Synthesis, structure, and properties of sodium *cis*-tetraethylcyclotetrasiloxanolate and new mesomorphic *cis*-tetra[ethyl(trimethylsiloxy)]cyclotetrasiloxane

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Hydrolytic condensation of ethyltriethoxysilane in the presence of NaOH (Si : NaOH = 1) gave crystal solvate of sodium *cis*-tetraethyltetrasiloxanolate $\{(Na^+)_4[EtSi(O)O^-]_4\}\cdot nL$ (**1**, L = EtOH, H_2O) in high yield. The molecular structure of compound **1** at L = EtOH, $n = 8$ was determined by X-ray diffraction analysis. The reaction of compound **1** with trimethylchlorosilane affords *cis*-tetra[ethyl(trimethylsiloxy)]cyclotetrasiloxane *cis*-$[EtSi(O)OSiMe_3]_4$, which is the first representative of the group of mesomorphic cyclotetrasiloxanes containing alkyl groups.

Key words: hydrolytic condensation, ethyltriethoxysilane, alkali organosiloxanolates, stereoregular organocyclosiloxanes, trimethylsilylation, molecular structure, crystal structure, mesomorphic state, phase transitions, plastic crystals, differential scanning calorimetry, X-ray diffraction analysis.

In the previous studies[1,2] of the hydrolytic condensation of phenyltrialkoxysilanes in the presence of alkaline and/or transition metal ions, we have found that this complicated multistep process can be directed to the formation of one individual compound, metallasiloxane, whose molecule contains only one type of the stereoregular cyclosiloxanolate fragment of a certain size, which is fixed on a metal ion matrix.[3—10] Removal of metal ions by the reactions with triorganylchlorosilanes or a dilute solution of hydrochloric acid made it possible to obtain a whole series of new stereoregular cyclosiloxanes with interesting and sometimes unique physical properties.[11—23] We have synthesized and characterized[14—19] the whole class of new mesomorphic cyclosiloxanes, whose mesomorphic properties depend on both the cycle size and character of side groups.

In particular, we have shown[14,17] that the replacement of some Ph groups in octaphenylcyclotetrasiloxane (OPhCTS) $[(Ph)_2SiO]_4$ by the Me_3SiO groups substantially extends the temperature region where the plastic-crystalline phase exists. Thus, the broken "up-and-down" symmetry inherent in *cis*-$[PhSi(O)OSiMe_3]_4$ molecules does not suppress the mesomorphic properties and, moreover, substantially extends the temperature region of its existence. Therefore, it was of interest to synthesize and study a series of cyclotetrasiloxanes with the broken "up-and-down" symmetry in which the Ph groups are substituted for aliphatic groups.

In the present work, we synthesized and studied the structures and properties of two new compounds: crystal solvate sodium *cis*-tetraethyltetrasiloxanolate $\{(Na^+)_4[EtSi(O)O^-]_4\}\cdot nL$ (**1**, L = EtOH, H_2O) and *cis*-tetra[ethyl(trimethylsiloxy)]cyclotetrasiloxane *cis*-$[EtSi(O)OSiMe_3]_4$ (**2**) synthesized from compound **1** by trimethylsilylation[24] and being the first representative of the group of mesomorphic cyclotetrasiloxanes with alkyl groups.

Results and Discussion

Synthesis and molecular and crystal structures of compound 1 at L = EtOH, $n = 8$. *cis*-Compound **1** was synthesized by the hydrolytic condensation of ethyltriethoxysilane in the presence of an equimolar amount of sodium hydroxide according to Scheme 1.

The hydrolytic condensation of ethyltriethoxysilane in the presence of an equimolar amount of NaOH in an alcoholic medium[3,5,7] can produce alkaline intermediate **A** containing an ion pair due to the simultaneous occurrence of at least seven reactions and fast proton-

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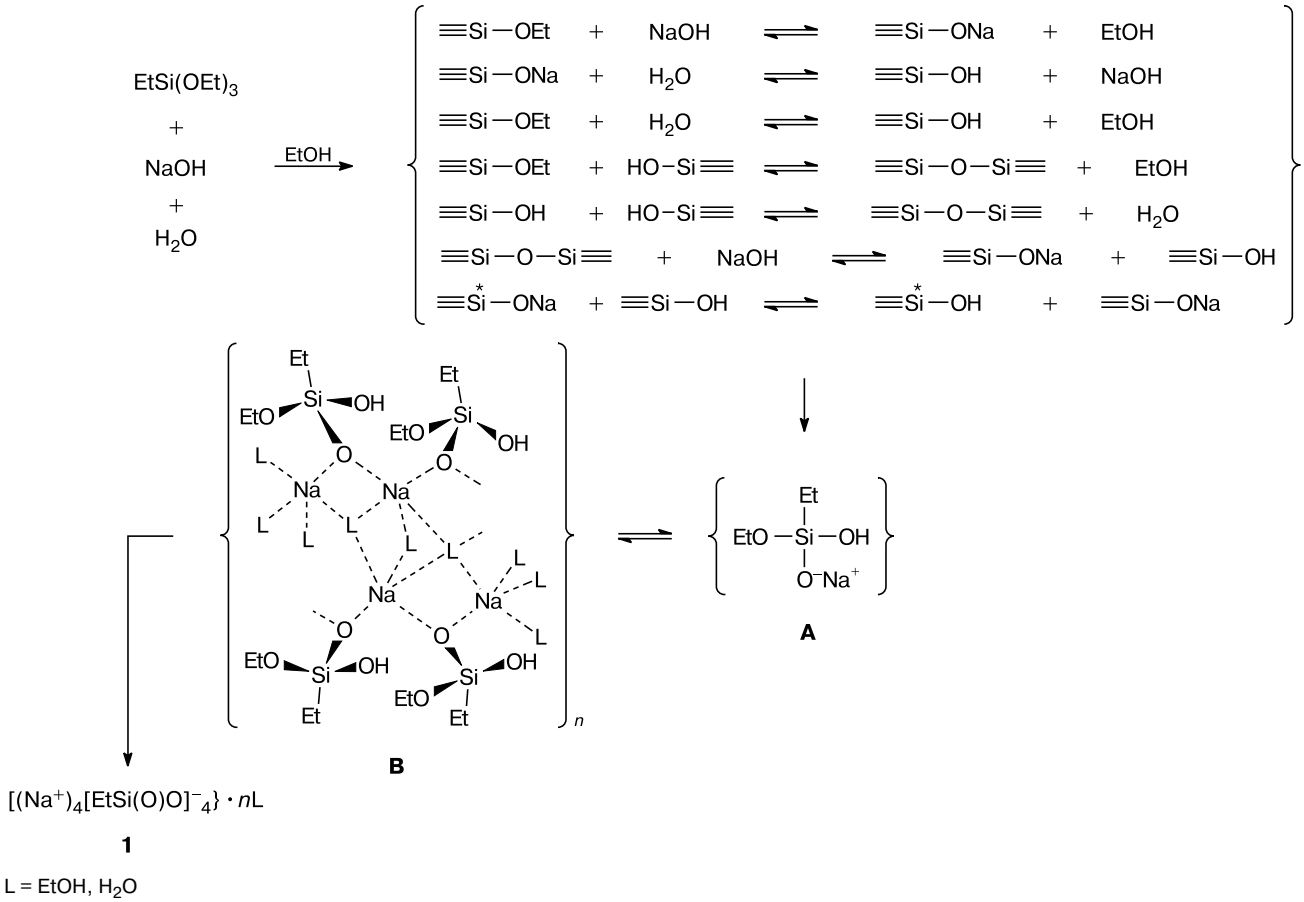
Scheme 1



cation exchange. In an organic medium these intermediates associate to form aggregates **B** in which the ionic component solvated by alcohol and water molecules is framed by organosilicon fragments containing functional groups. Under appropriate conditions, condensation occurs at these functional groups and is accompanied by oligocyclization.[7] At the ratio alkoxysiloxane : water : NaOH = 1 : 1 : 1 in EtOH, sodium ethylsiloxanolate is formed, whose main fragment is the *cis*-cyclotetrasiloxanolate anion bonded with four Na^+ cations.

The X-ray diffraction study of crystal **1** at L = EtOH, $n = 8$ showed that the independent part of the cell contains one eight-membered siloxanolate cycle, four Na^+ cations, and eight EtOH solvate molecules (Fig. 1). In crystal the siloxanolate cycle is in a special position at 2-fold axis. The siloxanolate cycle has a boat conformation, whose base is formed by the Si(1), Si(1A), O(2), and O(2A) atoms with a root-mean-square deviation from the plane of 0.03 Å, and the Si(2), O(1), Si(2A), and O(2A) atoms shift from the plane by 0.8—0.9 Å. The Si—O distances in the cycle range from 1.636(1) to 1.652(1) Å, and the *exo*-bond lengths are in the interval 1.596(1)—1.598(1) Å. The Si—O—Si and Si—O—Si angles are 138.83(8)—142.63(8) and 107.13(6)—108.61(6)°, respectively. The Na^+ cations form contacts with both the siloxanolate O atoms (Na...O, 2.290(1)—2.544(1) Å) and the O atoms of the EtOH solvate molecules (Na...O, 2.245(1)—2.573(1) Å). The coordination number of sodium is five. The EtOH solvate molecules form rather strong hydrogen bonds (O...O distances 2.572(2)—2.649(2) Å) with the siloxanolate O atoms. One of the siloxanolate atoms, namely, O(3), forms no hydrogen bonds, whereas the O(4) atom is involved in two O—H...O bonds.

Unlike the earlier studied salts with the six-membered cyclic anions,[7] whose crystals are characterized by double layers with the hydrophobic coating formed by the cations and anions, in crystal **1** at L = EtOH, $n = 8$ the anions are joined by the Na^+ cations due to the Na...O coordination bonds into tubes with the hydrophobic coating formed by the Et groups (Fig. 2).

Synthesis, phase transitions, and mesomorphic properties of compound 2. *cis*-Tetra[ethyl(trimethylsiloxy)]cyclotetrasiloxane (**2**) was synthesized by the treatment of compound **1** with trimethylchlorosilane in the presence of pyridine as HCl acceptor according to Scheme 2.

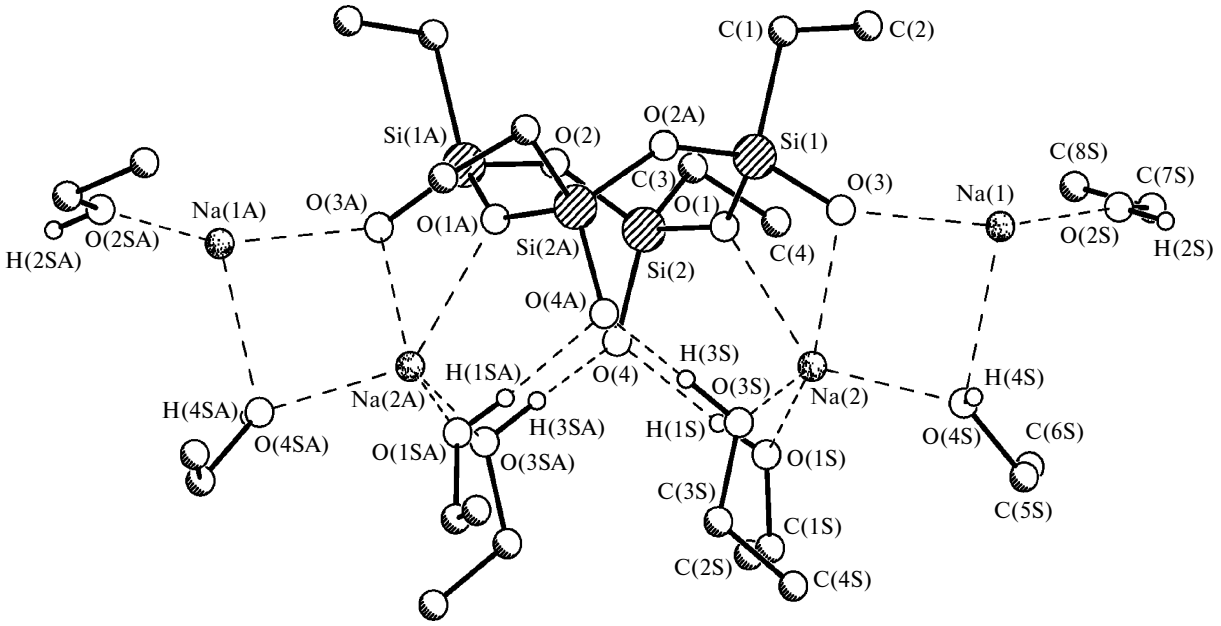


Fig. 1. General view of the siloxanolate eight-membered cycle and its nearest environment in crystal **1** at L = EtOH, $n = 8$.

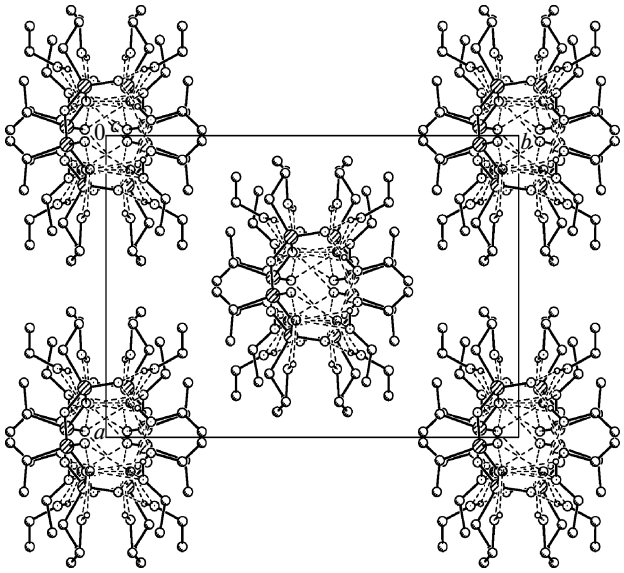


Fig. 2. Fragment of the crystal packing of compound **1** at L = EtOH, $n = 8$.

Scheme 2

$$\mathbf{1} + Me_3SiCl \xrightarrow[-HCl \cdot Py]{Py,\ C_6H_{14}} [EtSi(O)OSiMe_3]_4$$

2

Thermotropic changes in the phase state of compound **2** were studied by DSC, X-ray diffraction analysis, and polarization microscopy.

The thermogram of heating compound **2** (Fig. 3, curve *1*), which was obtained by sublimation *in vacuo*, contains two endotherms: a broad peak with a minimum at −17 °C and a narrow peak at 88 °C. The calculated heats of the transitions (ΔH) are 19 and 4 J g^{-1}, respectively. The low ΔH value for the second transition and the waxy appearance of compound **2** at 20 °C suggest that the second peak corresponds to mesophase melting and the transition to the isotropic state. At the same time, the ΔH value for the first *endo*-effect means that it corresponds to the crystal—mesophase transition. On cooling sample **2** with a rate of 20 deg min^{-1}, the maximum of heat release upon the melt—mesophase transition was detected at 79 °C (Fig. 3, curve *3*). The formation of the crystal structure requires more substantial overcooling: the exotherm corresponding to crystallization is localized at −55 °C (difference between the melting and crystalli-

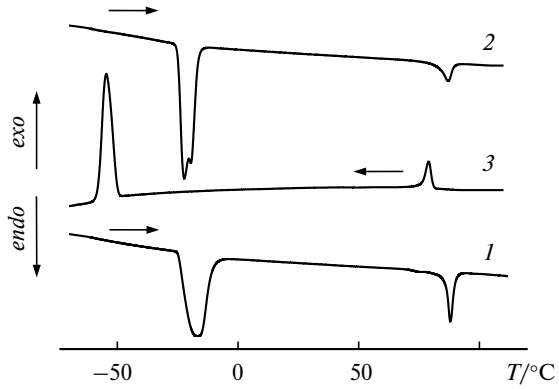


Fig. 3. DSC curves for compound **2** for the first (*1*) and second (*2*) heating and on cooling (*3*); heating and cooling rate 20 deg min^{-1}.

zation points is 38 °C). Upon repeated heating of the sample, the DSC thermogram (Fig. 3, curve *2*) exhibits melting of the crystalline phase formed under these conditions, which is two-step with a total melting heat of 18.5 J g^{-1}. The DSC thermogram distinctly manifests two minima (at −22 and −19 °C), whose positions in the temperature scale and the area ratio remain unchanged with a decrease in the heating rate from 20 to 5 deg min^{-1}. The position of the second endotherm in the temperature scale remains unchanged, being 88 °C, but the heat of the mesophase—melt transition decreases to 2.2 J g^{-1}, indicating that the prehistory of mesophase formation affects its perfection. The low-temperature peak can be bifurcated due to two factors: formation (upon cooling from the melt) of crystallites with substantially different sizes and perfection.

At 20 °C, the diffraction pattern of compound **2** (Fig. 4, curve *1*) is typical of plastic crystals; it contains a narrow ($\Delta_{1/2}$ = 0.17°) peak at 2θ = 9.59° and two low-intensity reflections at 2θ = 13.58 and 16.65°. The position of reflections of the mesophase is very sensitive to the temperature increase: for the first peak at T = 70 °C 2θ = 9.29° (see Fig. 2, curve *2*). The $\sin^2\theta$ values for the adjacent peaks detected at 20 °C relate to each other as 1 : 2 : 3, and this assumes a cubic symmetry for the mesophase. The latter was confirmed by optical polarization microscopy: at 20 °C compound **2** is optically isotropic. The reflections were indexed in all possible types of the cubic cell. The packing coefficient (k) served as a criterion for choosing the cell type.[25] For compound **2** only the packing coefficients calculated for the bcc cell give good coincidence with the real k values for the mesomorphic state.

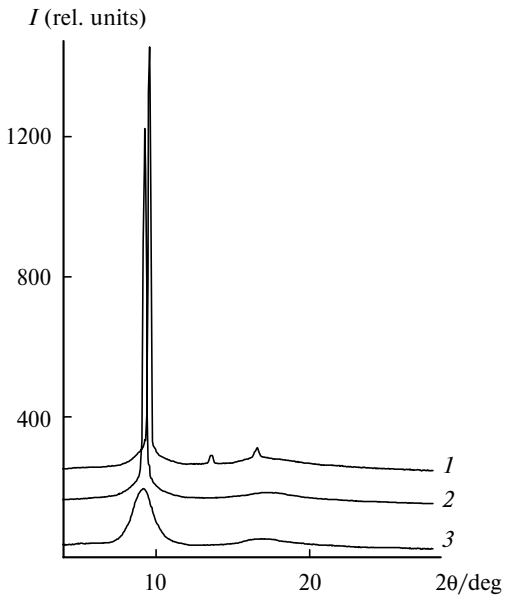


Fig. 4. Diffraction patterns of compound **2** at 20 (*1*), 75 (*2*), and 90 °C (*3*).

The peaks observed in the mesophase for compound **2** can be indexed as reflections 110, 200, and 211 of the three-dimensional bcc lattice. The *cis*-$[EtSi(O)OSiMe_3]_4$ cell contains two molecules; at T = 20 °C a = 13.03±0.05 Å, V = 2210.74 $Å^3$, k = 0.58; at T = 70 °C, *i.e.*, near the isotropization point, a = 13.45±0.05 Å, V = 2431.98 $Å^3$, k = 0.53.

According to the X-ray diffraction data (see Fig. 4, curve *3*), this compound is transformed from the mesomorphic into isotropic state at ~90 °C, which is consistent with the DSC data (see Fig. 3). After isotropization, the diffraction patterns exhibit only two broad maxima: one relatively narrow halo at 2θ = 9.16° ($\Delta_{1/2}$ = 1.5°, d = 9.64 Å) and the second very broad and low-intensity halo at 2θ = 14—26°.

Thus, when the Ph group in *cis*-$[PhSi(O)OSiMe_3]_4$ is substituted for the Et group, *i.e.*, on going to compound **2**, the temperature region of mesophase existence becomes narrower and shifts to the low-temperature region. On heating compound **2**, the mesophase region ranges from 17 to 88 °C, and for *cis*-$[PhSi(O)OSiMe_3]_4$ this region is 78—260 °C.

Comparison of cyclotetrasiloxane **2** under study and octaethylcyclotetrasiloxane $[(Et)_2SiO]_4$ (which, according to published data,[26] is not mesomorphic and represents a liquid at 20 °C) shows that the replacement of some Et groups in a $[(Et)_2SiO]_4$ molecule by the Me_3SiO groups leading to the loss of the "up-and-down" symmetry by the molecule results in the appearance of the mesomorphic properties. This agrees well with the data on substitution of some Ph groups in the $[Ph_2SiO]_4$ molecule for the Me_3SiO groups,[14,17] due to which the temperature region of the mesophase existence is considerably extended. It can be concluded that the absence of the "up-and-down" symmetry in molecules of cyclotetrasiloxanes is one of the factors favoring the enhancement of stability of the mesomorphic state.

Experimental

Ethyltriethoxysiloxane and trimethylchlorosilane (Aldrich) and NaOH (analytical grade) were used. Solvents were purified using earlier described procedures.[27]

^1H, ^{13}C, and ^{29}Si NMR spectra were recorded on a Bruker AMX spectrometer (^1H, 400 MHz; ^{13}C, 100.1 MHz; ^{29}Si, 79 MHz) at 20 °C in $CDCl_3$.

DSC studies were carried out under argon on a Mettler-822e differential scanning calorimeter with a heating rate of 20 deg min^{-1}.

A Dron-3M diffractometer (Cu-Kα radiation, bent quartz single crystal as a focusing monochromator) equipped with a high-temperature chamber (accuracy of temperature maintenance ±1°) was used for X-ray diffraction study. Diffraction patterns were obtained using a transmission mode.

Crystal solvate of sodium *cis*-tetraethylcyclotetrasiloxanolate (1). Anhydrous EtOH (60 mL), NaOH (3.79 g, 94.7 mmol),

and H_2O (1.7 mL, 94.7 mmol) were charged into a three-neck flask equipped with a stirrer and a reflux condenser with a calcium chloride tube. The reaction mixture was stirred for 15 min at 40 °C, and then a solution of ethyltrimethoxysilane (14.23 g, 94.7 mmol) in anhydrous EtOH (30 mL) was added. The reaction mixture was stirred under reflux for 30 min more. The resulting solution was concentrated to a volume of 60 mL on a rotary evaporator (40 °C, 10 Torr). After 1 day at +4 °C, colorless needle-like crystals precipitated from the solution. The crystals were filtered off, washed with hexane, and dried on a rotary evaporator (40 °C, 10 mm Hg) to a constant weight. A white crystalline powder was obtained in a yield of 9.34 g (52.8%). Found (%): C, 16.64; H, 5.14; Na, 11.18; Si, 15.29. $\{(Na^+)_4[EtSi(O)O^-]_4\}\cdot EtOH\cdot 14H_2O$, $C_{10}H_{54}Na_4O_{23}Si_4$, molecular weight 746.0. Calculated (%): C, 16.08; H, 7.29; Na, 12.31; Si, 15.04.

It should be mentioned that mainly all metallasiloxanes contain solvate molecules of alcohol or water, whose amounts in the single crystal and in the whole crystalline mass obtained after drying never coincide. In this case, alcohol molecules are often substituted for water molecules, whereas the metallasiloxane molecule itself remains unchanged. The number of solvate molecules of alcohol and water can change from entry to entry. Therefore, excess trimethylchlorosilane and pyridine is always used for trimethylsilylation to avoid side reactions.

***cis*-Tetra[ethyl(trimethylsiloxy)]cyclotetrasiloxane (2).** A waxy substance (6.4 g, 79.0%) was synthesized from compound **1** (9.34 g, 12.5 mmol), trimethylchlorosilane (27.2 g, 0.25 mol), and pyridine (15.8 g, 0.2 mol) in hexane (125 mL). Found (%): C, 36.88; H, 8.58; Si, 34.66. $[EtSi(O)OSiMe_3]_4$, $C_{20}H_{56}O_8Si_8$, molecular weight 649.3. Calculated (%): C, 36.99; H, 8.69; Si, 34.60. ^{1}H NMR, δ: 0.13 (s, 9 H, $OSiMe_3$); 0.45—0.53 (q, 2 H, $C\underline{H}_2—CH_3$); 0.92—0.97 (t, 3 H, $CH_2—C\underline{H}_3$); the ratio of integral intensities of signals from protons of the $OSiMe_3$, $C\underline{H}_2—CH_3$, and $CH_2—C\underline{H}_3$ groups being 9 : 2 : 3. ^{13}C NMR, δ: 1.90 (s, $OSiMe_3$); 6.11, 7.10 (both s, Et). ^{29}Si NMR, δ: 8.5 (s, $OSiMe_3$); –67.4 (s, O_3SiEt).

Table 1. Selected crystallographic data and refinement parameters for crystal **1** at L = EtOH, $n = 8$

Parameter	Value
Empirical formula	$C_{24}H_{68}Na_4O_{16}Si_4$
Molecular weight	817.10
Crystal system	Monoclinic
Space group	$C2/c$
T/K	120
Z (Z')	4 (0.5)
a/Å	12.847(1)
b/Å	18.156(16)
c/Å	19.1424(15)
β/deg	97.056(3)
V/Å^3	4431.3(6)
d_{calc}/g cm^{-3}	1.225
Linear absorption, μ/mm^{-1}	2.29
F(000)	1760
$2\theta_{max}$/deg	60
Number of measured reflections	17539 (0.0215)
Number of independent reflections	6348
Number of reflected parameters	227
Number of reflections with $I \geq 2\sigma(I)$	5040
R_1	0.0451
wR_2	0.1146
GOOF	1.009
Residual electron density/e Å^{-3}, ρ_{max}/ρ_{min}	0.989/–0.607

X-ray diffraction study of compound 1 at L = EtOH, $n = 8$ was carried out on a SMART CCD three-circle diffractometer (Mo-Kα radiation, graphite monochromator, ω scan mode). The structure was solved by the direct method and refined by the full-matrix least-squares method. The H atoms of the OH groups of solvate molecules were located from the difference Fourier synthesis, and the other H atoms were placed in calculated positions. Selected crystallographic data and refinement parameters are given in Table 1.

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