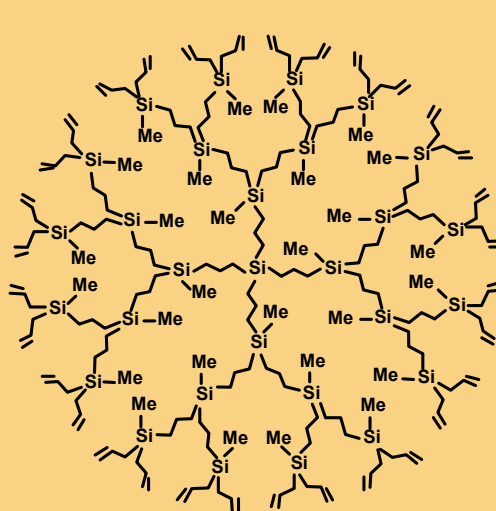
Synthesis of functional and non-functional derivatives of carbosilane dendrimers



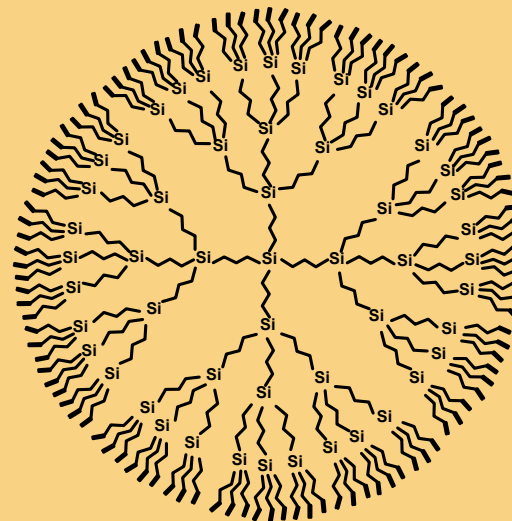
Гомологические ряды карбосилановых дендримеров



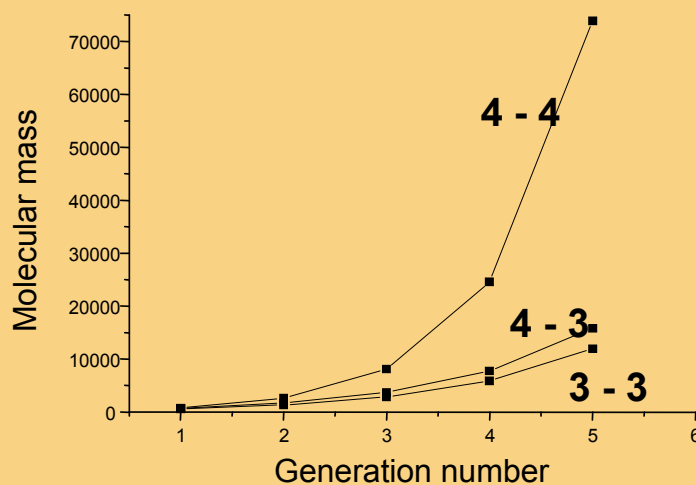
3 - 3



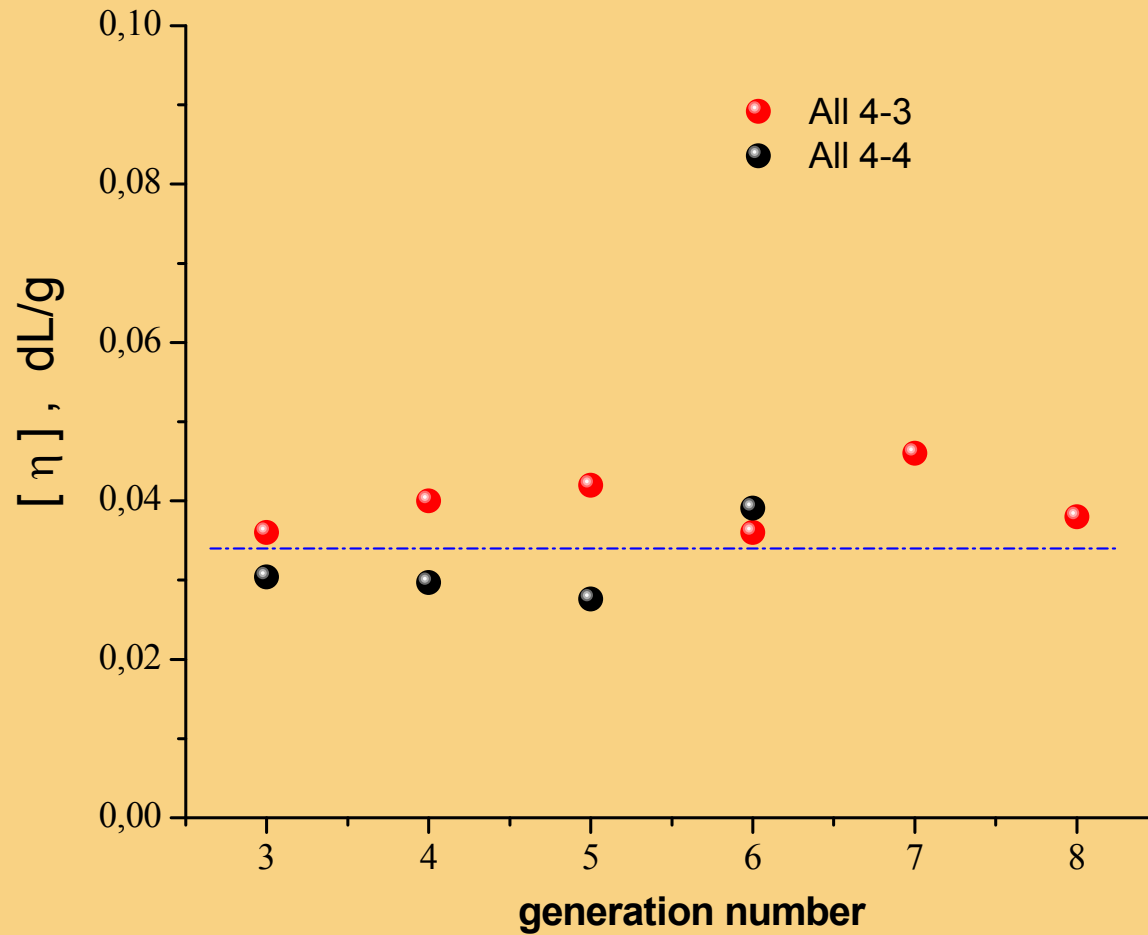
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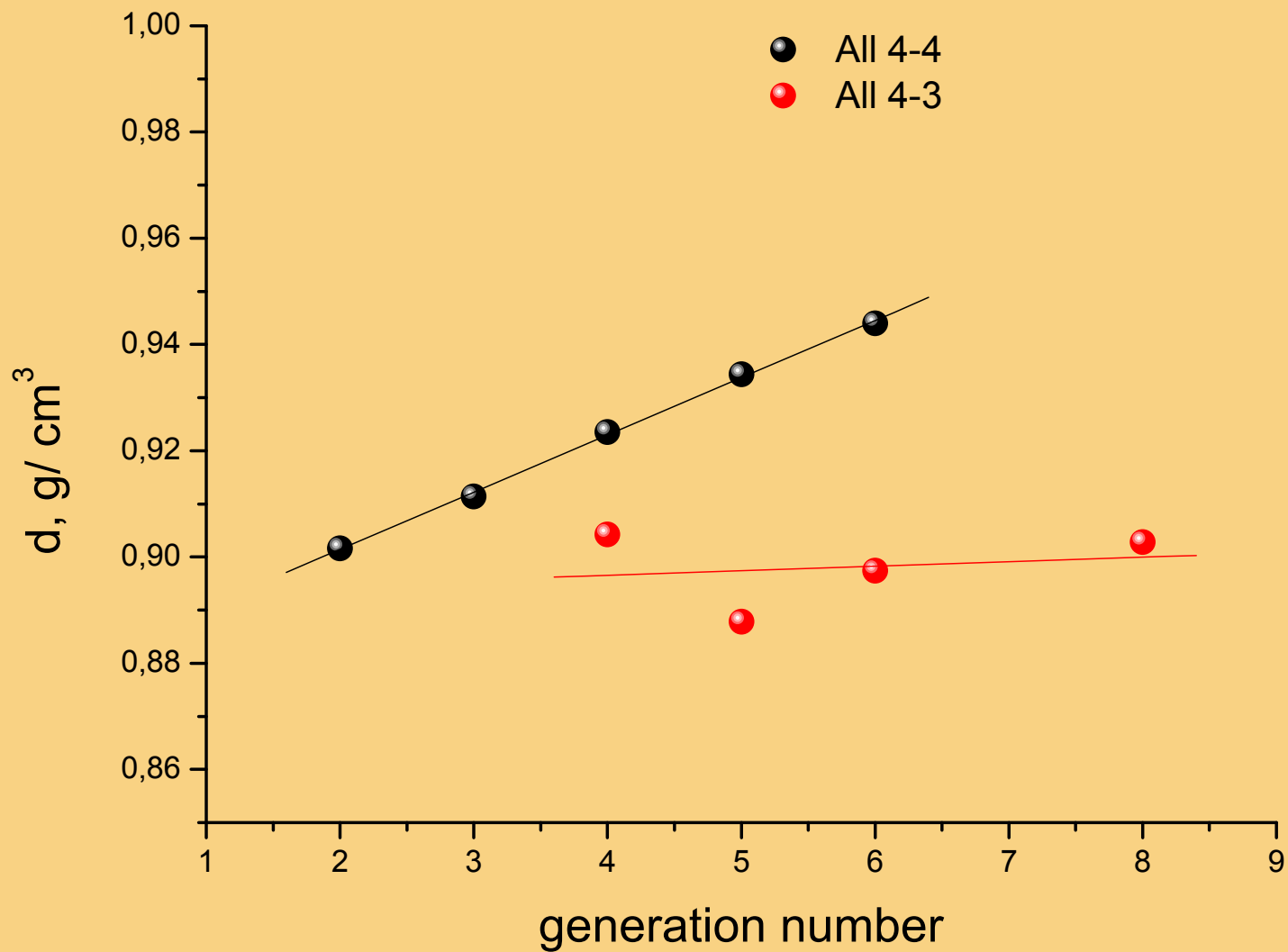
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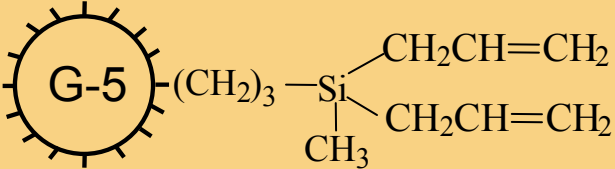
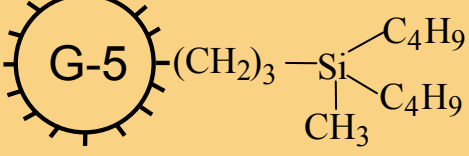
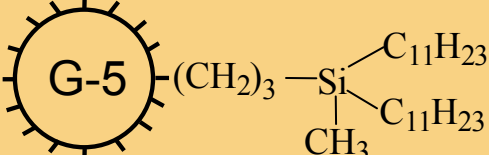
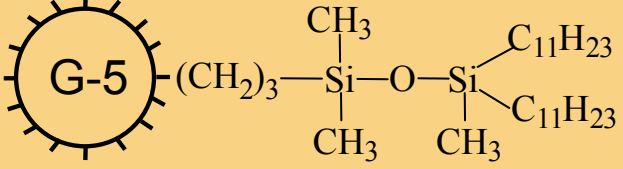
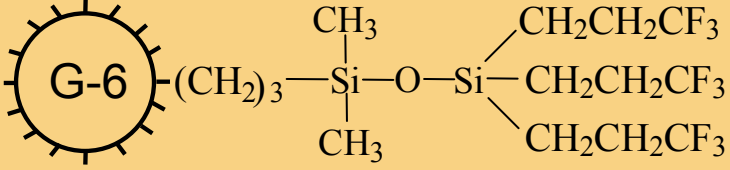
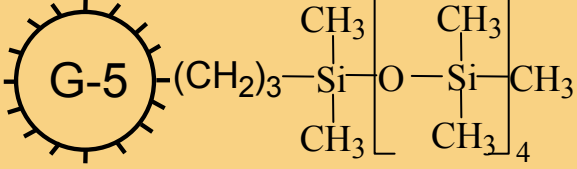


Зависимость характеристической вязкости растворов карбосилановых дендримеров

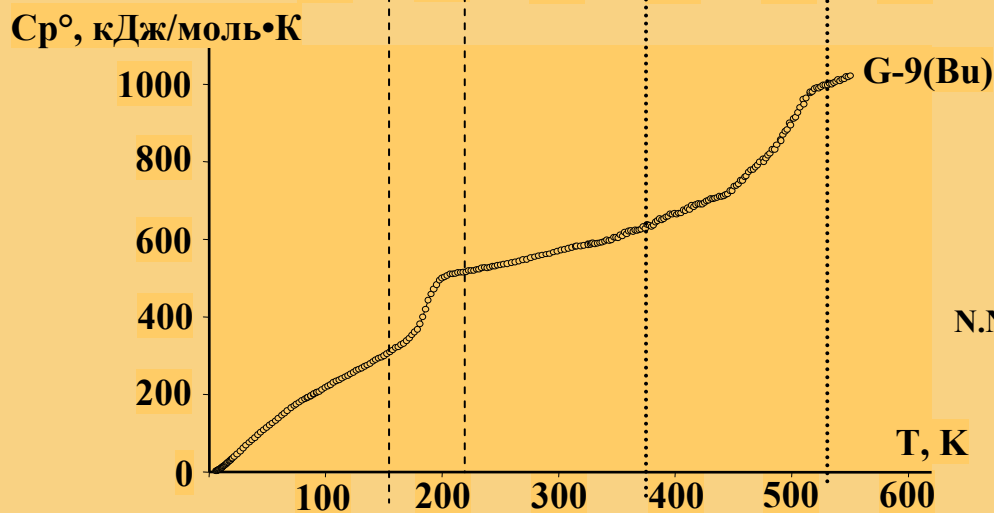
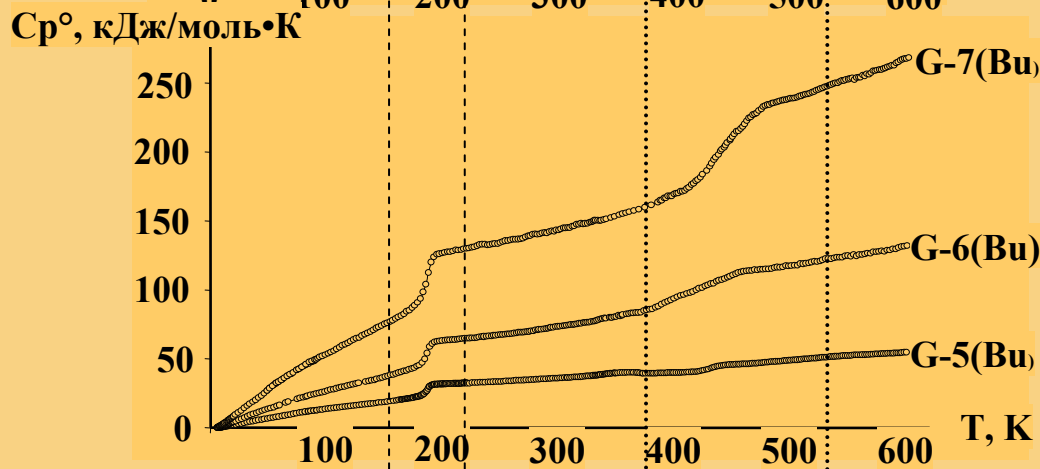
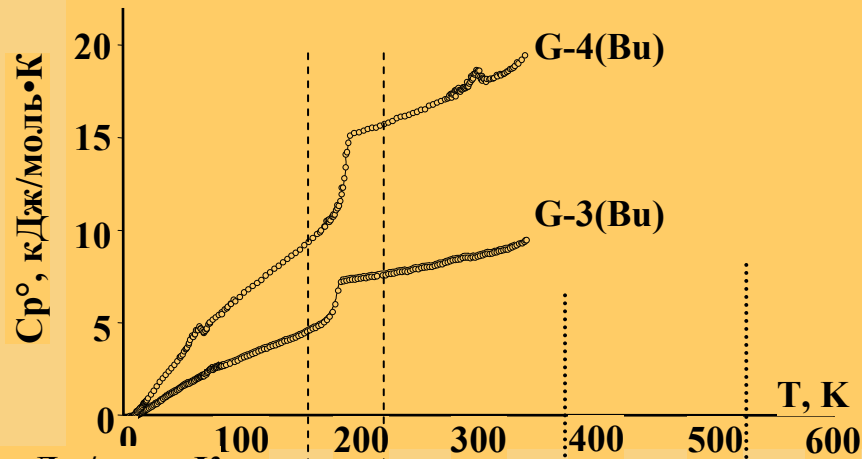


Зависимость плотности карбосилановых дендримеров от номера поколения



Dendrimers	$T_g, ^\circ\text{C}$
 <p>Chemical structure of G-5 dendrimer: A central silicon atom is bonded to a (CH₂)₃ group, a methyl group (CH₃), and two vinyl groups (CH₂CH=CH₂).</p>	- 93±1
 <p>Chemical structure of G-5 dendrimer: A central silicon atom is bonded to a (CH₂)₃ group, a methyl group (CH₃), and two butyl groups (C₄H₉).</p>	- 87 ± 1
 <p>Chemical structure of G-5 dendrimer: A central silicon atom is bonded to a (CH₂)₃ group, a methyl group (CH₃), and two dodecyl groups (C₁₁H₂₃).</p>	<p style="text-align: center;">Tail wags the dog...</p> <p style="text-align: center;">-64 ± 1</p>
 <p>Chemical structure of G-5 dendrimer: A central silicon atom is bonded to a (CH₂)₃ group, two methyl groups (CH₃), and an oxygen atom. The oxygen atom is bonded to another silicon atom, which is bonded to two methyl groups (CH₃) and two dodecyl groups (C₁₁H₂₃).</p>	-76 ± 3
 <p>Chemical structure of G-6 dendrimer: A central silicon atom is bonded to a (CH₂)₃ group, two methyl groups (CH₃), and an oxygen atom. The oxygen atom is bonded to another silicon atom, which is bonded to three trifluoroethyl groups (CH₂CH₂CF₃).</p>	-50 ± 3
 <p>Chemical structure of G-5 dendrimer: A central silicon atom is bonded to a (CH₂)₃ group, two methyl groups (CH₃), and an oxygen atom. The oxygen atom is bonded to a silicon atom that is part of a chain of four methyl groups (CH₃)₄.</p>	-106.5 ± 1

Зависимость теплоемкости от температуры образцов полибутилкарбосилановых дендримеров 3-9 генераций

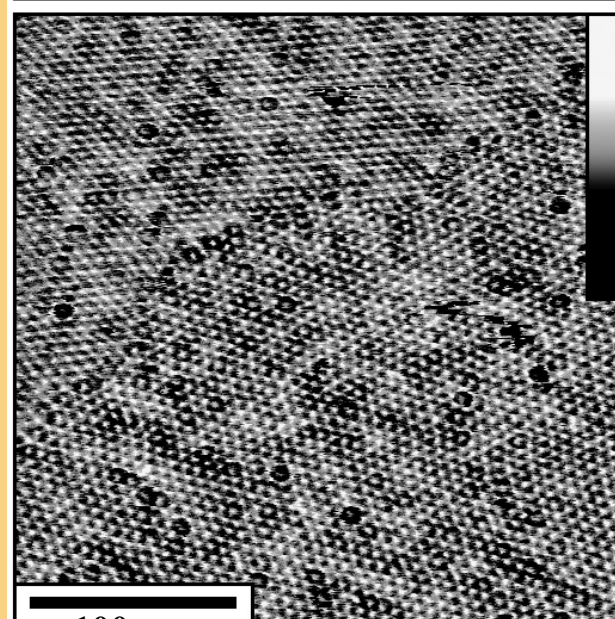
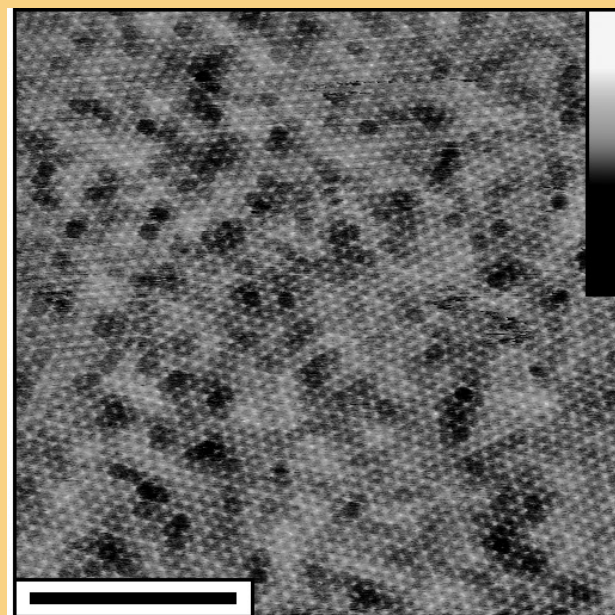
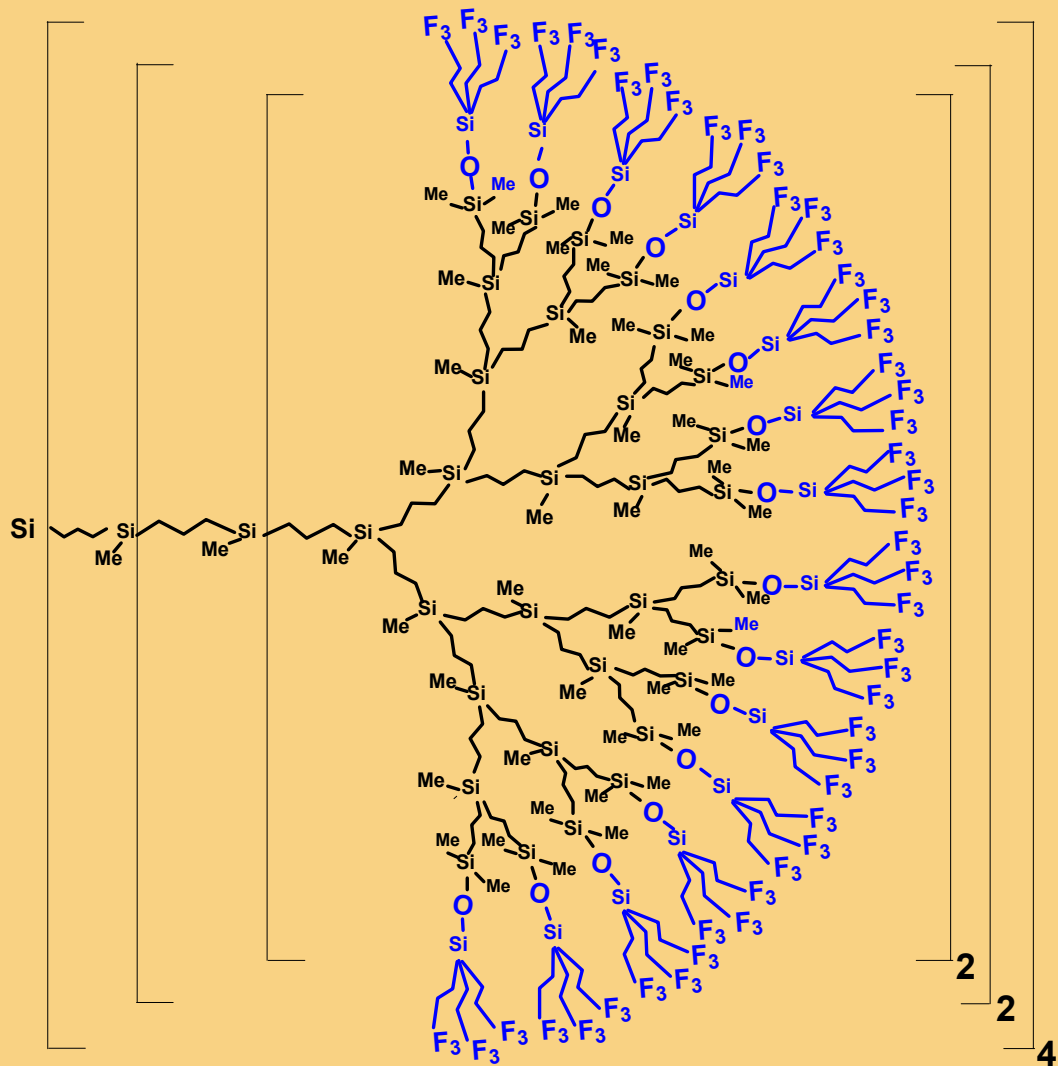


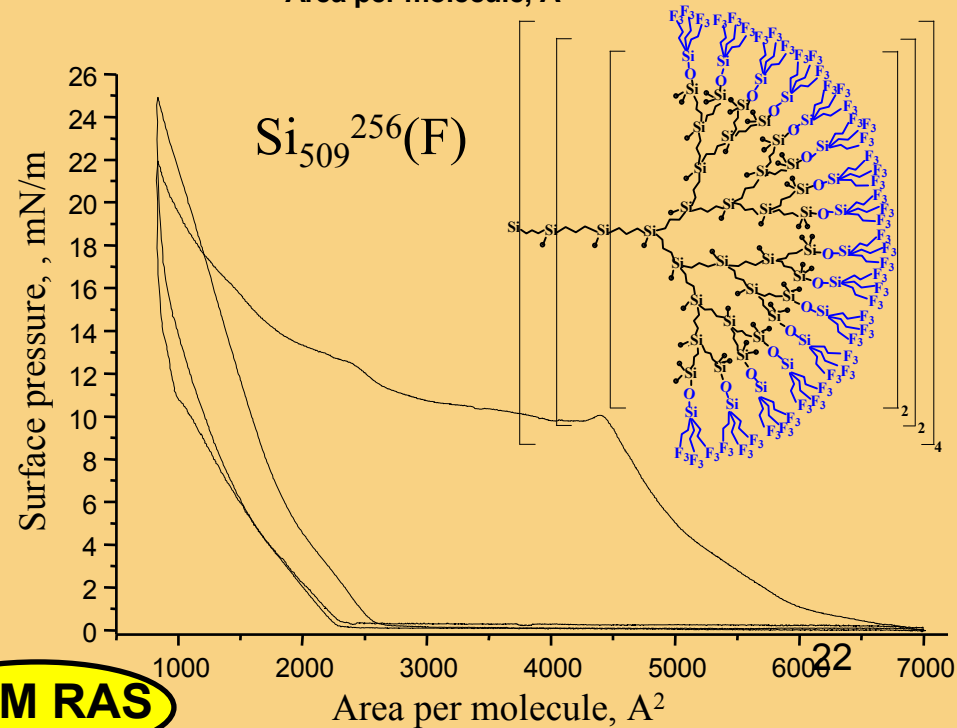
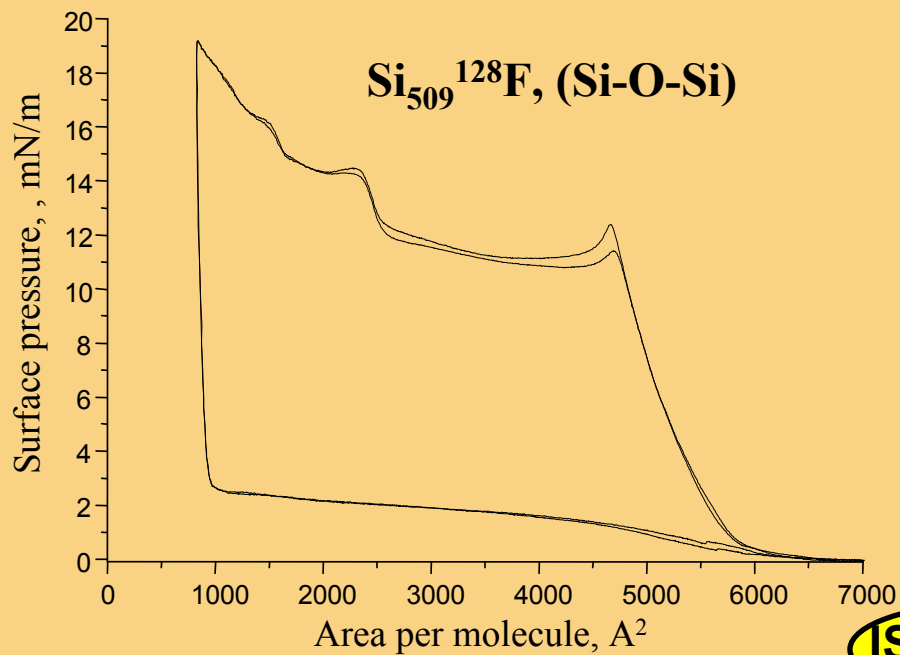
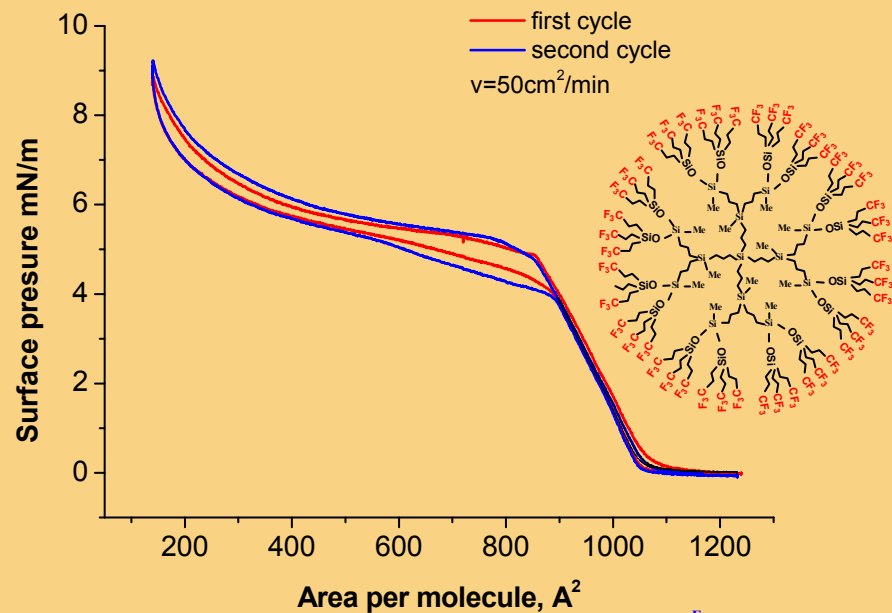
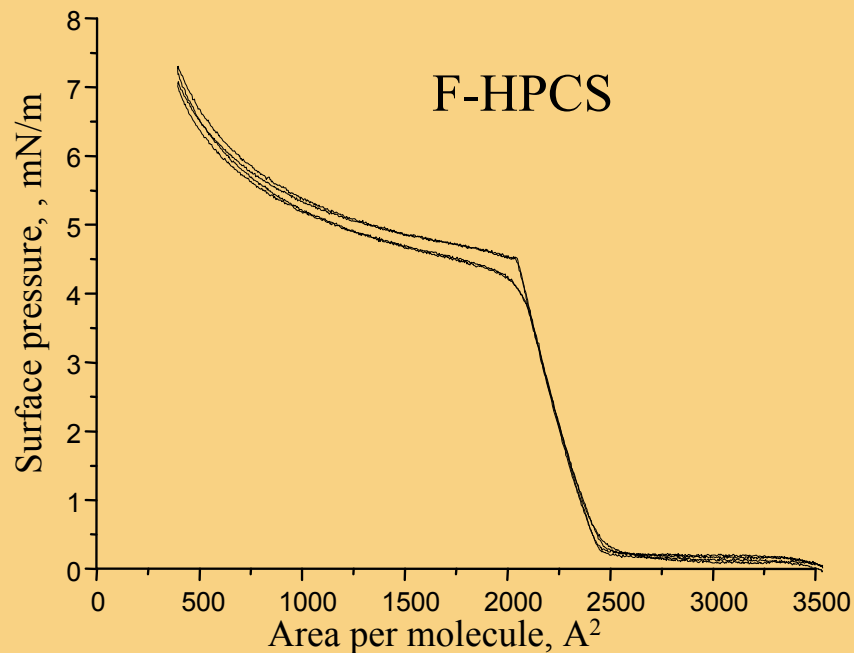
Дендри мер	ΔT , K	T_g° , K
G-3(Bu)	174-186	179 ± 1
G-4(Bu)	181-195	186 ± 1
G-5(Bu)	177-195	186 ± 1
G-6(Bu)	178-196	186 ± 1
G-7(Bu)	178-196	186 ± 1
G-9(Bu)	174-204	187 ± 1

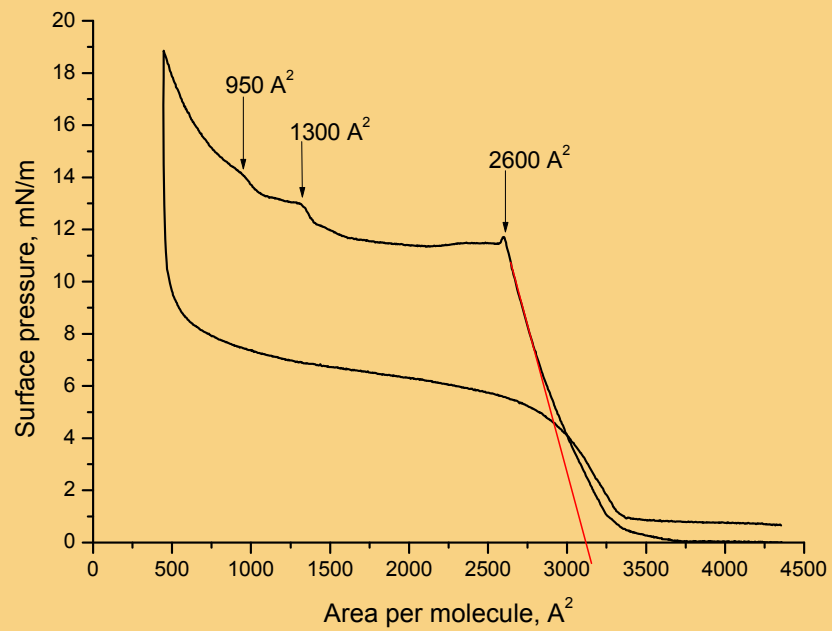
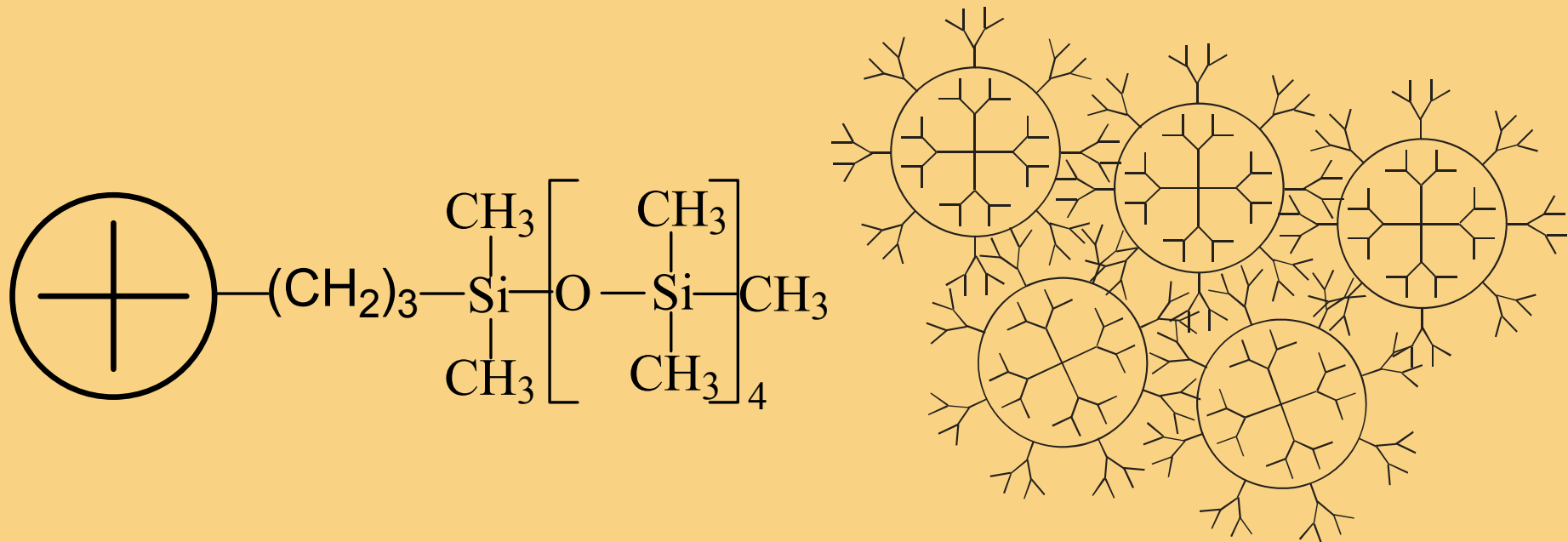
N.N. Smirnova et al. *Thermochimica Acta*. 2006. V.440. P.188-194

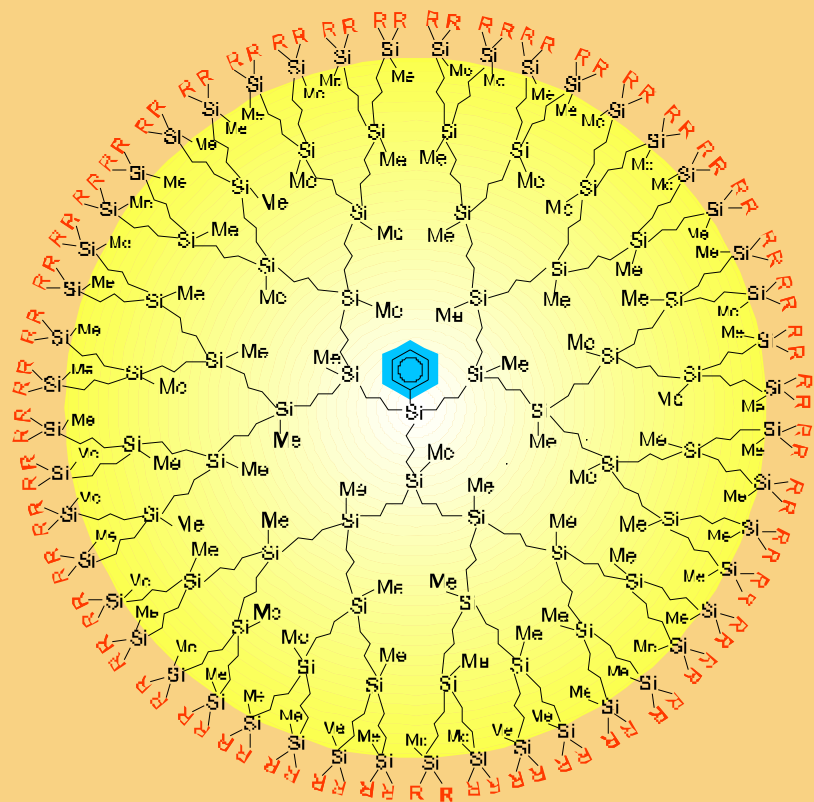
N.N. Smirnova et al. *Russ. Chem. Bull.* 2007. in print

Fluorocarbon Derivatives of Carbosilane Dendrimers of High Generation

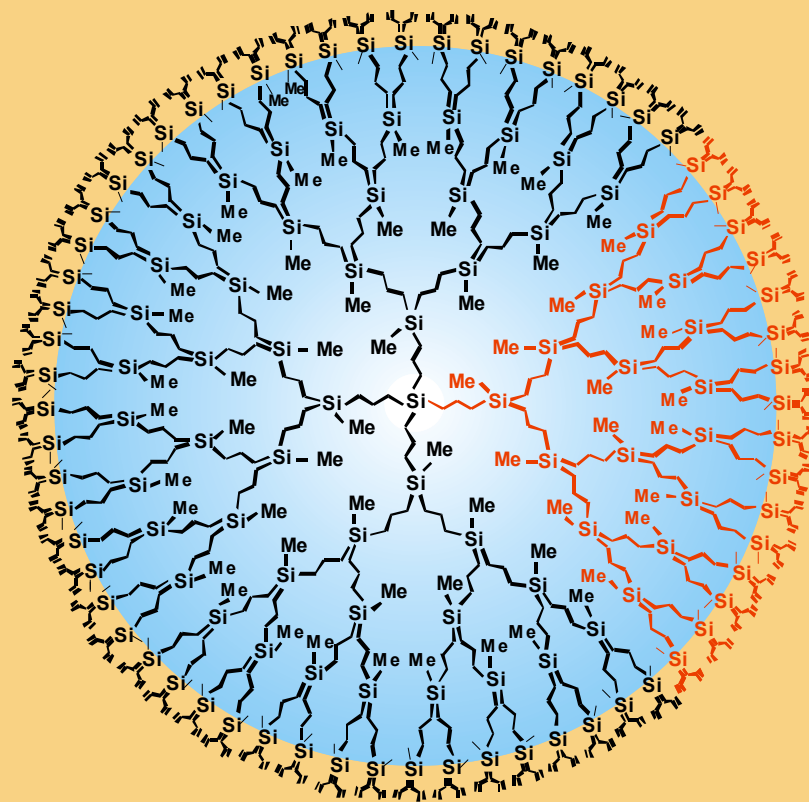




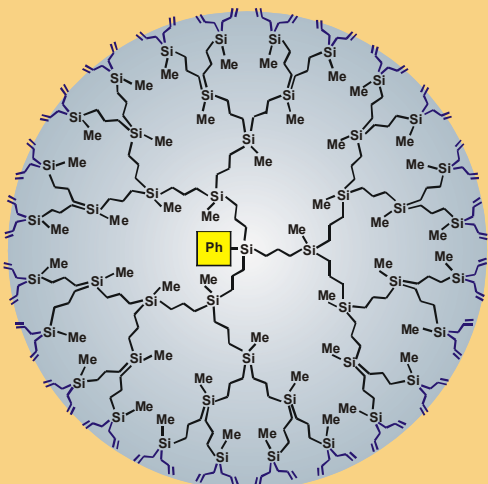




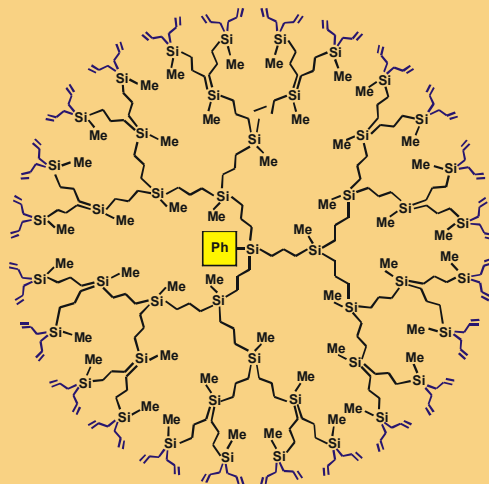
$\text{Si}_{92}, \text{G-5}$



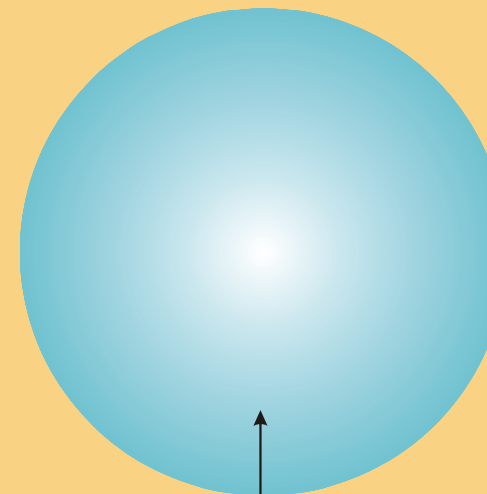
$\text{Si}_{128}, \text{G-5}$



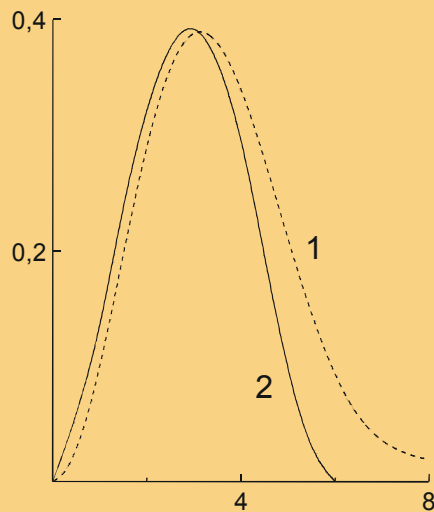
—



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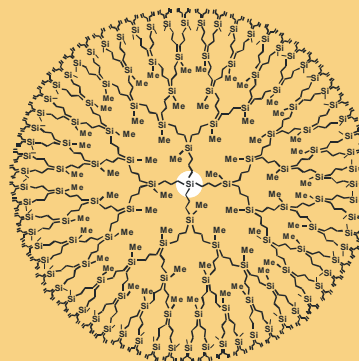
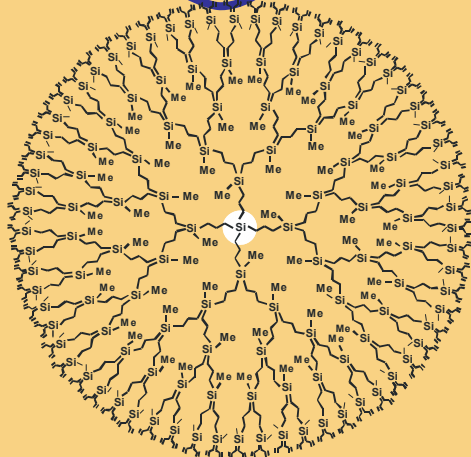
Free space : 30 - 40%



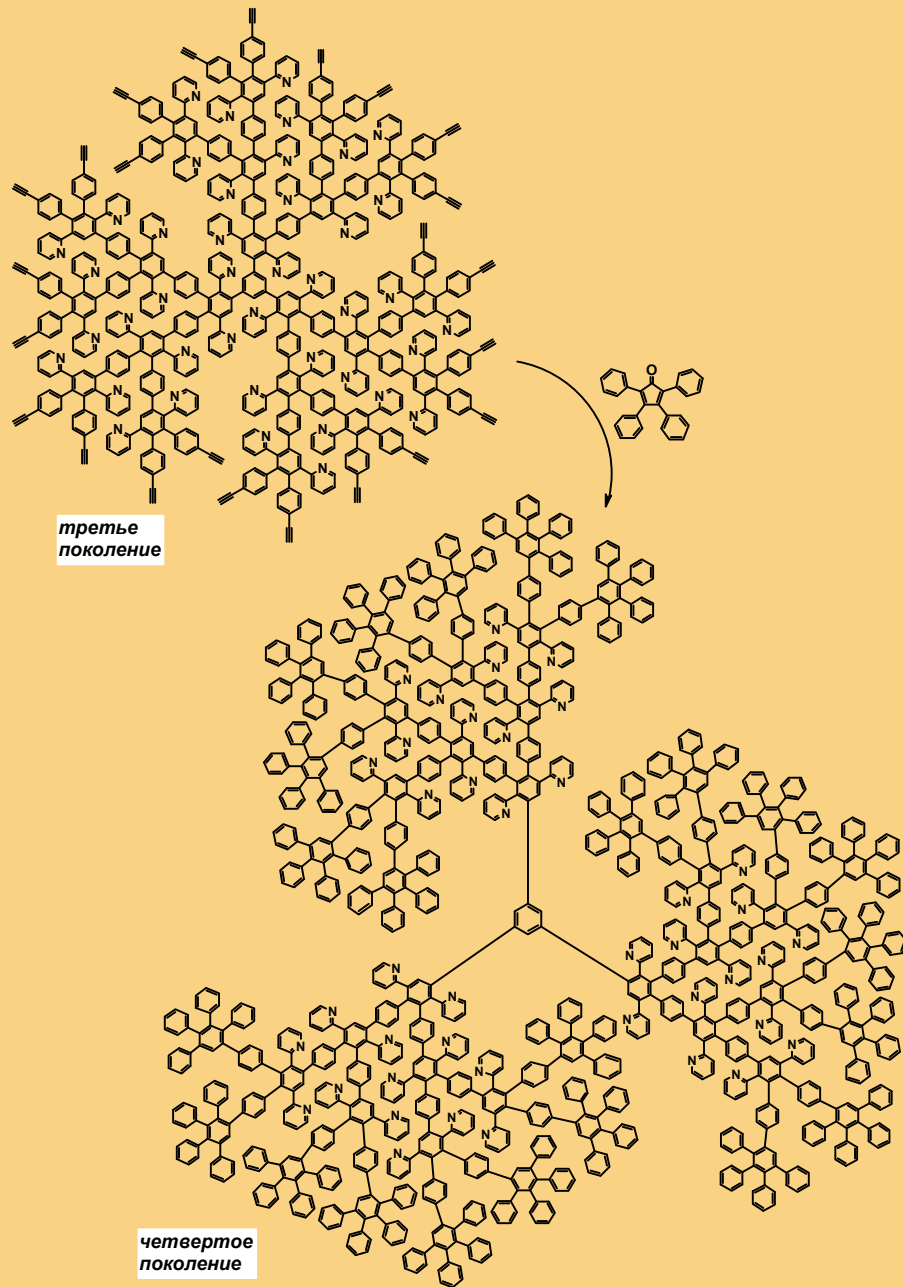
A. Kulkin, G. Ignat'eva, L. Ozerina, A. Islamov,
 R. Muhamedzyanov, N. Shumilkina, V. Myakushev,
 E. Sharipov, A. Muzafarov, A. Ozerin,
Polymer Science, 2002 series A, v. 44, № 12, 1273

1- SAXS plot G-7 (polyallylcarbosilane dendrimer)

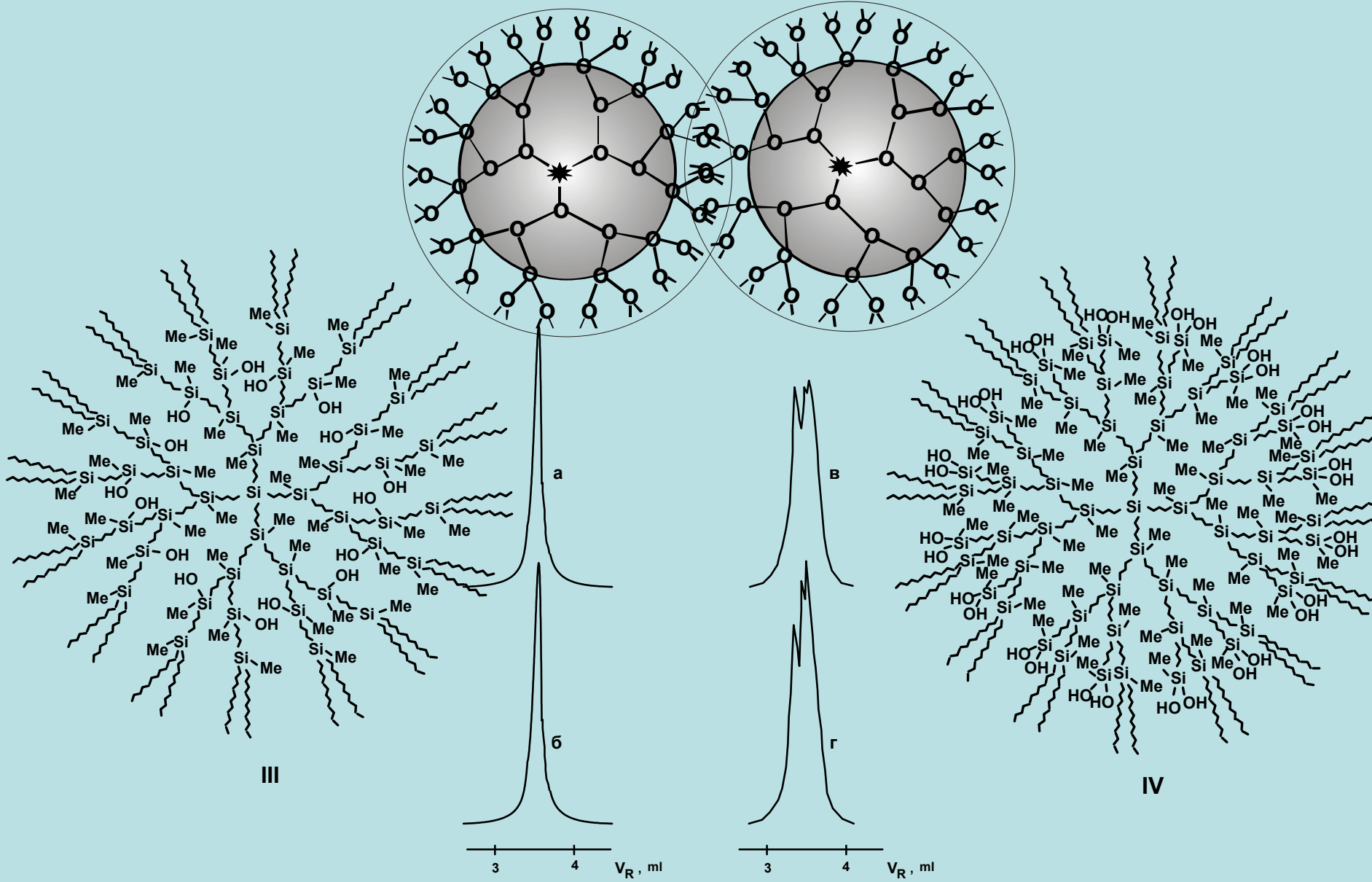
2- theoretical scattering curve of sphere like particle with $R_g = 2.3$ nm

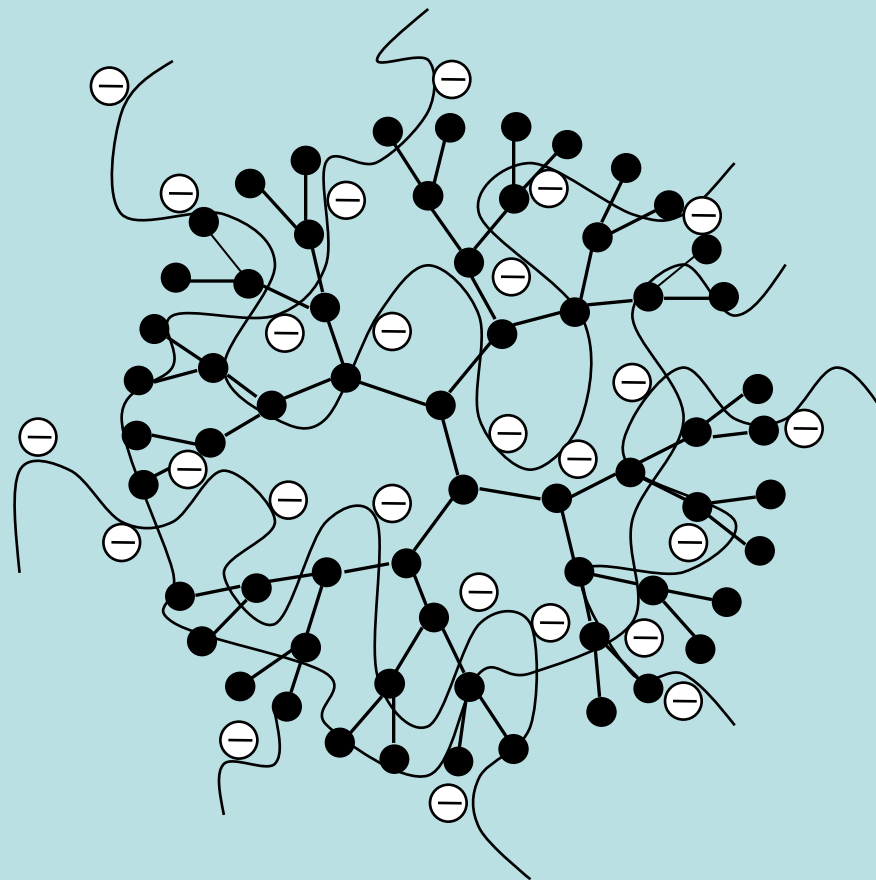


Solvent quality



T_g > T разл.





Kabanov V.A., Zezin A.B., Rogacheva V.B., Gulyaeva Zh.G., Zansochova M.F., Joosten J.G.H., Brackman J. Interaction of Astramol poly(propyleneimine) dendrimers with linear polyanions//Macromolecules, 1999, v. 32, № 6, p.1904-1909.

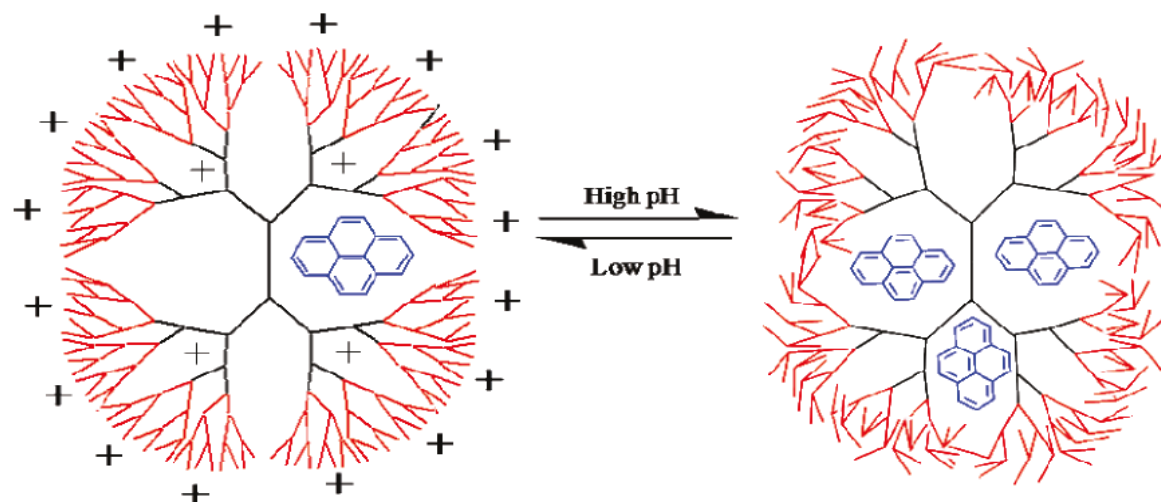
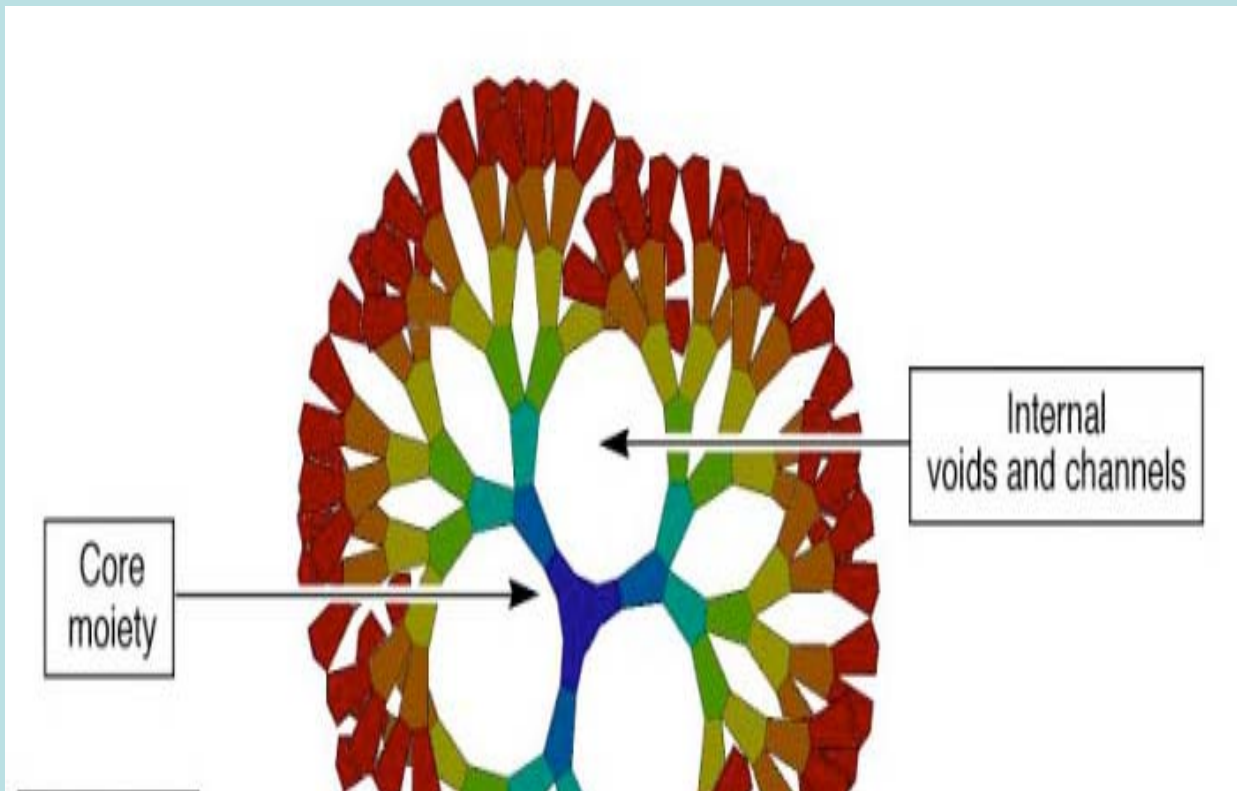
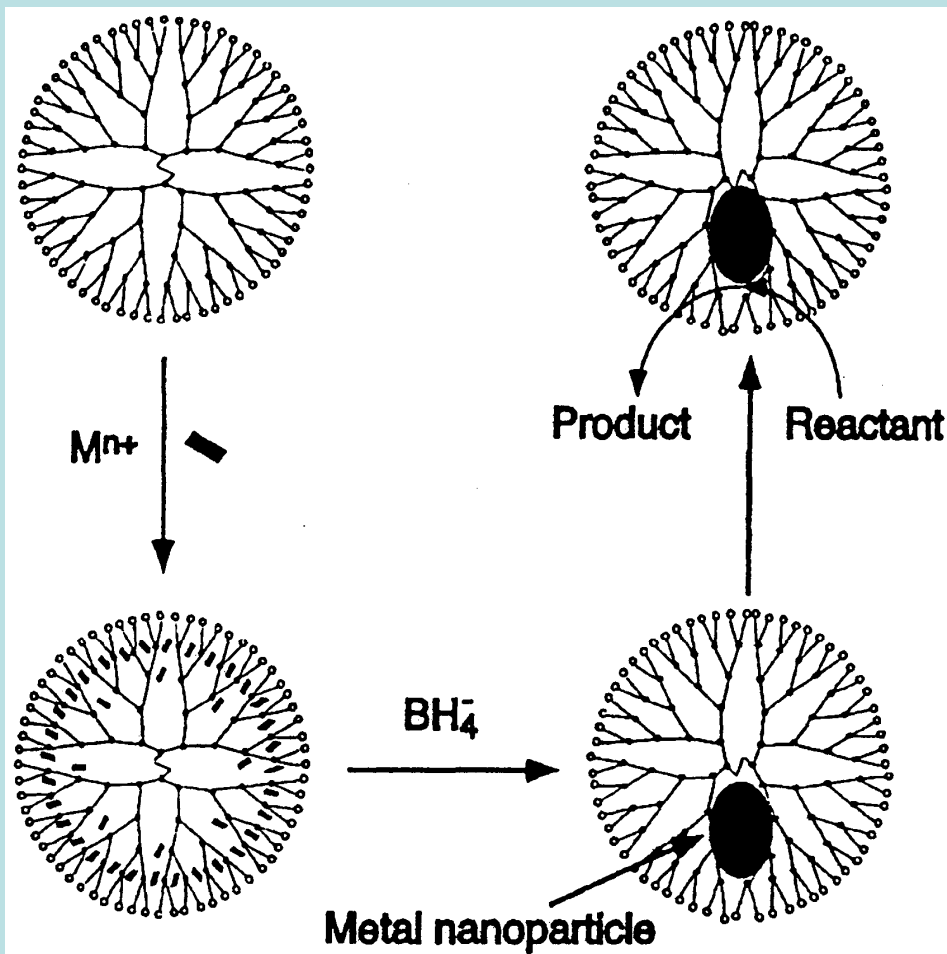


Figure 6. Schematic representation of pyrene encapsulation in PPI-core:PAMAM-shell dendrimers at acidic and basic pH.

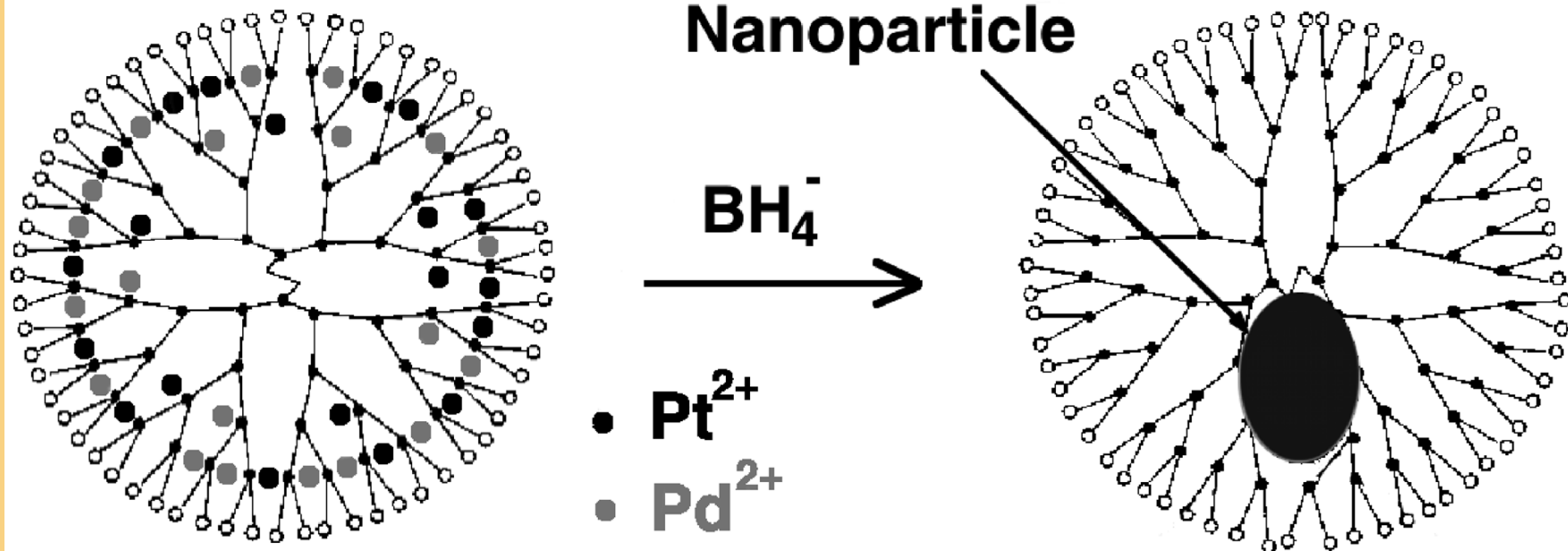
Scheme of step by step nanoclusters formation within dendrimer molecule



Prof. Crooks R. M.,
USA

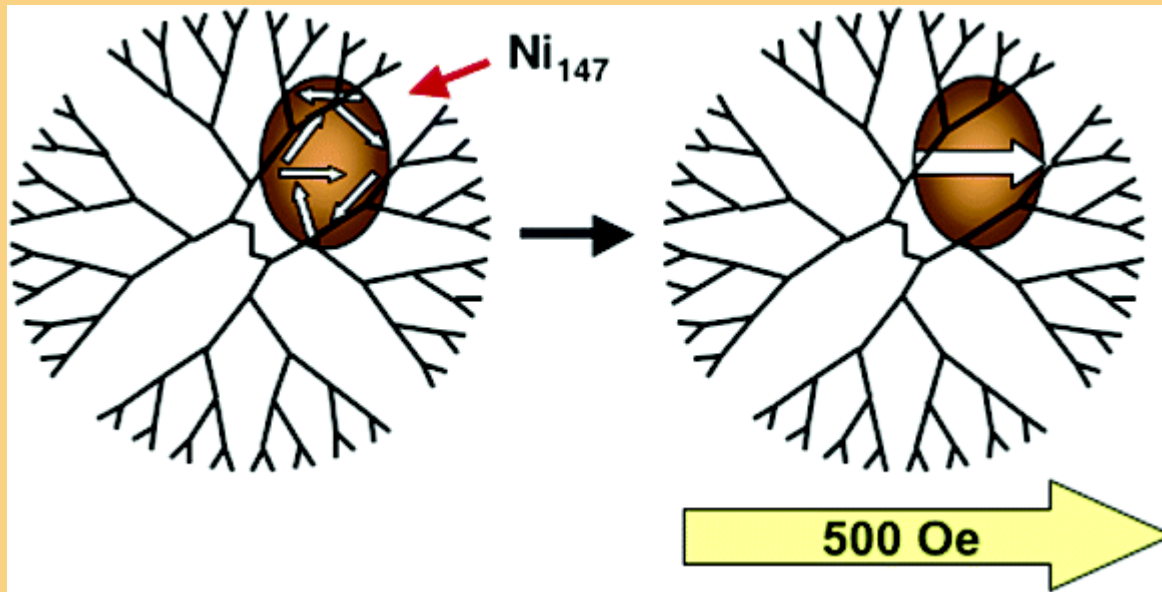
Dr. Balogh L.
USA

Bimetallic Nanoparticle



Synthesis, characterization, and catalytic activity of bimetallic palladium–platinum dendrimer-encapsulated catalysts (DECs). These materials are prepared by co-complexation of different ratios of palladium and platinum salts to the interior tertiary amines of fourth-generation, hydroxyl-terminated poly(amidoamine) (PAMAM) dendrimers. Chemical reduction of these composites yields stable, fairly monodisperse, water-soluble bimetallic DECs having sizes on the order of 1.9 ± 0.4 nm. Evidence that these nanoparticles are bimetallic comes from single-particle X-ray energy dispersive spectroscopy (EDS) and catalysis experiments. The latter indicate that the hydrogenation rate of allyl alcohol is enhanced in the presence of the bimetallic nanoparticles compared to DECs containing only platinum or only palladium nanoparticles. EDS results indicate that the percentage composition of the bimetallics is reflected by the percentage of metal salts initially complexed with the dendrimer.

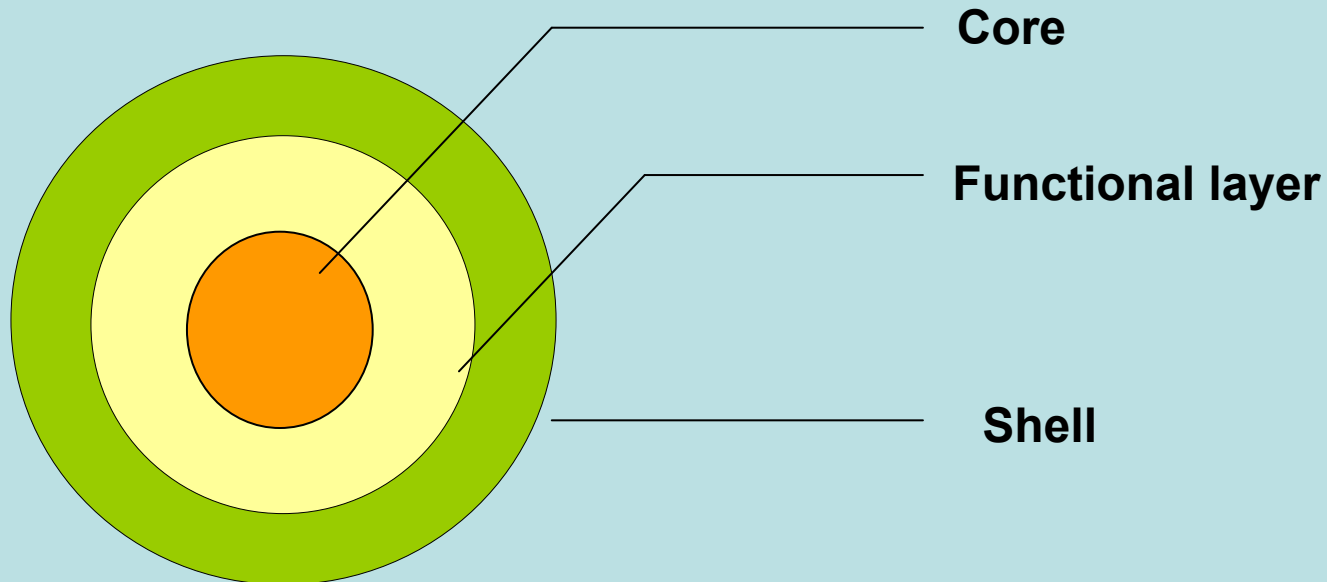
J. Am. Chem. Soc., **2003**, *125* (13), pp 3708–3709



The synthesis and magnetic properties of Ni nanoparticles containing <150 atoms are described. These materials are prepared in an organic solvent using hydrophobic dendrimers as templates, which results in a high degree of size monodispersity. The dendrimer-encapsulated nanoparticles were characterized by UV-vis spectroscopy, transmission electron microscopy, and energy dispersive spectroscopy. Magnetic (SQUID) studies indicate that the particles are ferromagnetic at 5 K with magnetic saturations that vary depending upon the particle size.

Chem. Mater., **2006**, *18* (21), pp 5039–5044

Model of dendrimer matrix with active inner sphere



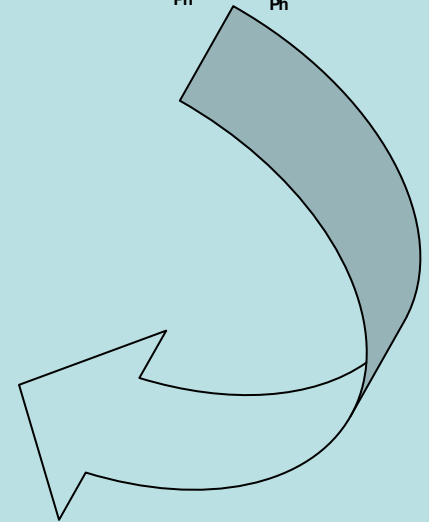
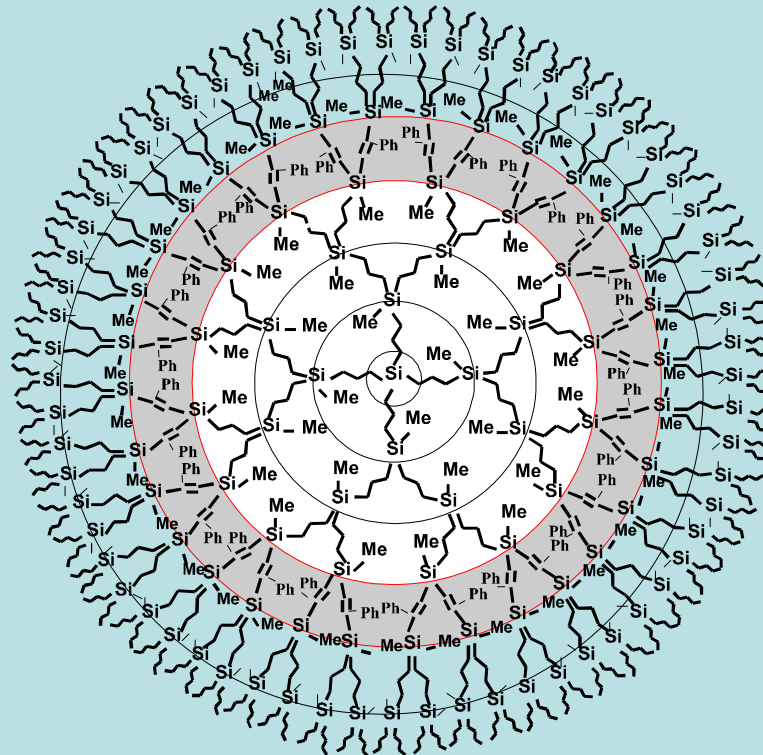
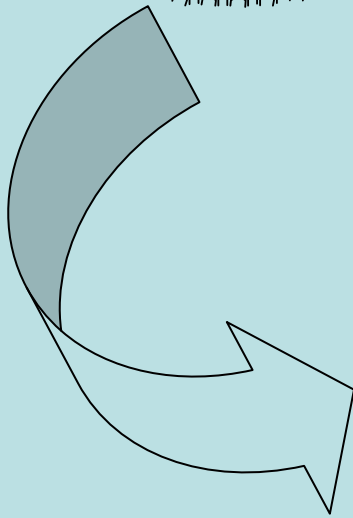
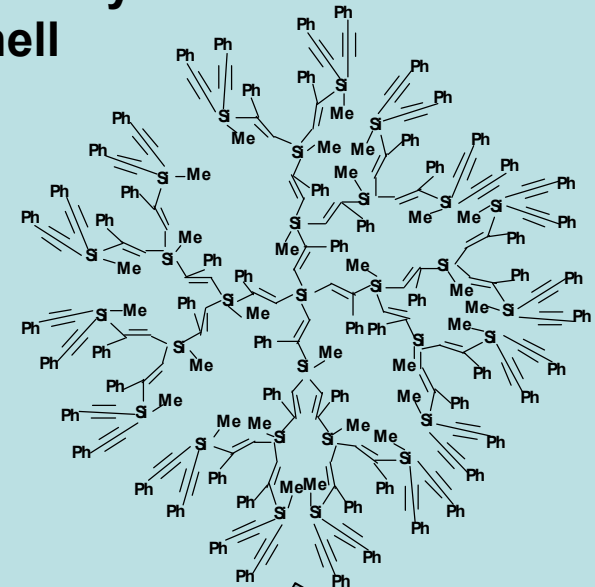
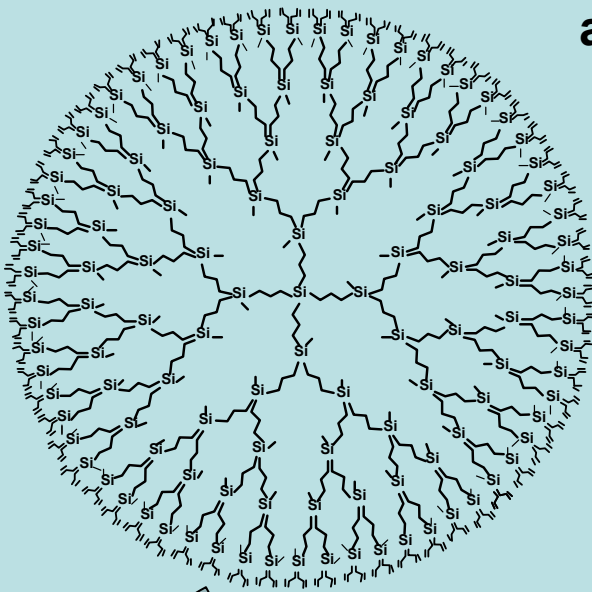
Merits:

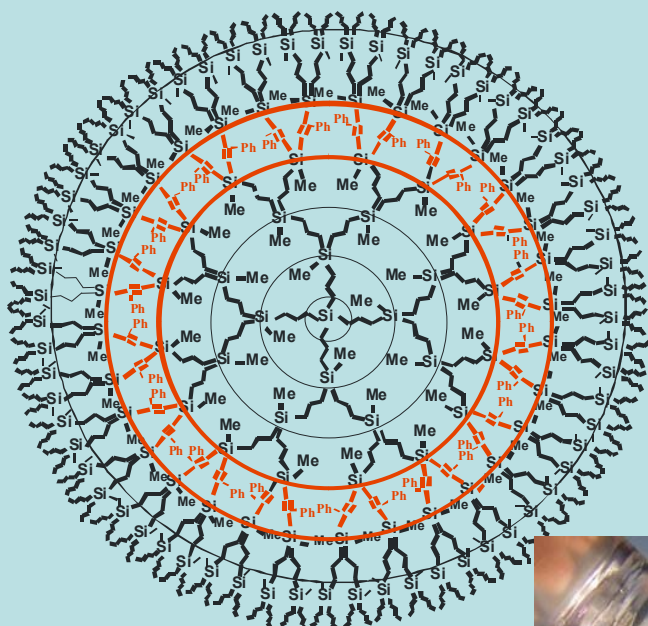
Dendritic clusters are monodispersed

Clusters are stabilized by dendritic structure

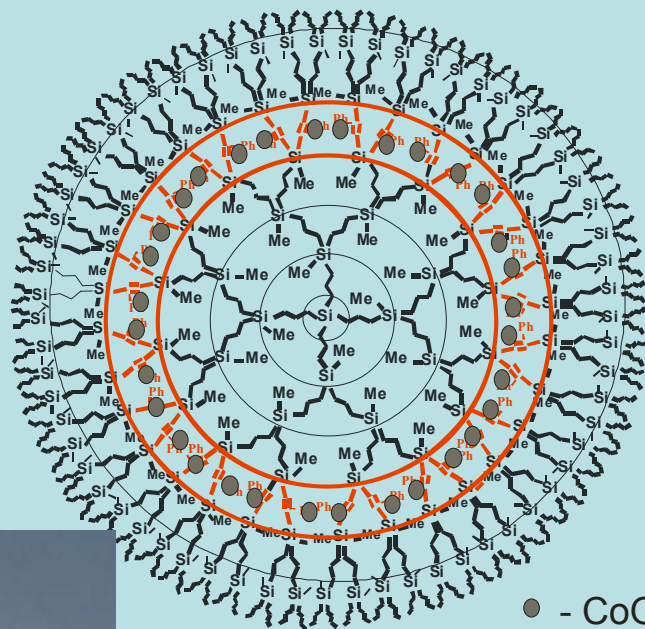
Possibility of cluster localization inside molecule

Dendrimer G5 with inner functional layer and non-functional outer shell



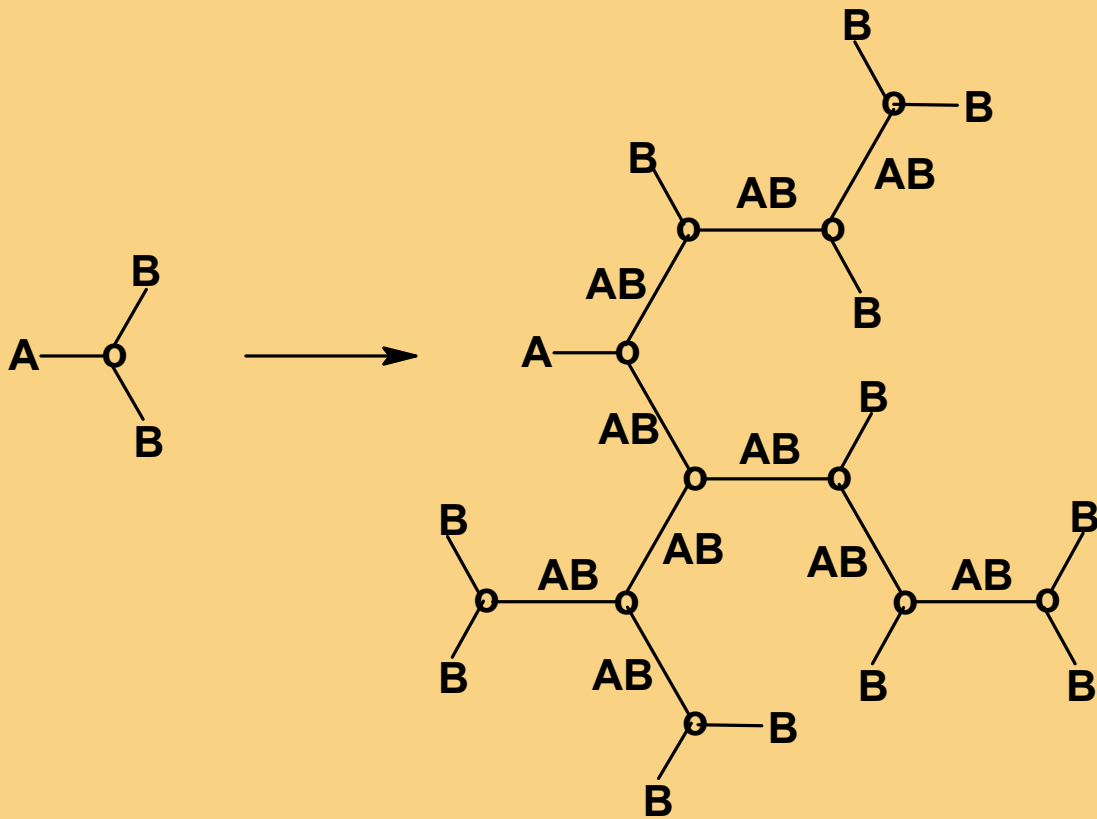


G5 (Bu)

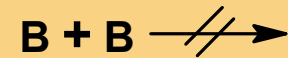
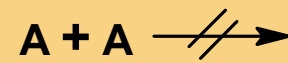


G5 (Bu)+CoO



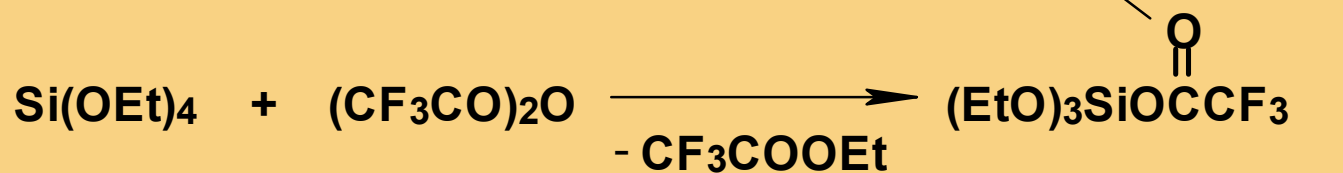
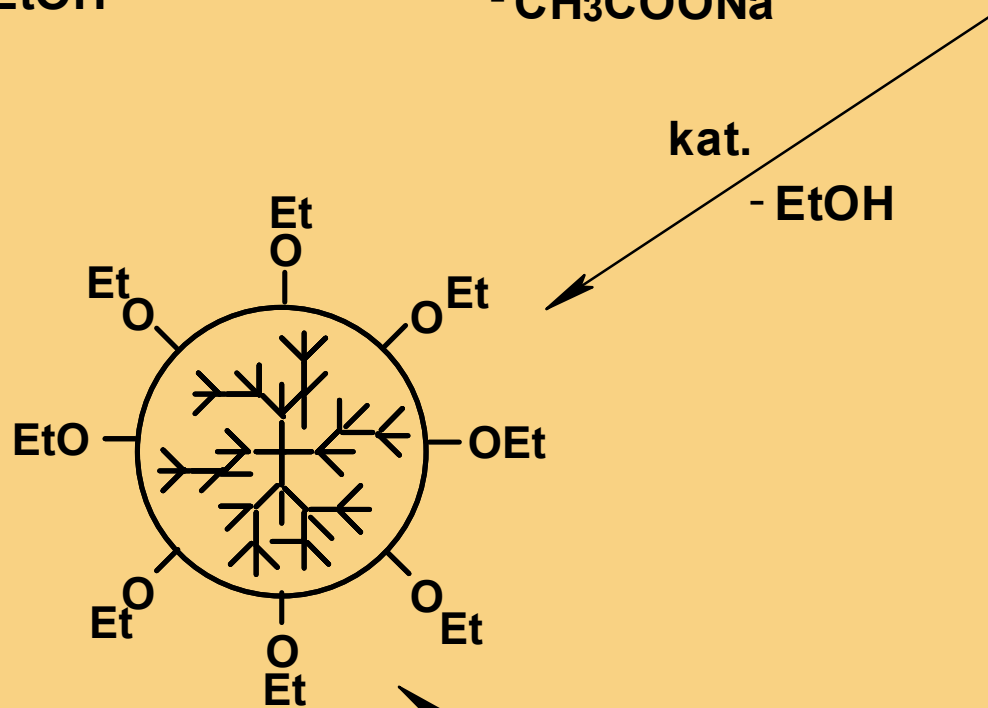
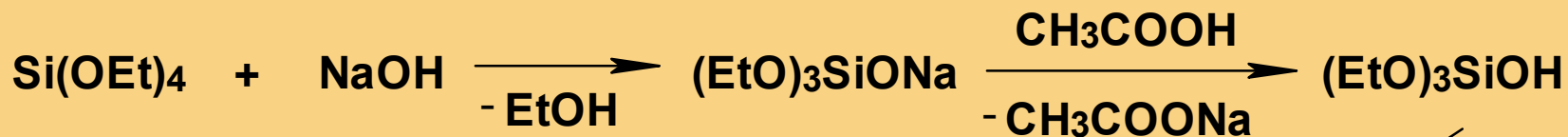


Условия Флори

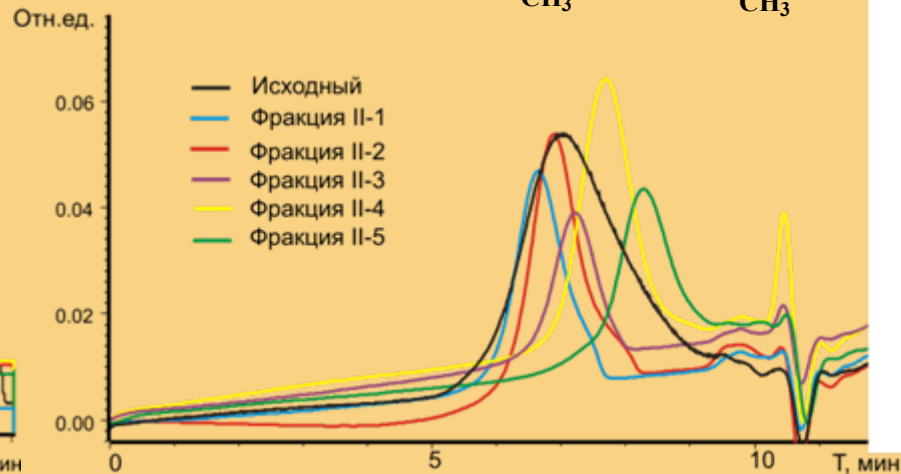
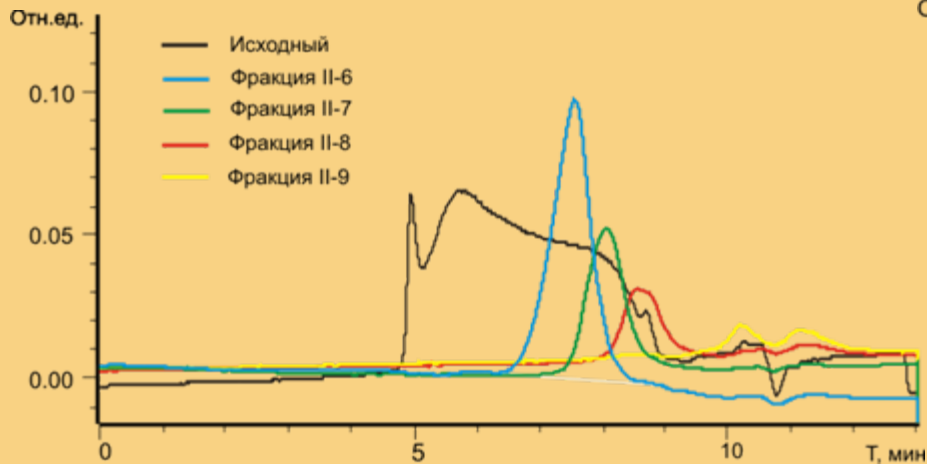
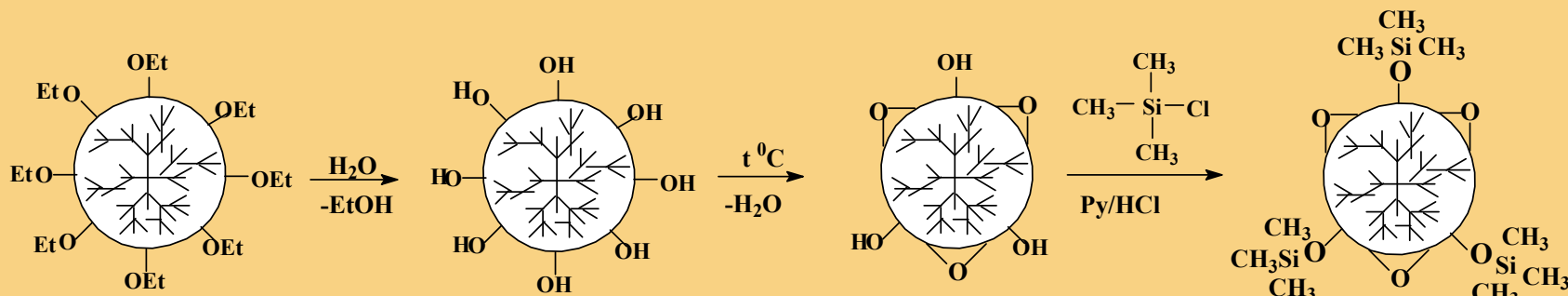
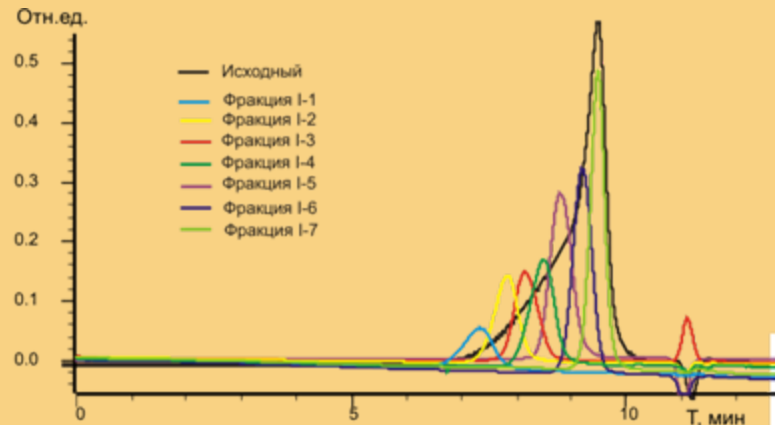
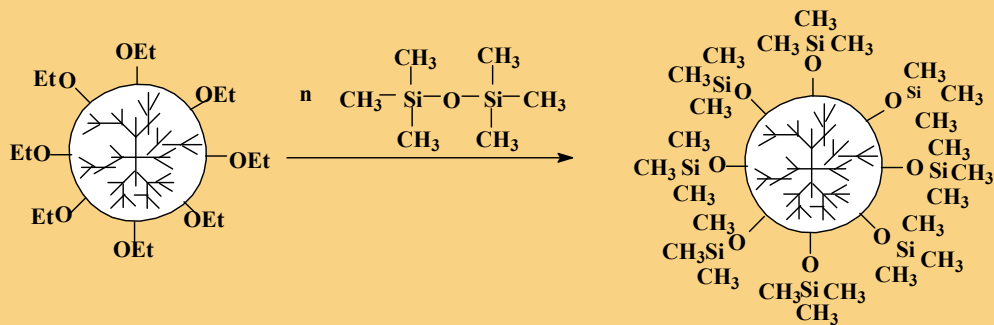


$$DB = \frac{T+D}{T+D+L}$$

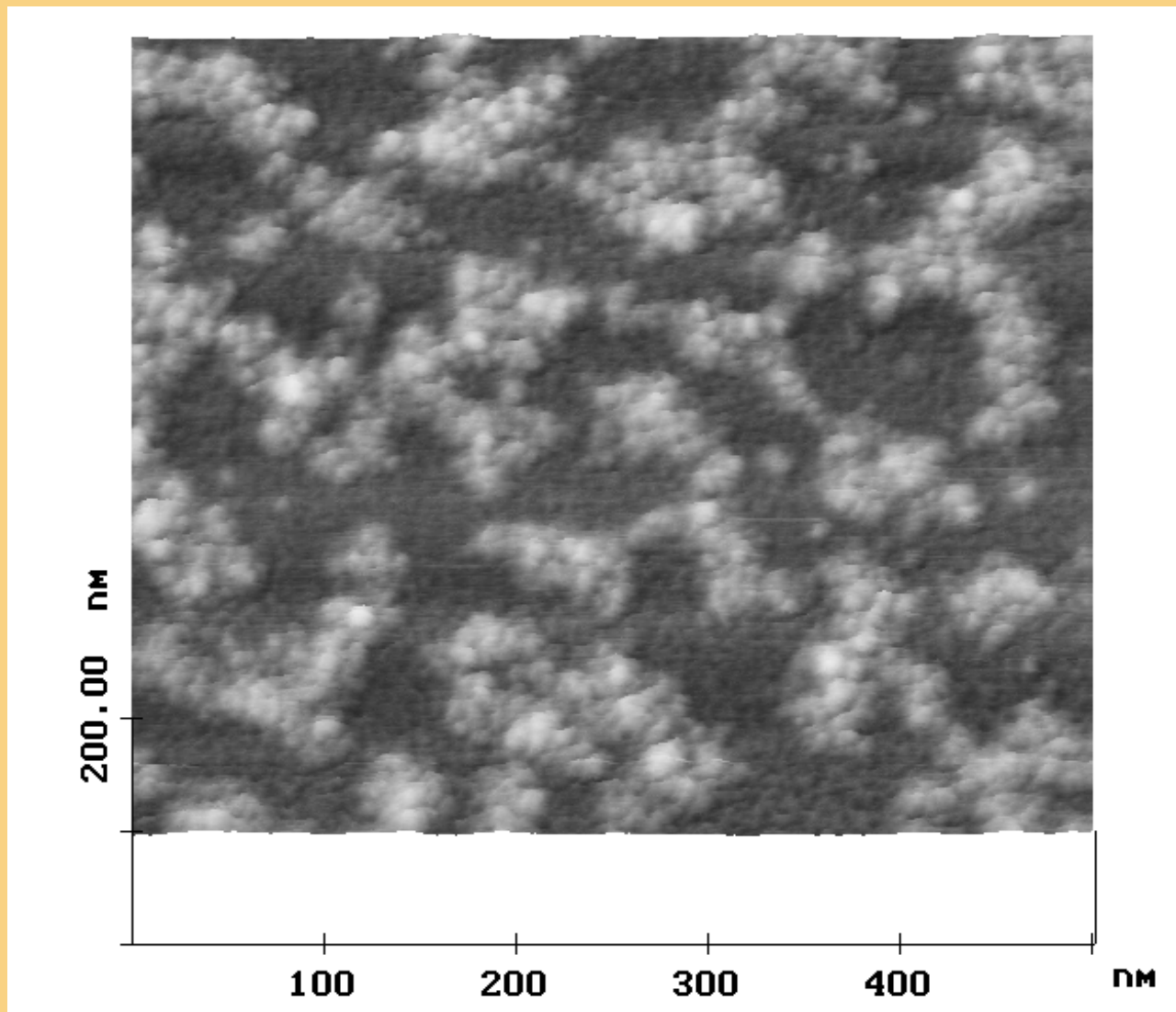
Неорганическая дендритная структура



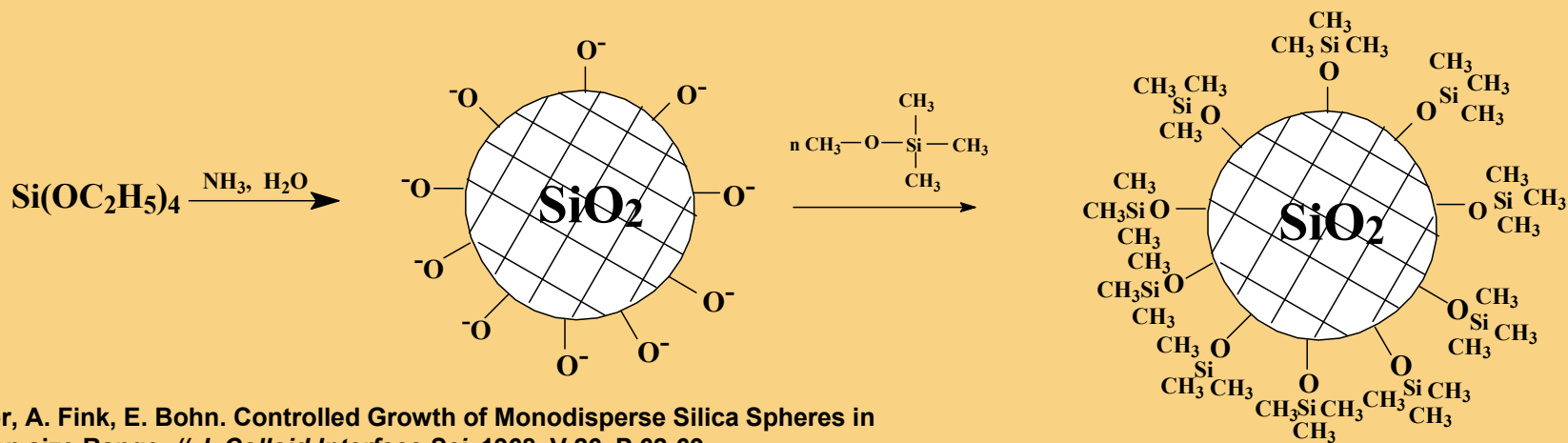
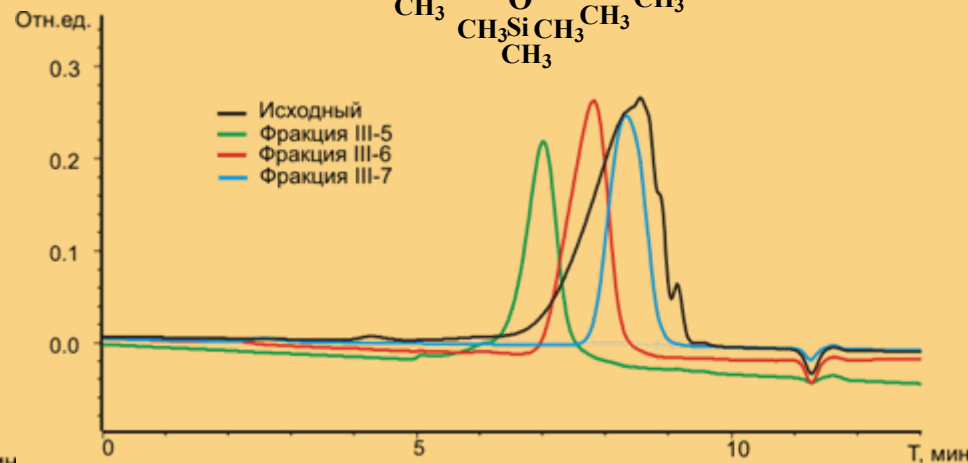
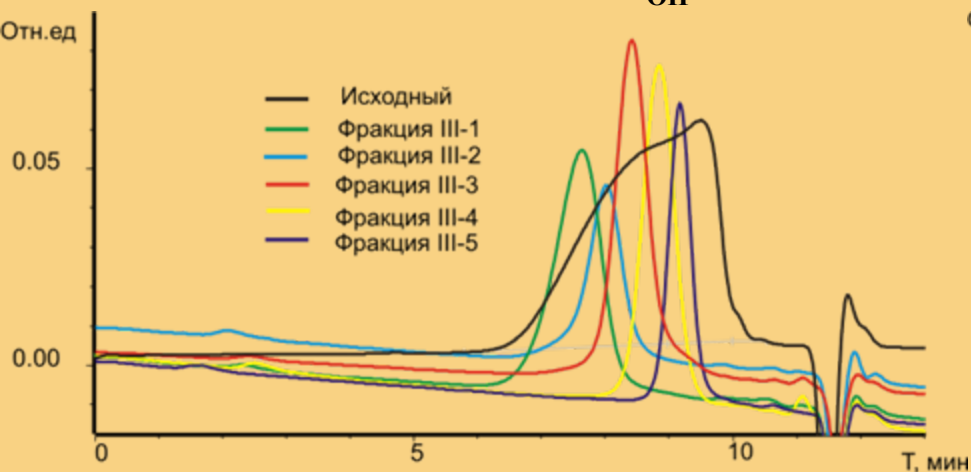
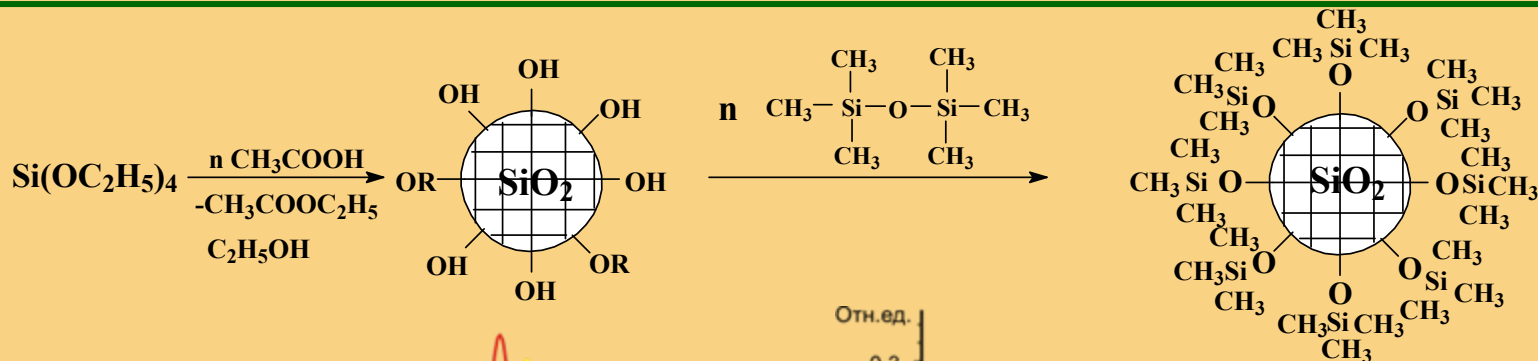
Первый и второй способы получения гибридных наночастиц на основе сверхразветвленного полиэтоксисилоксана и силиказоля.



Scanning force photomicrograph of silica-sol on mica

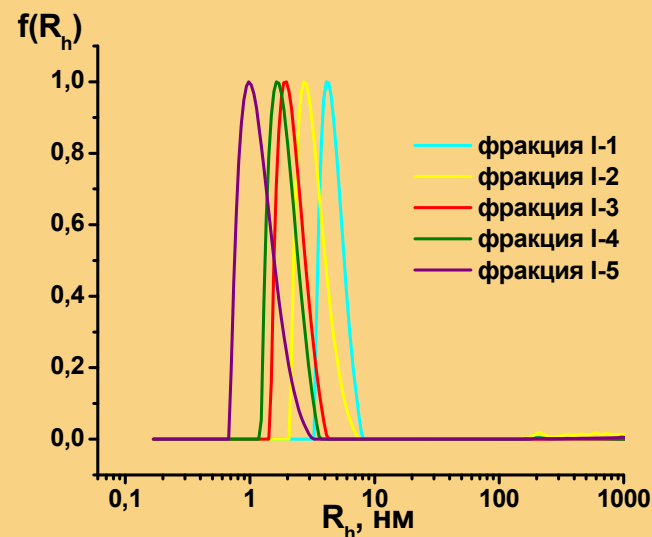
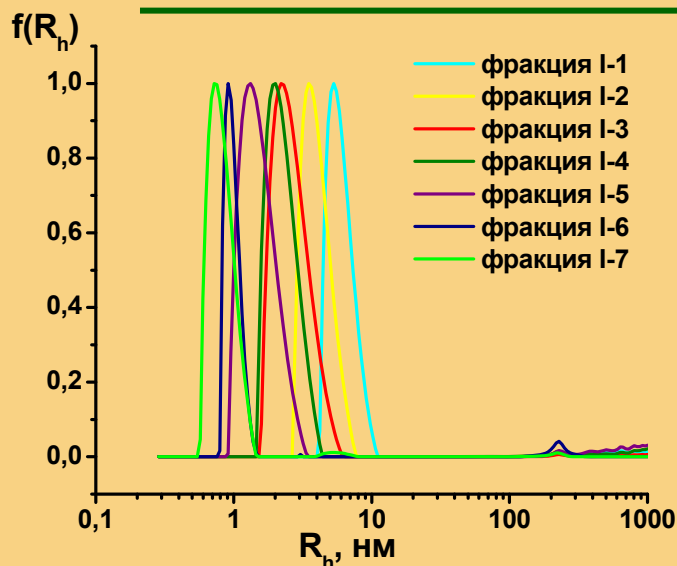


Третий и четвертый способы получения гибридных наночастиц на основе тетраэтоксисилана в кислой и щелочной средах.



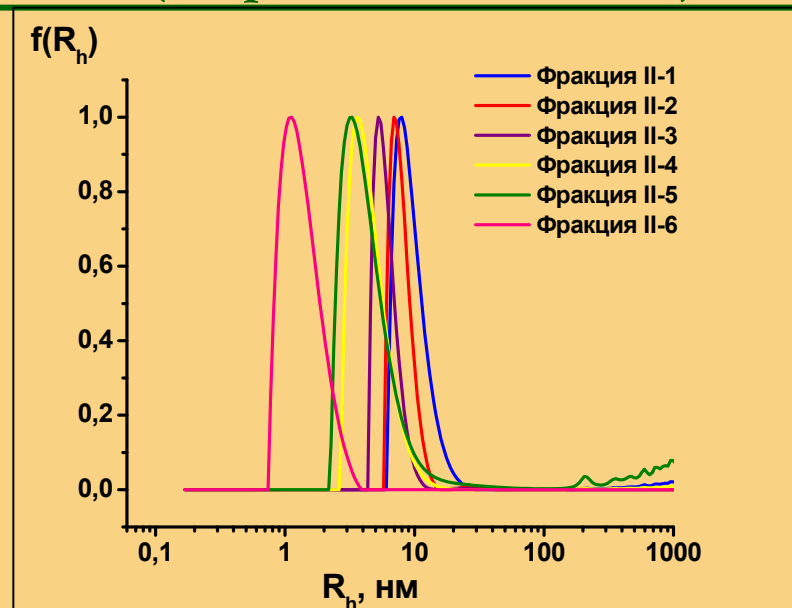
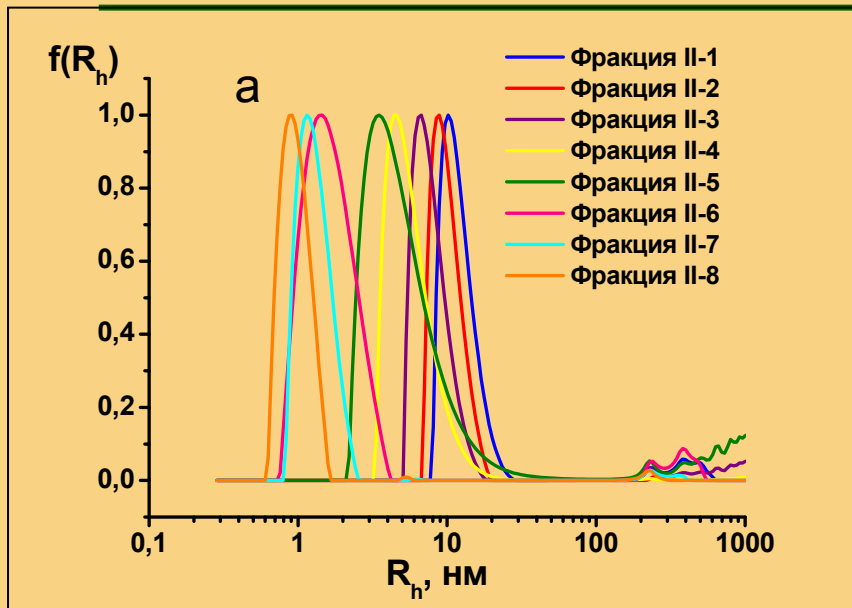
Гидродинамические свойства наночастиц.

Наночастицы на основе полиэтоксисилоксана (первый метод синтеза).



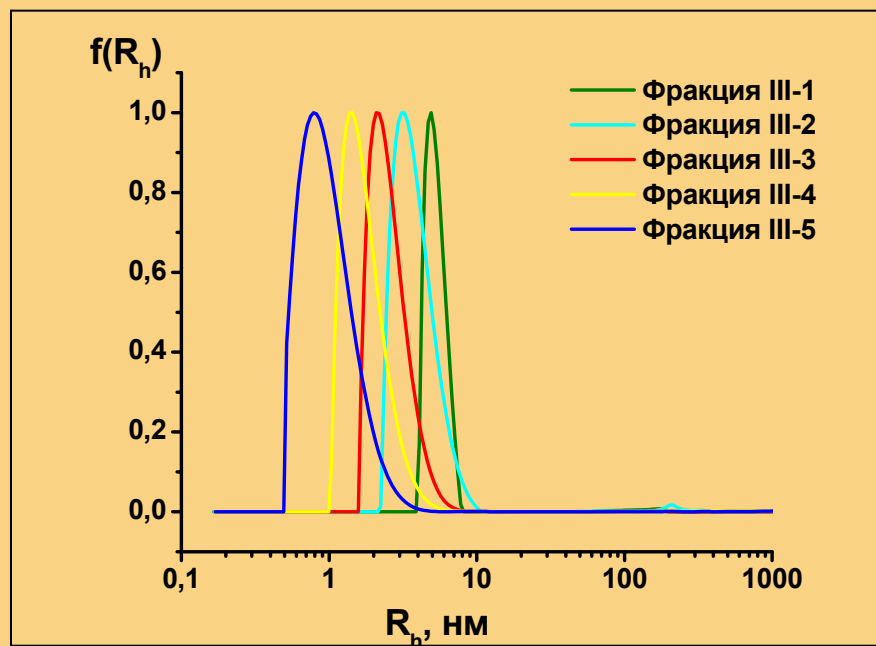
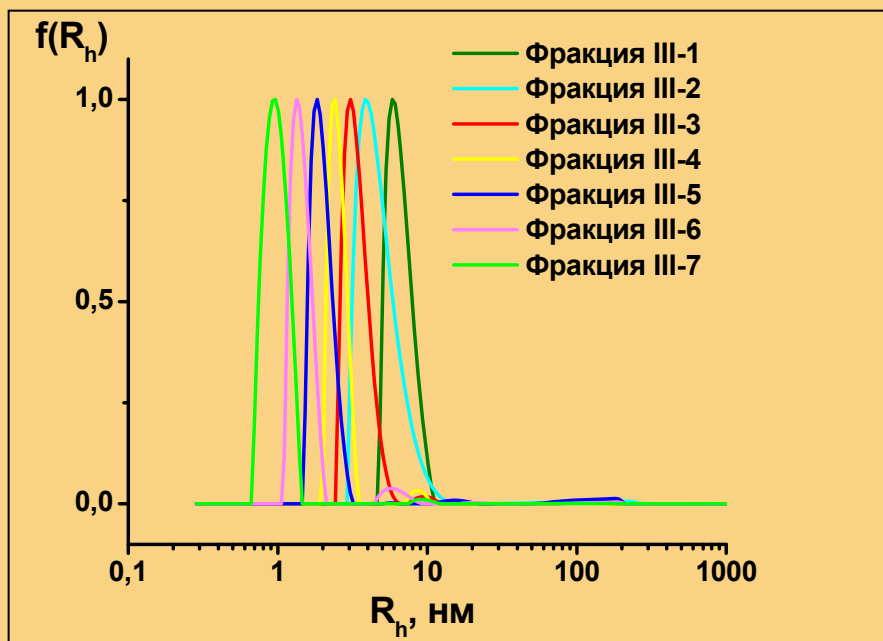
Образец № фракции	M, универсальная калибровка	R, нм (ГПХ)	R, нм (ДСР) Толуол	R, нм (ДСР) МТБЭ	η (Толуол), дл/г	η (МТБЭ), дл/г
		Толуол+2 %ТГФ				
I-1	152000±15000	4,5±0,5	4,1±0,2	5,3±0,3	0,040±0,002	0,061±0,003
I-2	59300±6000	3,0±0,3	2,7±0,2	3,5±0,2	0,033±0,002	0,043±0,002
I-3	34700±3500	2,3±0,2	2,0±0,1	2,2±0,2	0,025±0,001	0,030±0,002
I-4	21500±2000	1,8±0,2	1,7±0,1	2,0±0,1	0,020±0,001	0,028±0,001
I-5	12600±1500	1,4±0,2	1,0±0,1	1,3±0,1	0,017±0,001	0,021±0,001
I-6	6650±1200	1,0±0,2	<1	<1	0,013±0,001	0,019±0,001
I-7	4500±800	0,8	<1	<1	0,010±0,001	0,014±0,001

Наночастицы на основе силиказоля (второй метод синтеза).



Образец	M, универсальная калибровка	R, нм (ГПХ) ТГФ	R, нм (ДСР) Толуол	R, нм (ДСР) МТБЭ	η (Толуол), дл/г	η (МТБЭ), дл/г
№ фракции						
II-1	511000±51000	8,3±0,8	8,0±0,4	10,2±0,5	-	0,072±0,004
II-2	390000±40000	6,9±0,7	7,0±0,4	8,9±0,4	0,055±0,003	0,059±0,003
II-3	220000±22000	5,4±0,5	5,2±0,3	6,7±0,3	0,047±0,002	0,055±0,003
II-4	93000±9000	3,8±0,4	3,6±0,2	4,4±0,2	0,037±0,002	0,041±0,002
II-5	25000±2500	2,4±0,2	3,3±0,2	3,5±0,2	-	-
II-6	11500±1700	1,5±0,2	1,1±0,1	1,5±0,1	0,017±0,001	-
II-7	5000±1000	1,0±0,1	-	1,2±0,1	0,014±0,001	-
II-8	2400±500	0,7±0,1	-	<1	0,012±0,001	-

Наночастицы на основе тетраэтоксисилана (третий метод синтеза).

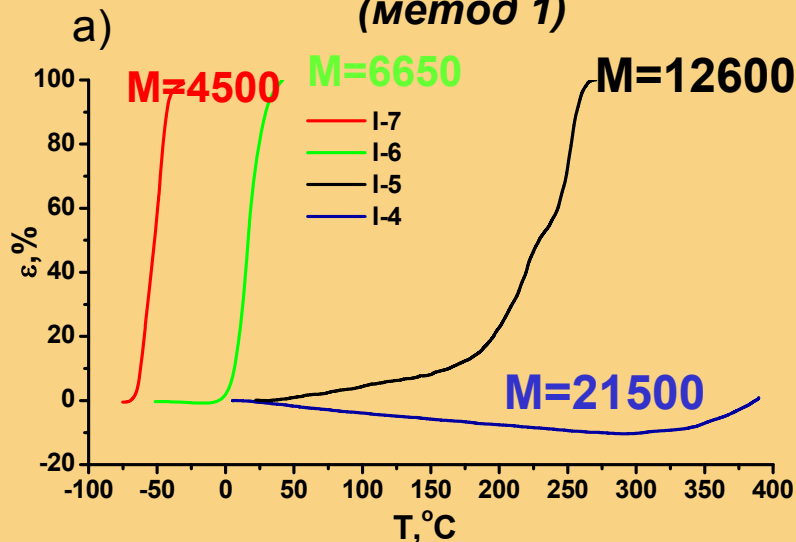


Образец № фракции	M, универсальная калибровка	R, нм (ГПХ)	R, нм (ДСР)	R, нм (ДСР)	η (Толуол), дл/г	η (МТБЭ), дл/г
		Толуол+2 %ТГФ	Толуол	МТБЭ		
III-1	116100±12000	4,5±0,5	4,9±0,2	5,8±0,3	-	0,053±0,003
III-2	61000±6100	3,4±0,3	3,1±0,2	3,8±0,2	-	0,044±0,002
III-3	26800±2700	2,5±0,3	2,1±0,1	3,1±0,2	0,042±0,002	0,042±0,002
III-4	13450±1300	1,8±0,2	1,4±0,1	2,4±0,1	0,033±0,002	0,033±0,002
III-5	8300±1200	1,4±0,2	<1	1,8±0,1	-	-
III-6	7300±1200	1,1±0,2	<1	1,3±0,1	0,019±0,001	0,023±0,001
III-7	3300±700	0,8±0,2	<1	1,0±0,1	0,017±0,001	0,023±0,001

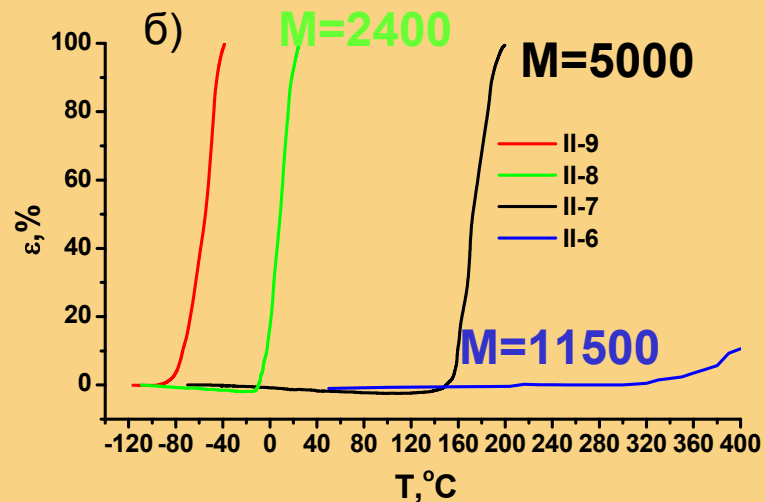
Определение температуры стеклования.

Частицы на основе полиэтоксисилоксана

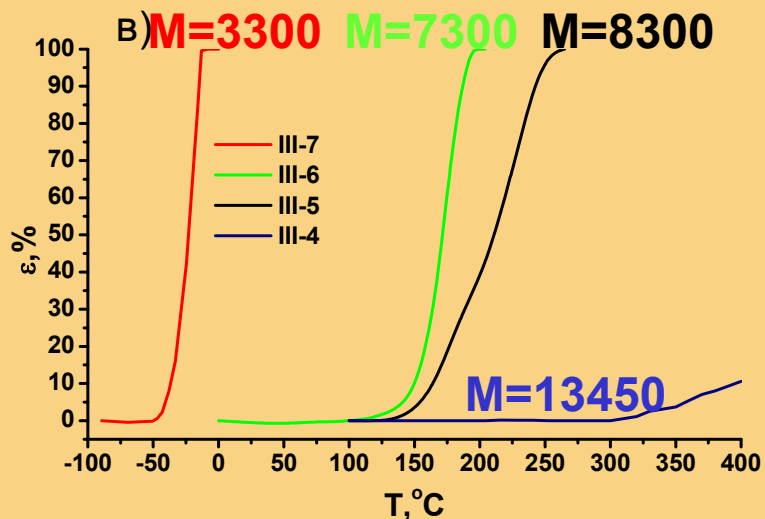
(метод 1)



Частицы на основе силиказоля (метод 2)

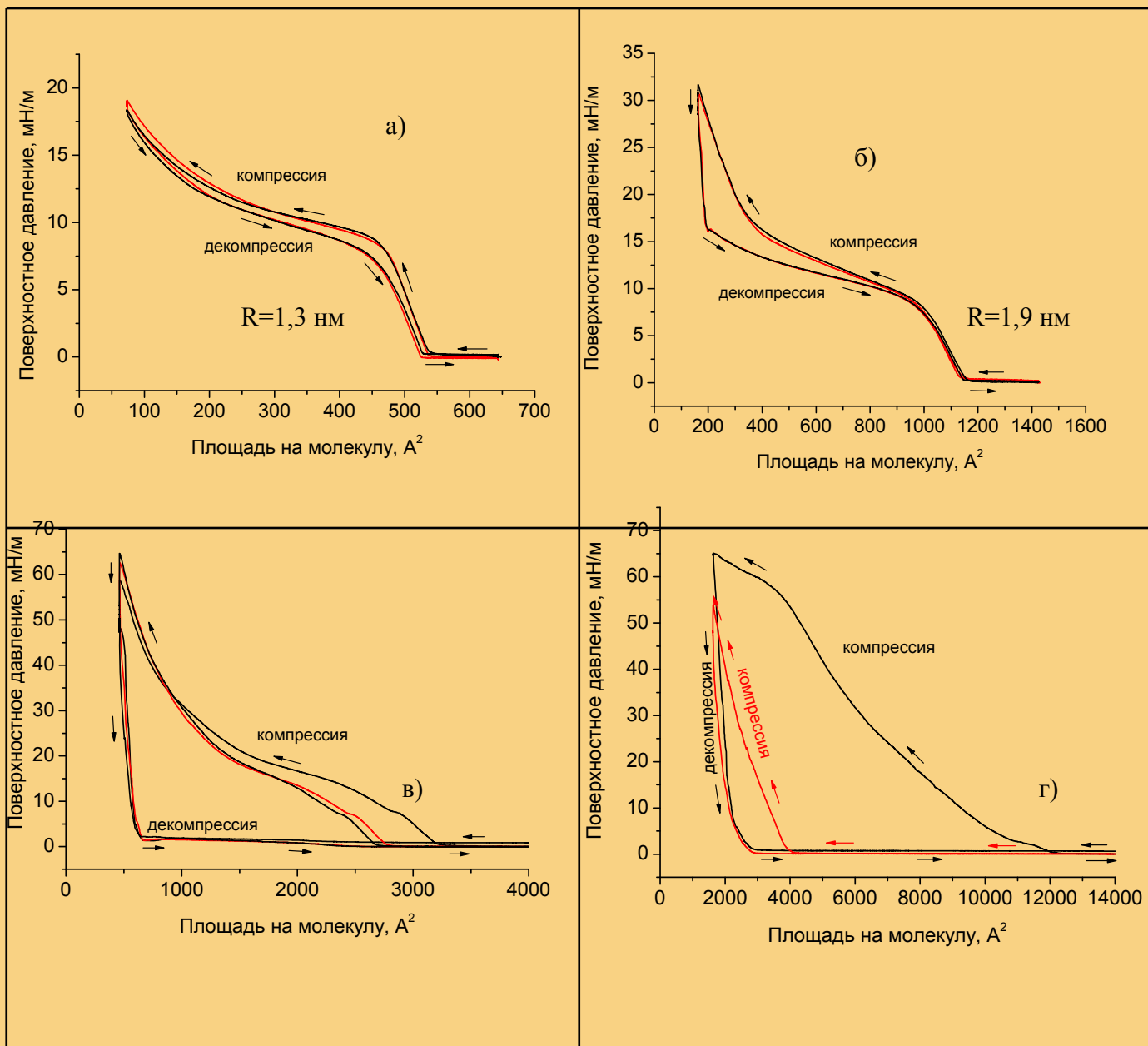


Частицы на основе тетраэтоксисилана
в кислой среде (метод 3)



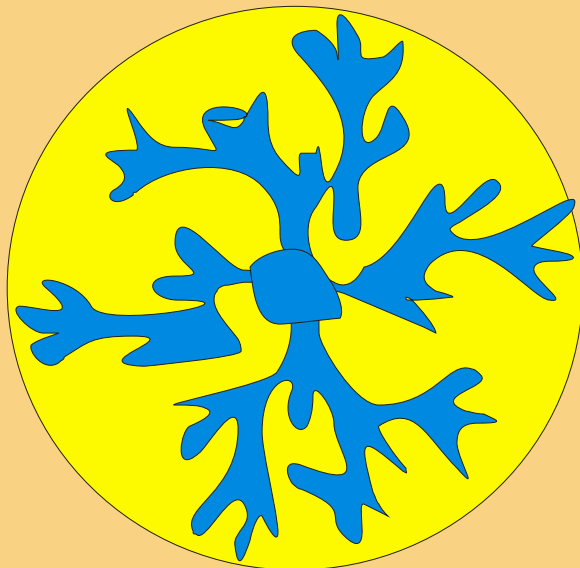
Образец	M	SiO ₂ : (CH ₃) ₃ Si	T _{ст} , °C
I-2	59300	1:0,73	>T _{разл}
I-7	4500	1:1,2	-60
II-4	93000	1:0,5	>T _{разл}
II-8	2400	1:1	0
III-2	61000	1:0,6	>T _{разл}
III-7	3300	1:1,1	-40

Изотермы монослоёв модифицированных кремнеземных частиц на основе тетраэтоксисилана



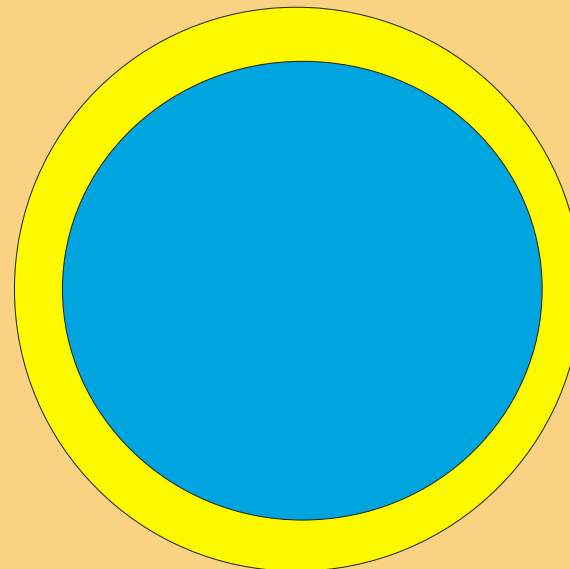
Изотермы поверхностного давления фракций кремнеземов а)2-3; б)2-2; в)2-1; г)3-1

Модели макромолекулы и частицы.



Макромолекула

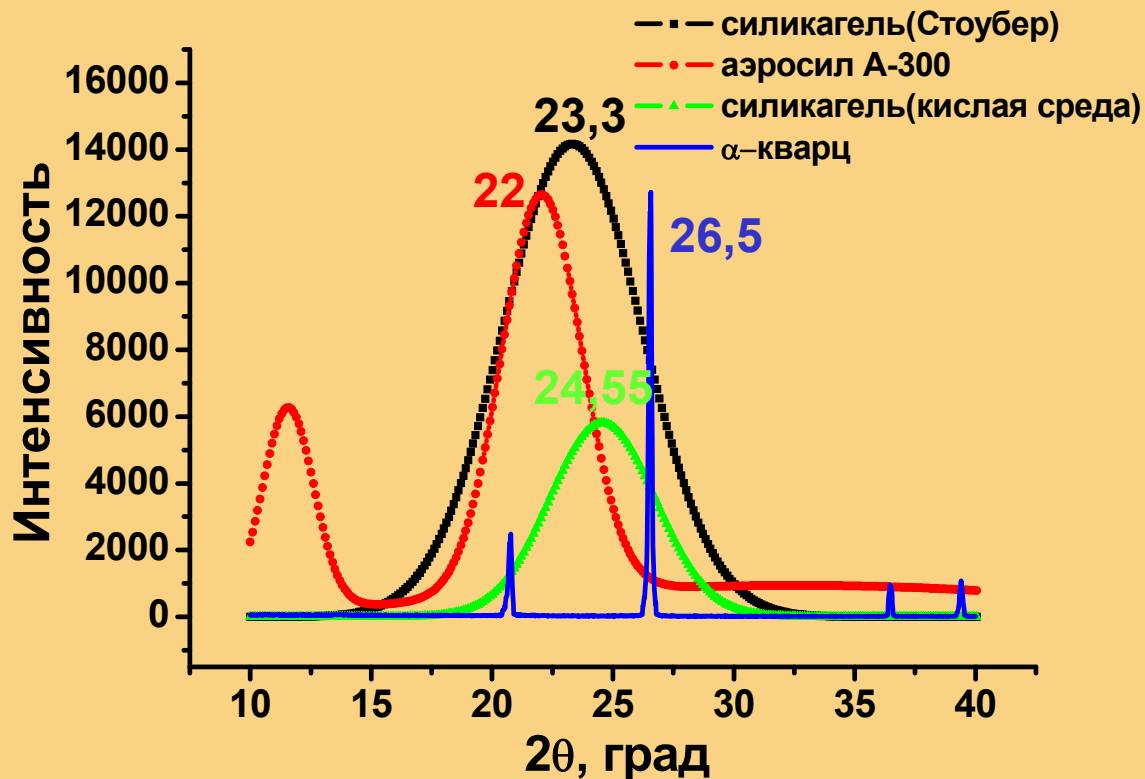
-молекулярная структура
обладает определенной
подвижностью (Тст. <Тразл.)
- полностью сольватируется
растворителем



Частица

- сольватируется только
поверхностный слой
- ядро жесткая –
частосшитая сетка
(Тст.>Тразл.)

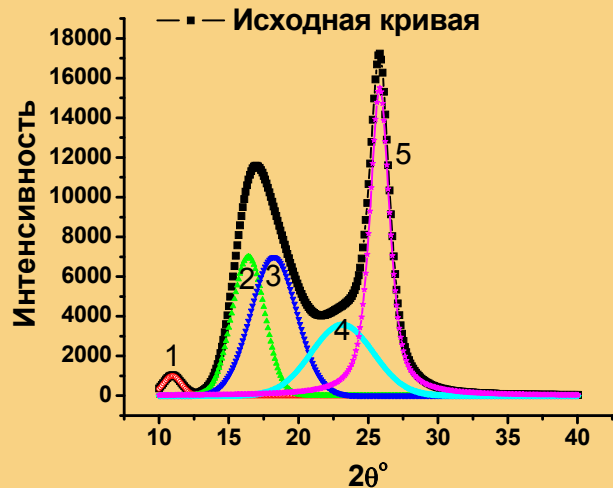
Рентгеновская дифракция в области больших углов



Основной рефлекс α-кварца	Силикагель (кислая среда)	Силикагель (Стоубер)	Аэросил А-300
26,5	24,55	23,3	22

«Полиэтоксисилоксановое» ядро – триметилсилильная оболочка

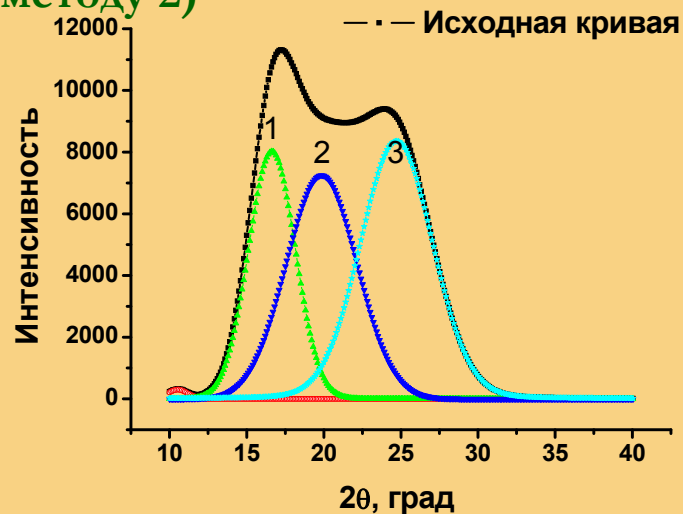
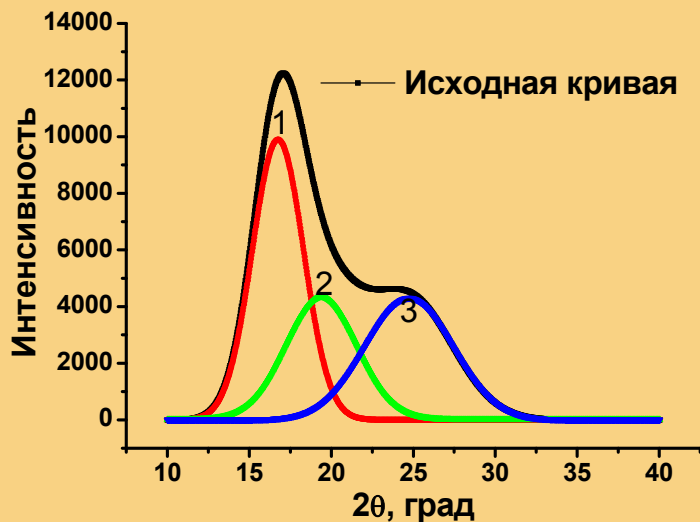
(образцы по методу 1)



Молек.масса	$T_{ст}$	Содержание SiO_2 , %	$SiO_2:Me_3Si$
59300	$>T_{разл}$	33	1:0,73

Силиказольное ядро – триметилсилильная оболочка

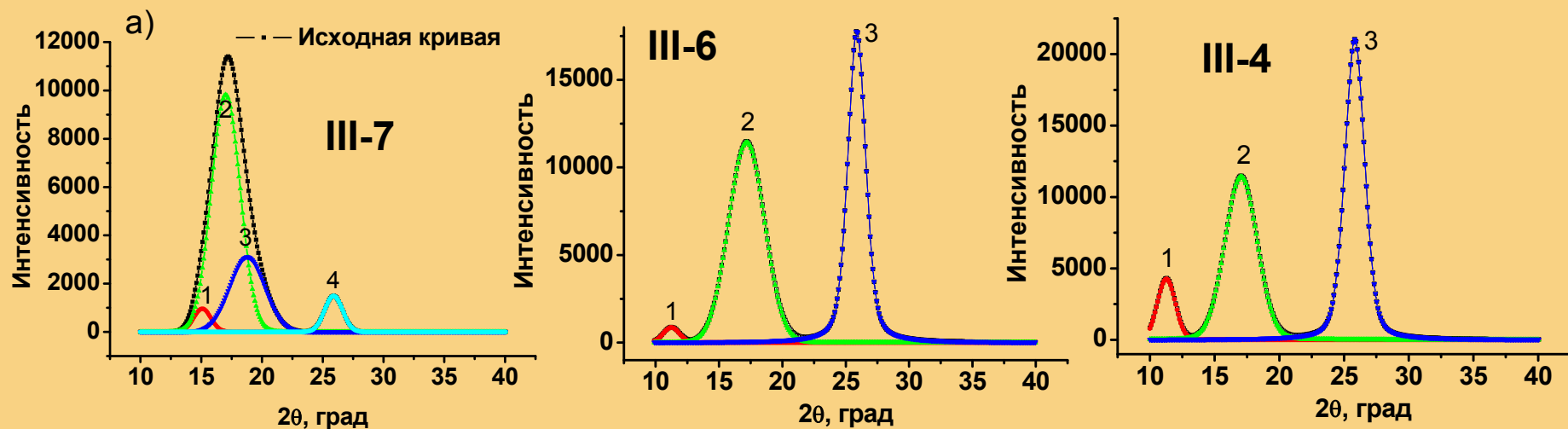
(образцы по методу 2)



Молек. масса	$T_{ст}$	C	Содержание SiO_2 , %	$SiO_2:Me_3Si$
2400	0		31,4	1:1
220000	$>T_{разл}$		41	1:1,5

Ядро из «тетраэтоксисилана» – триметилсилильная оболочка.

(образцы по методу 3)

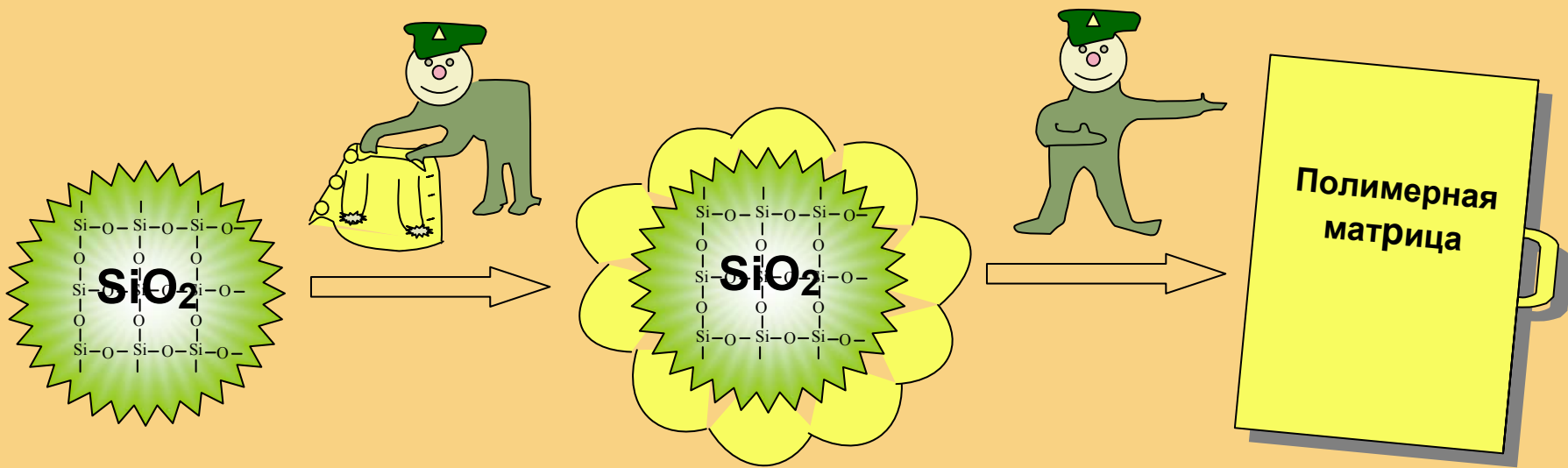


Образец	Вид	Молекулярная масса	$T_{ст}, ^\circ C$	Содержание $SiO_2, \%$	$SiO_2:Me_3Si$
III-7		3300	-50	6,2	1:1,8
III-6		7300	150	46,4	1:1
III-4		13450	$>T_{разл}$	51	1:0,8 ₅₀

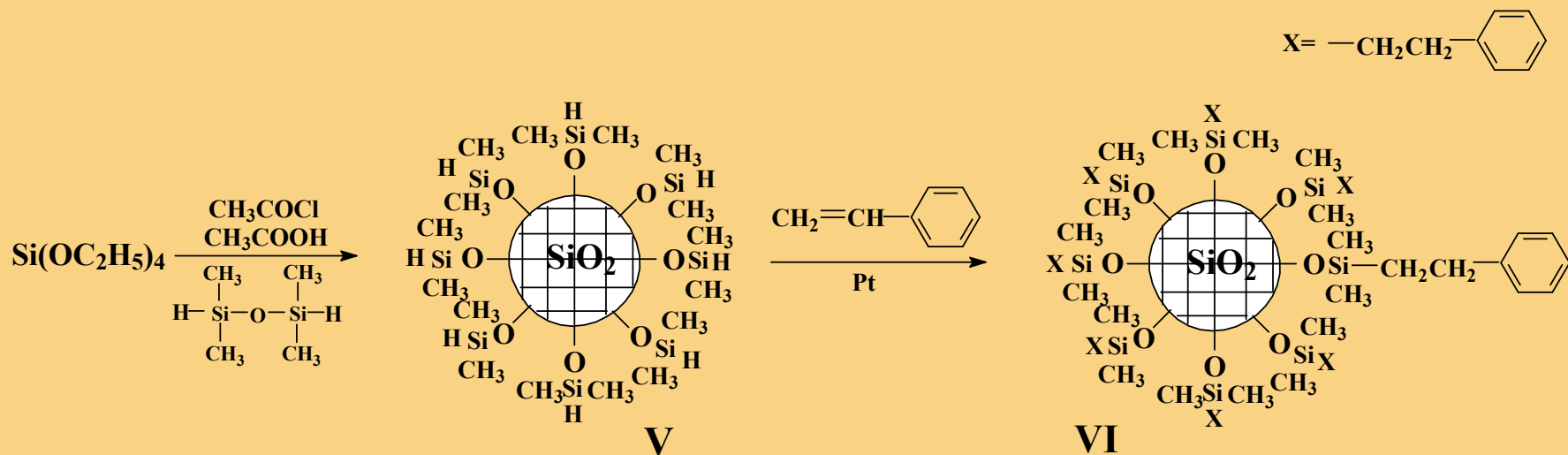
Свойства гибридных наночастиц.

Из полиэтоксисилоксана (первый метод синтеза)				
№	состояние	м.м	R, nm	T _{ст} , C
I-1-I-3	Ч	34700-152000	2-5,3	T _{ст} >T _{разл}
I-4	М-Ч	21500	1,7-2	T _{ст} >T _{разл}
I-5	М	12600	1-1,4	160
I-6	М	6650	1	-5
I-7	М	4500	<1	-60

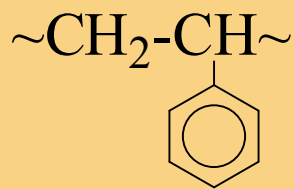
Из силиказоля (второй метод синтеза)					Из тетраэтоксисилана (третий метод синтеза)				
№	состояние	м.м	R, nm	T стекл	№	состояние	м.м	R, nm	T стекл
II-1- II-5	Ч	25000- 512000	2,4-10,2	T _{ст} >T _{разл}	III-1- III-3	Ч	26800- 116100	2,1-5,8	T _{ст} >T _{разл}
II-6	М-Ч	11500	1,1-1,5	T _{ст} >T _{разл}	III-4	М-Ч	13450	1,4-2,4	T _{ст} >T _{разл}
II-7	М	5000	1-1,2	160	III-5	М	8300	1-1,8	170
II-8	М	2400	<1	0	III-6	М	7300	1-1,3	150
II-9	М	-	<1	-80	III-7	М	3300	<1	-50



Синтез кремнезема с 2-фенилэтильными группами для создания нанокомпозитов на основе полистирола.

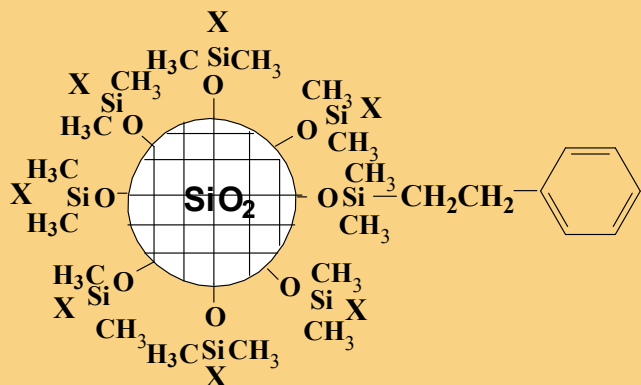


полистирол

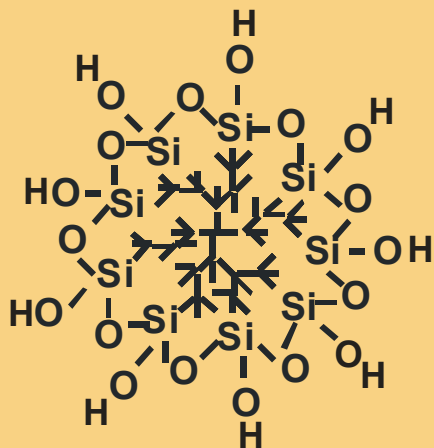


Получение нанокомпозитов на основе полистирола.

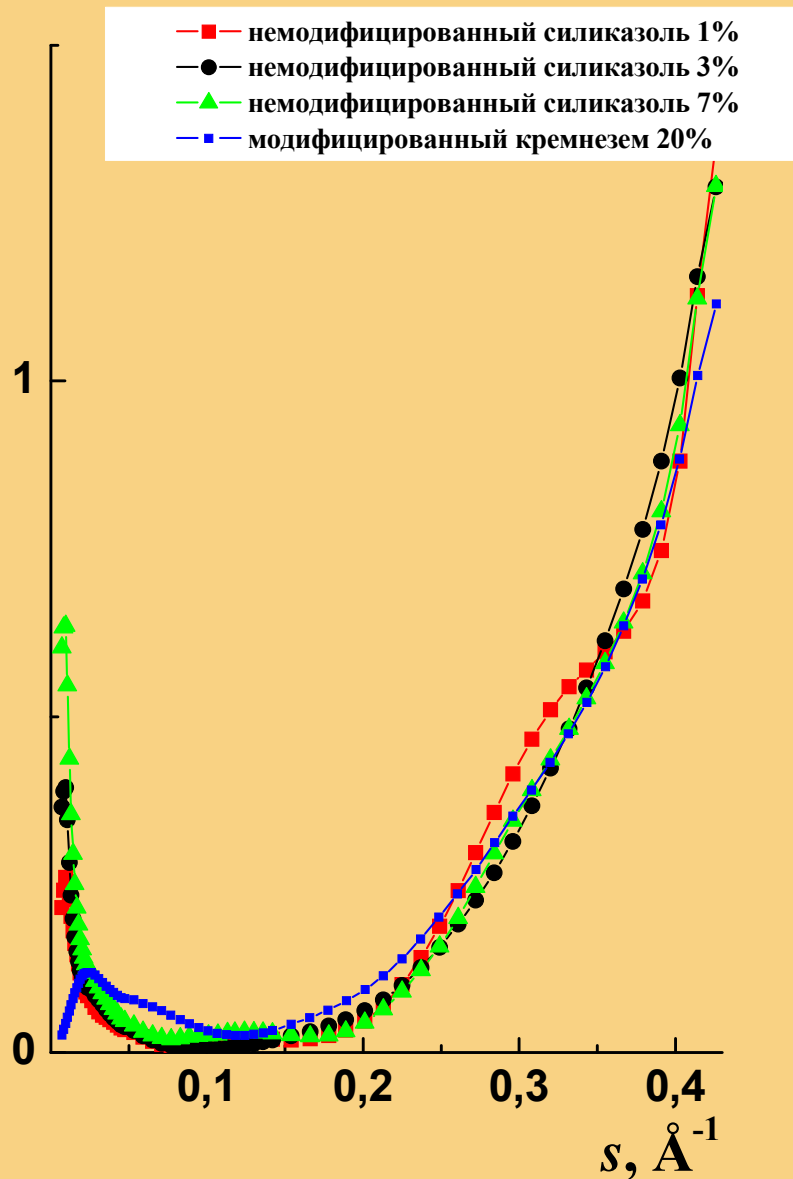
кремнезем, модифицированный
2-фенилэтильными группами

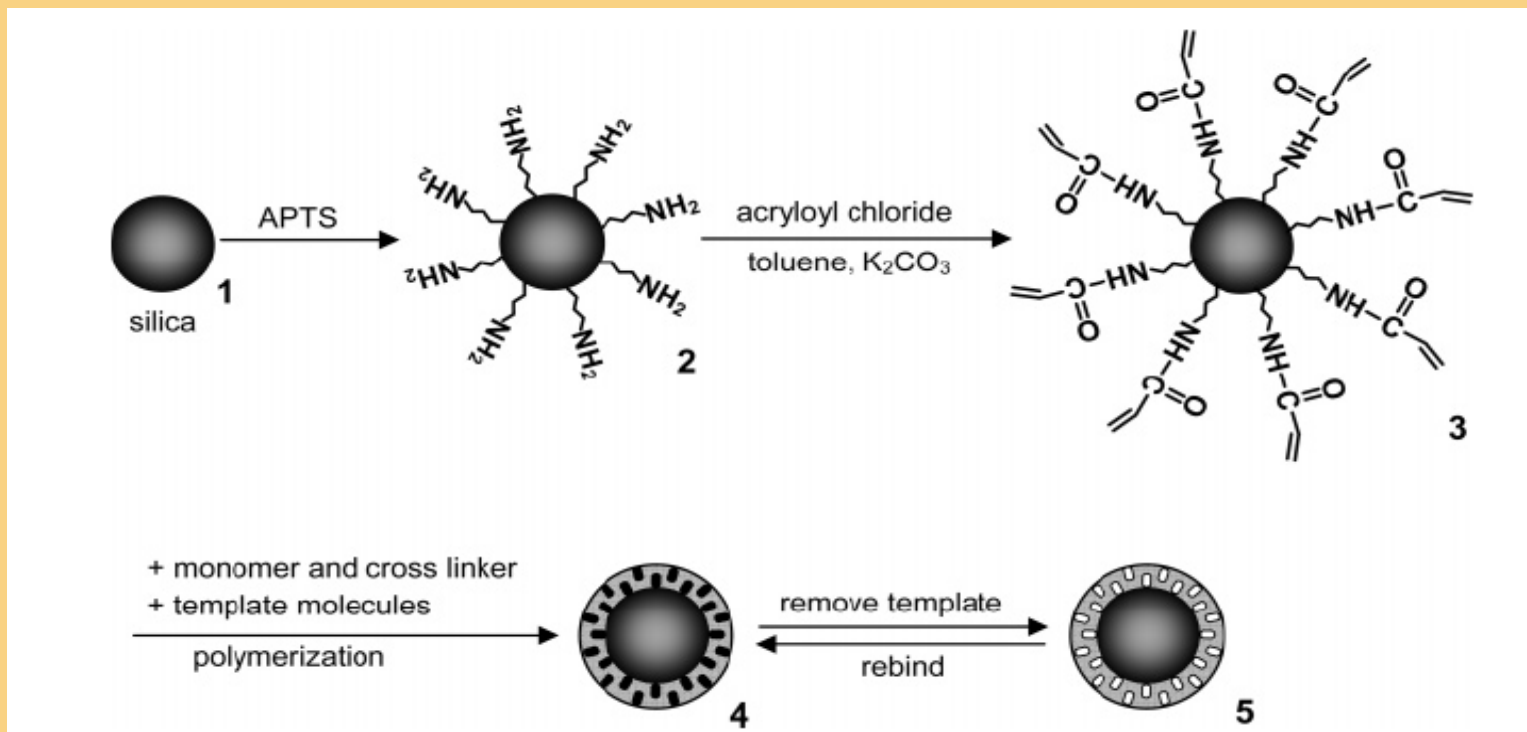


Немодифицированный силиказоль



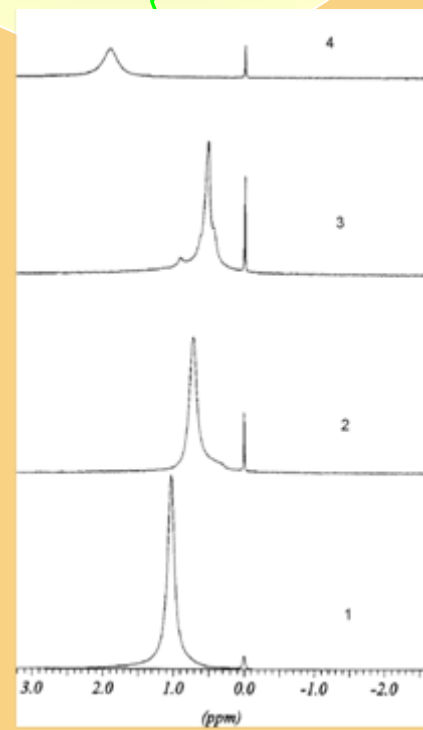
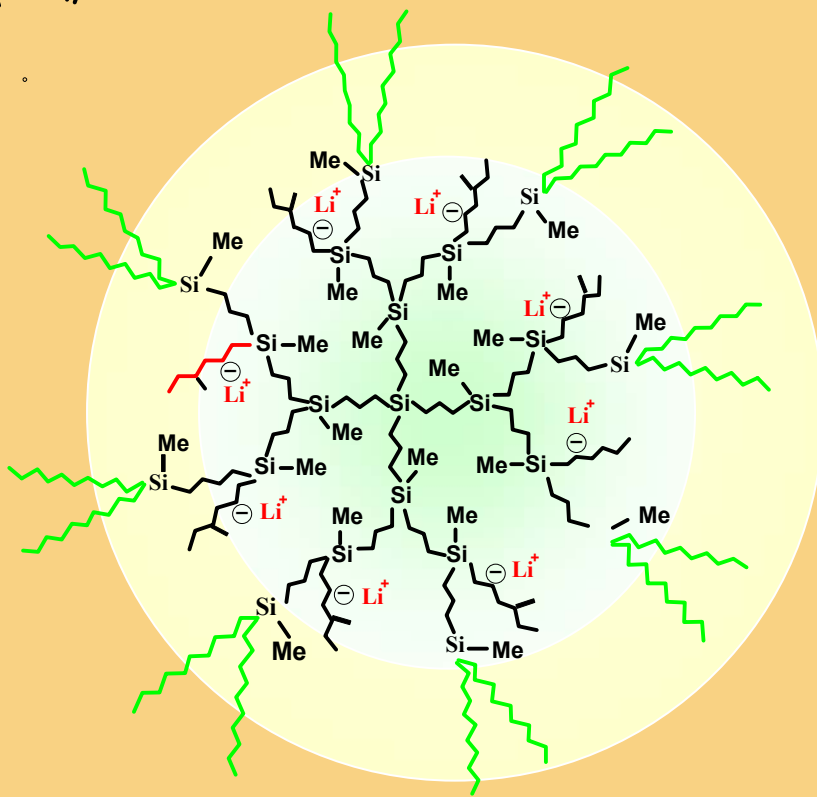
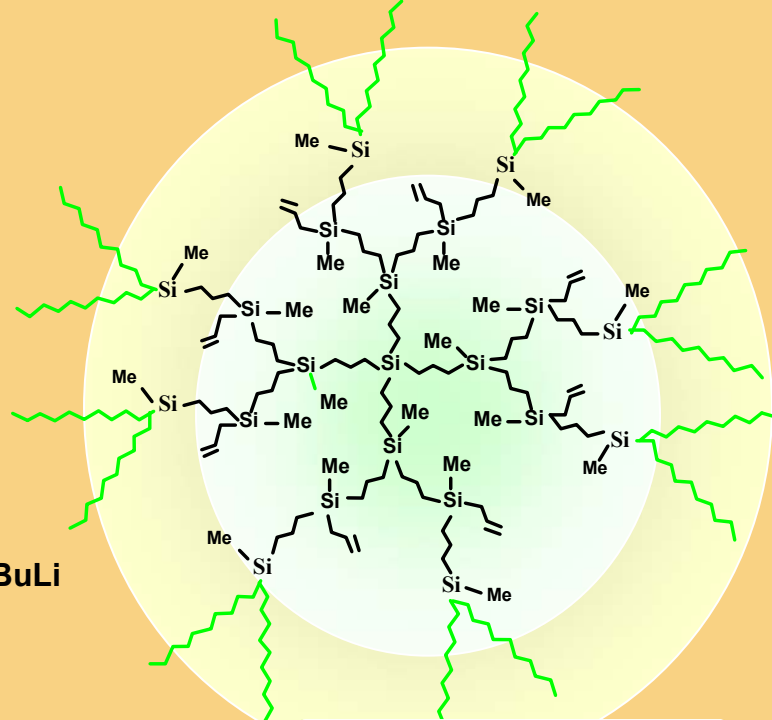
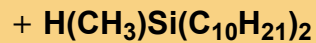
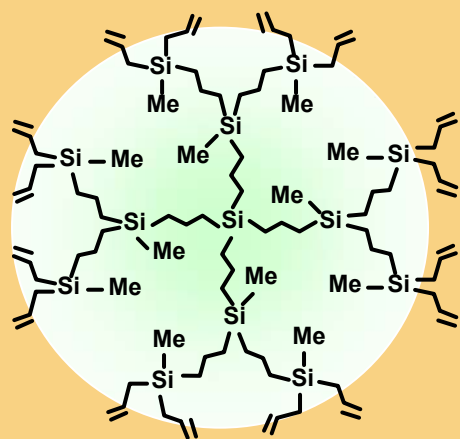
$I*s^2$



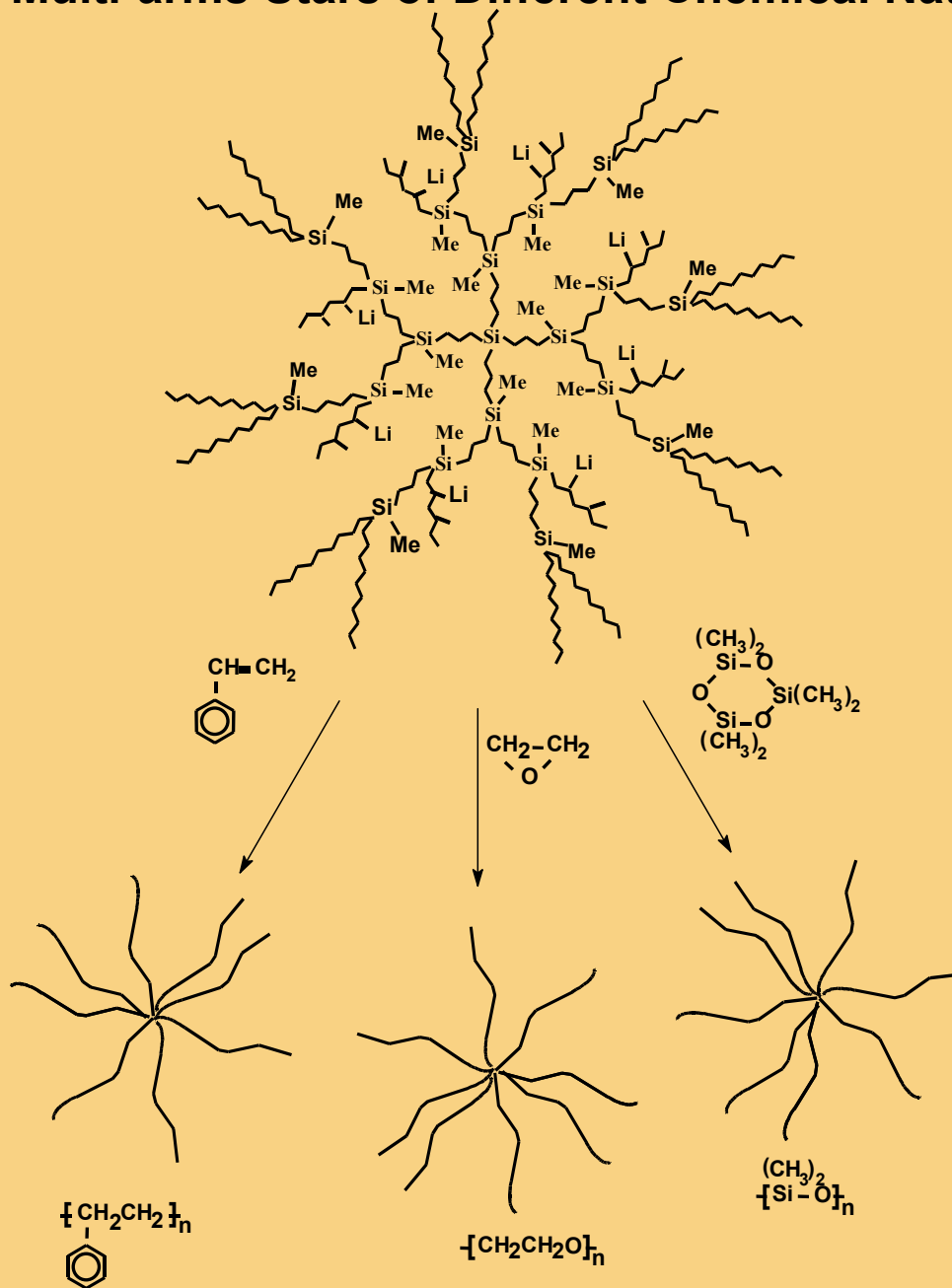


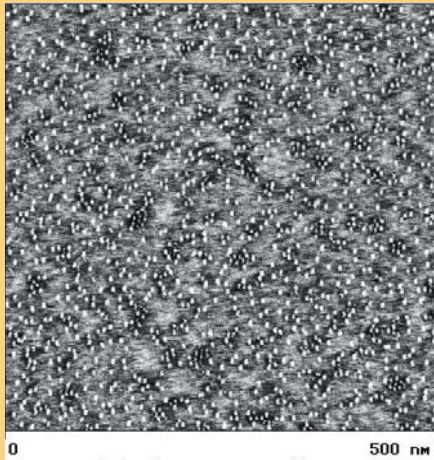
D Gao, Z Zhang, M Wu, C Xie G Guan, and D Wang A Surface Functional Monomer-Directing Strategy for Highly Dense Imprinting of TNT at Surface of Silica Nanoparticles. // J. Am. Chem. Soc. 2007, 129, 7859-7866

Synthesis of Polylithium initiators

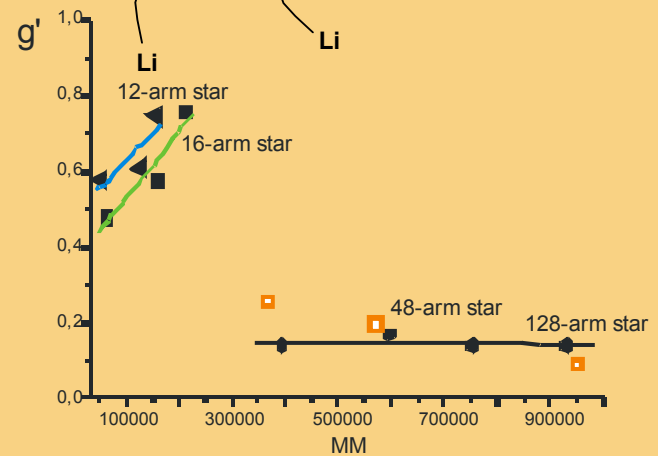
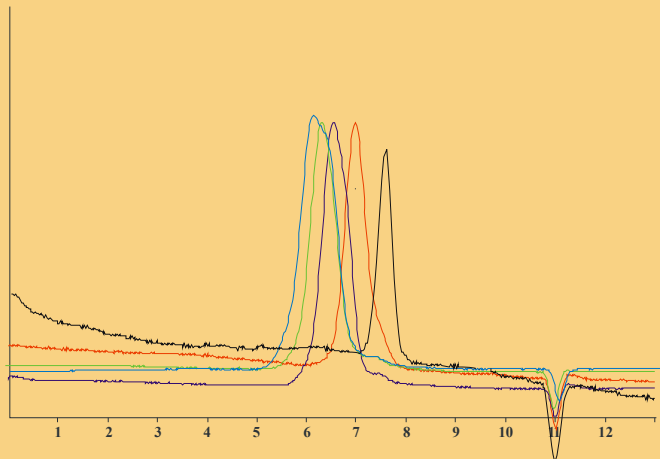
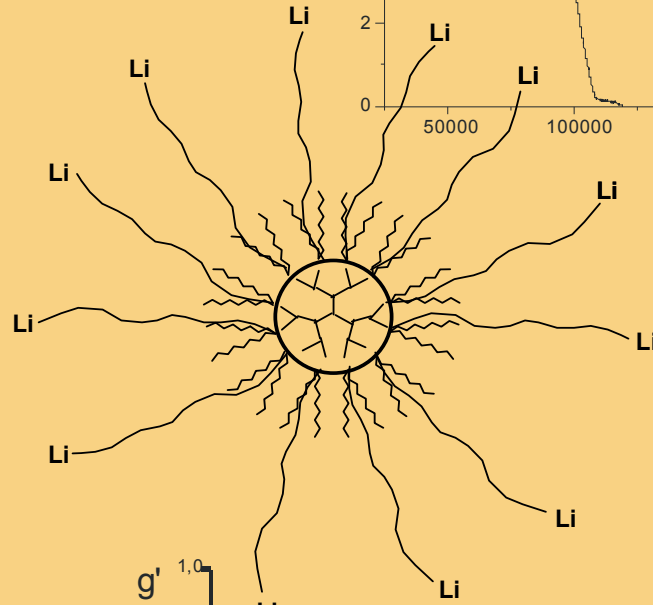
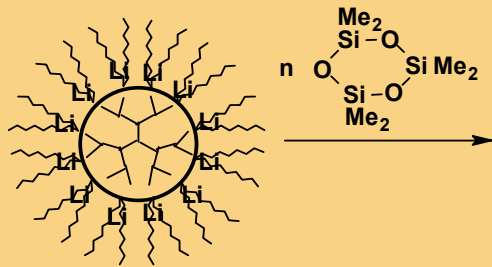
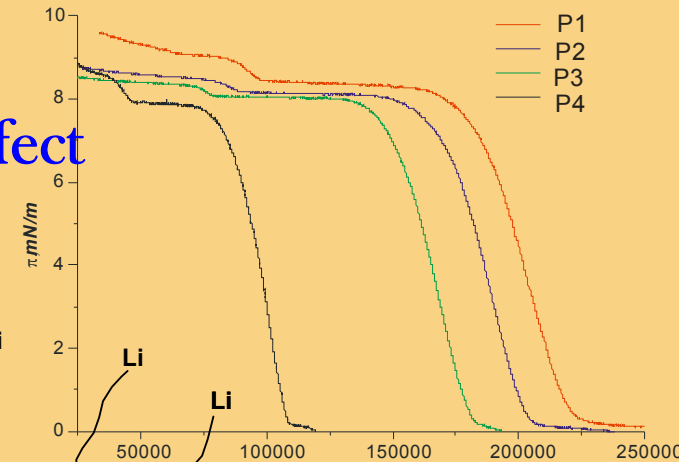


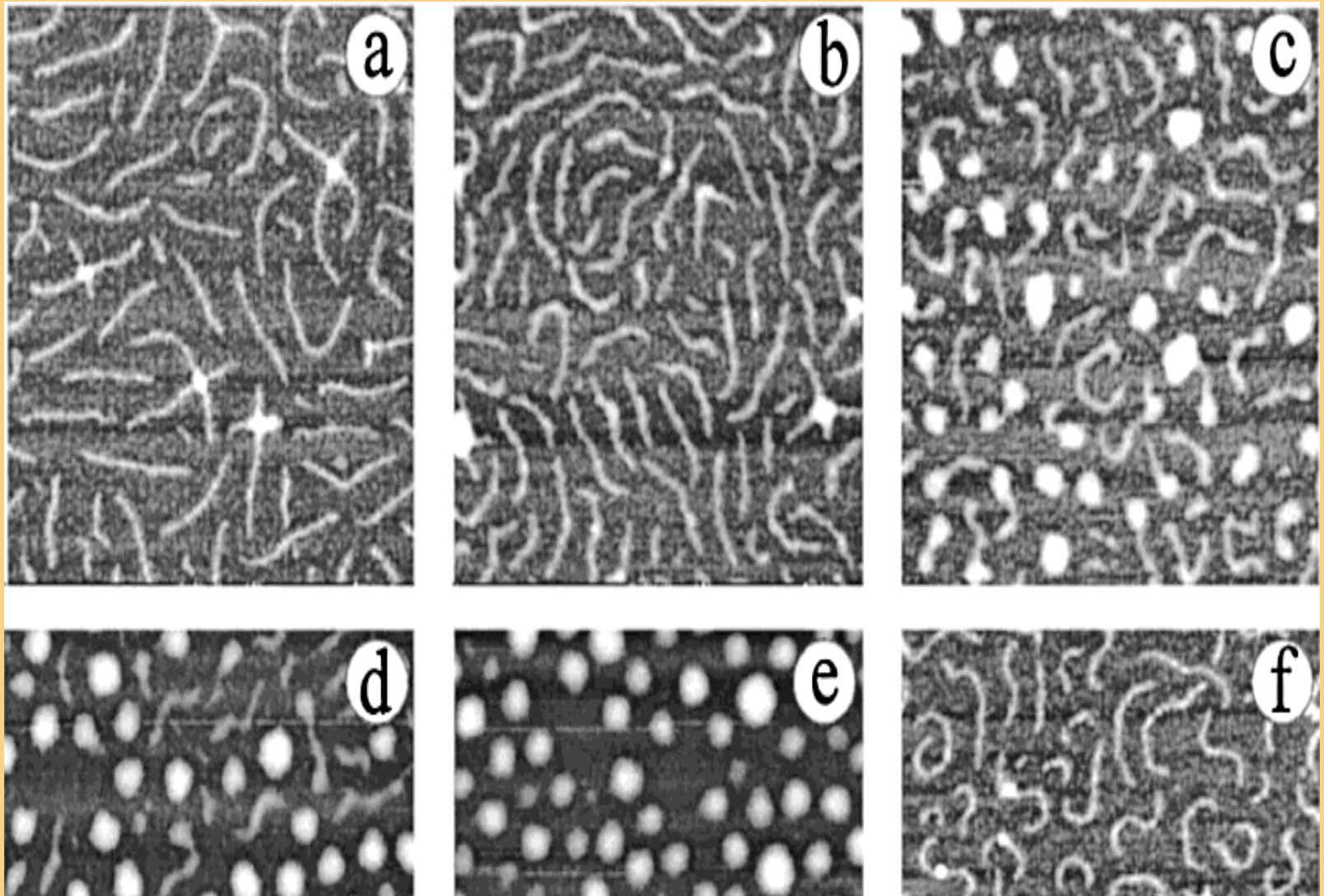
Multi-arms Stars of Different Chemical Nature





Molecular Membrane Effect





SFM micrographs of monolayers of pnBA brushes (sample A in Table 5) transferred on mica at different degrees of compression: (a) 30 \AA^2 , (b) 23 \AA^2 , (c) 21 \AA^2 , (d) 17 \AA^2 , (e) 13 \AA^2 , (f) 30 \AA^2 (after expansion).

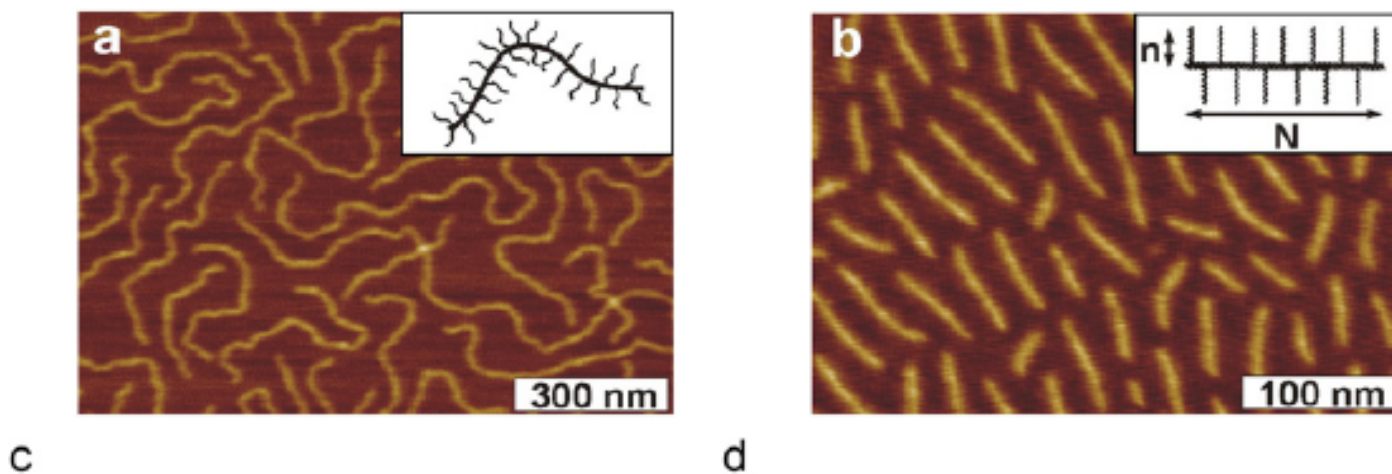
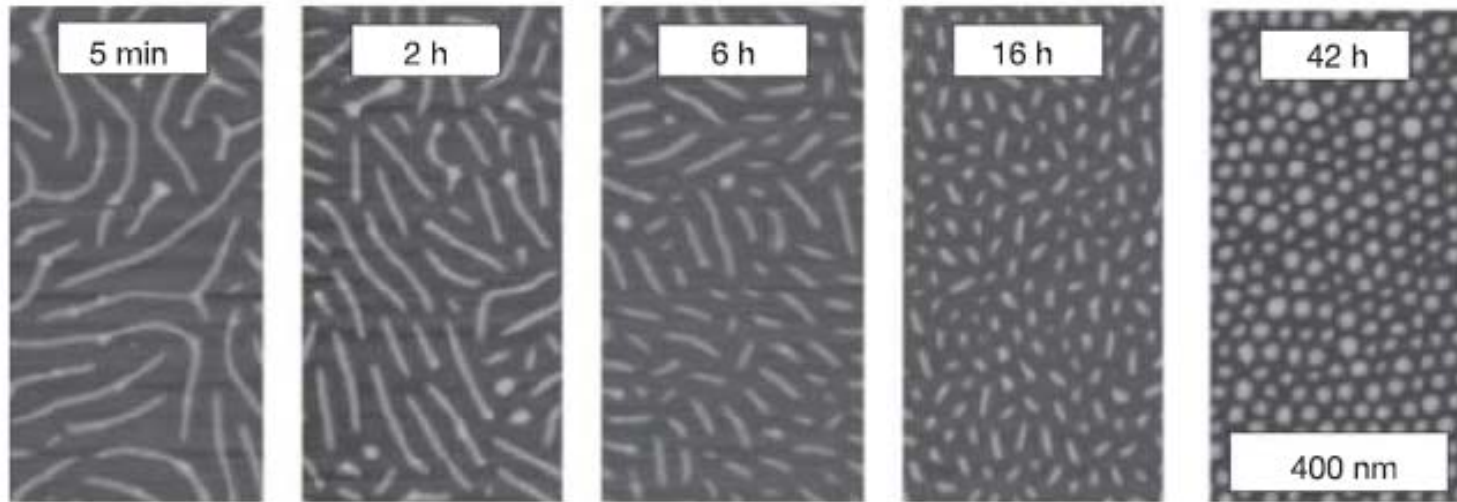
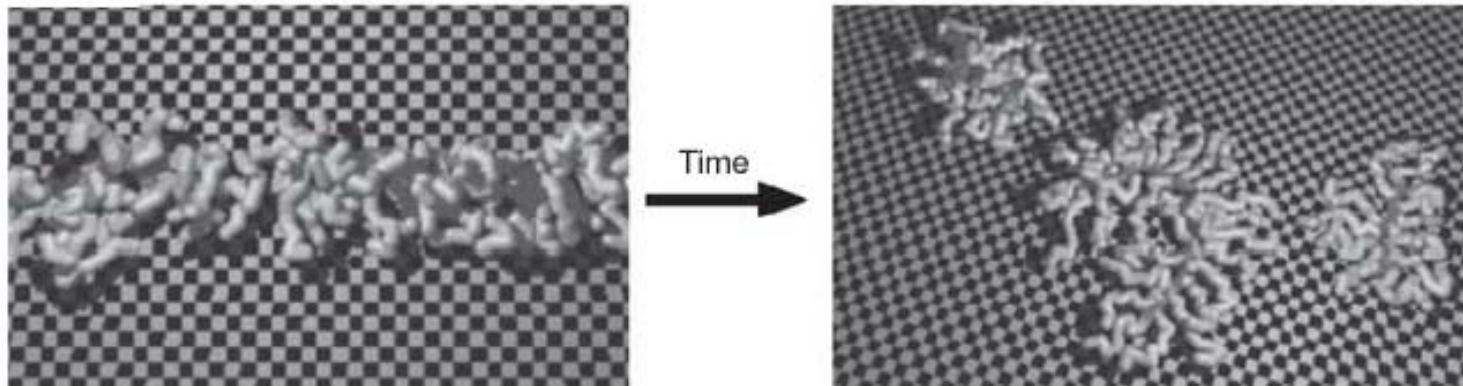


Fig. 16. Brush molecules change from (a) flexible to (b) rod-like conformation with increasing degree of polymerization of side chains n .

a**b**

Adsorption-induced degradation of macromolecules. a, The molecular degradation of brush-like macromolecules with long side chains ($n \approx 140$) on mica was monitored using AFM height imaging after each sample was exposed for different time periods (as indicated in the images) to a water/propanol (99.8/0.2wt/wt%) substrate.

Заключение

- Совокупность молекулярных нанобъектов можно рассматривать как своеобразный конструктор для создания наноматериалов и наноустройств.
- Потенциал практического применения молекулярных нанобъектов самым непосредственным образом зависит от глубины нашего понимания взаимосвязи структура - свойства для каждого из типов нанобъектов, которое, в свою очередь, подразумевает огромную работу по синтезу новых объектов и последовательному изучению их свойств