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Six Peroxide Groups in One Molecule – Synthesis of Nine-Membered Bicyclic Silyl Peroxides

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Compounds containing two nine-membered peroxide rings bridged by an ethane, ethene, or ethyne group were synthesized by the reactions of 1,2-bis[dichloro(alkyl)silyl]ethanes, (*E*)-1,2-bis[dichloro(methyl)silyl]ethene, or 1,2-bis[dichloro(methyl)silyl]ethyne with 1,1′-dihydroperoxydicycloalkyl peroxides. Each ring is formed by the replacement of two geminal chlorine atoms at the silicon atom with the hydroperoxide groups of one peroxide molecule. The cyclization is regioselective. The possible 12-membered bicyclic peroxide products are not produced. Unexpectedly, the peroxides are extremely explosive. The reactions of 1,2-bis[dichloro(alkyl)silyl]ethanes with 1,1-dihydroperoxy-*tert*-butylcyclohexane give only bicyclic peroxides containing two six-membered rings also through the replacement of two geminal chlorine atoms at the silicon atom by the hydroperoxide groups of one dihydroperoxide molecule.

Introduction

Organic peroxides have been widely used for more than half a century in industry and scientific research as oxidants, initiators for radical polymerization and crosslinking, and synthetic intermediates.[1] Peroxides containing the C–O–O group are traditionally considered as possible explosives. For instance, hexamethylene triperoxide diamine, acetone di- and triperoxides, and peroxides of other ketones are of particular interest.[2] In the past three decades, organic peroxides have attracted interest as pharmaceutically active compounds, for example, with antimalarial[3] or antihelminthic properties.[4] In the last decade, these compounds have been tested for antitumor activity.[5]

Most of the known organic peroxides contain COO– groups, whereas peroxides with SiOO– groups are much less common. The chemistry of organosilicon peroxides is less developed compared to that of organic peroxides mainly because of their lower hydrolytic stability and the limited number of methods for the selective synthesis of these compounds. There are several routes to peroxides with the Si–O–O group. These methods are based on the Mukaiyama–Isayama peroxidation of unsaturated compounds[6] and the reactions of chlorosilanes with hydroperoxides in the presence of bases,[7] compounds containing the Si–H bond with ozone,[8] singlet oxygen with silyl enolates,[9] and hydroperoxides with *N*,*O*-bis(trimethylsilyl)acetamide.[10]

Compounds containing the Si–O–O group have specific fields of application and properties. They also can be used in the same areas as organic peroxides and have some properties similar to the latter. The specificity of organosilicon peroxides is associated with the fact that the silicon atom is highly prone to oxidation, which is usually accompanied by Si–C bond cleavage. For example, silyl peroxides undergo thermal transformations,[11] which is not typical of alkyl peroxides. Organosilicon peroxides are used for peroxidation,[12] initiation of polymerization,[13] hydroxylation of arenes,[14] and for the synthesis of 1,2-dioxolanes,[15] 1,2-dioxanes,[15e,16] 1,2,4-trioxanes,[17] 1,2-dioxepanes,[15e] 1,2,4,5-tetraoxepanes,[18] and 1,2,4,5-tetraoxanes.[19] Organo silicon peroxides are produced as intermediates in the Fleming[20] and Tamao–Kumada[21] oxidations. The physicochemical properties of Si–O–O peroxides have received considerable attention from researchers.[22] The main data on

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organosilicon peroxides have been discussed in several reviews.[23]

The chemistry of cyclic silyl peroxides, in particular, the synthesis of these compounds, has received little study. The following cyclic structures containing the O–O–Si–O–O fragment have been reported: 3,3,6,6,9,9-hexamethyl-1,2,4,5,7,8-hexaoxa-3,6,9-trisilonane,[24] 1,2,4,5,7,8-hexaoxa-3-silonanes,[25] macrocycles containing the SiOOC moiety,[26] and some other related structures.[27,22f,22g] The small body of data on peroxide macrocycles is largely because these compounds are synthesized from two bifunctional reagents, which easily form linear products (oligomers and polymers) when they react with each other. Hence, it is not always possible to use this approach for the synthesis of cyclic peroxides with a specific composition.

In the present study, we successfully synthesized peroxides consisting of two Si-containing rings with three peroxide groups in each ring and, as a result, the products contain six peroxide groups per molecule.

Results and Discussion

Synthesis of Nine-Membered Bicyclic Silyl Peroxides

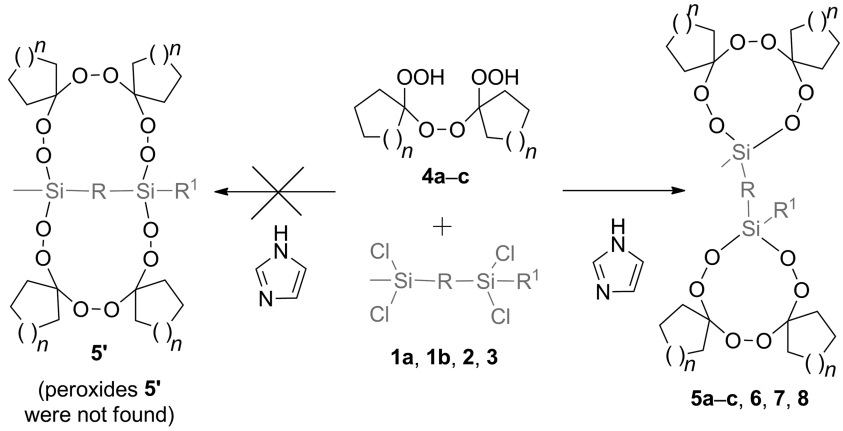
The reactions of the tetrachlorodisilanes **1a**, **1b**, **2**, and **3** with the dihydroperoxyperoxides **4a**–**4c** under mild conditions resulted in the formation of two nine-membered Si-containing peroxide rings bridged by an ethane, ethene, or ethyne group (Scheme 1).

The chlorine atoms in the disilanes **1a**, **1b**, **2**, and **3** are selectively replaced by peroxide groups to form the bicyclic silyl peroxides **5a**–**5c**, **6**, **7**, and **8** rather than the possible 12-membered ring products **5′**. The yields of the peroxides vary from 52 to 95% and increase with increasing size of the cycloalkyl moiety in the starting dihydroperoxyperoxide **4a**–**4c**. The reactions were performed in diethyl ether at 20–25 °C in the presence of imidazole as a base at a chlorodisilane/dihydroperoxide/imidazole molar ratio of 1:2.4:5. Under these conditions, the probable 12-membered rings are not formed (Table 1).

The synthesized peroxides were characterized by NMR spectroscopy and high-resolution mass spectrometry. The determination of their structures appeared to be a complex problem. The formation of either nine- or twelve-membered rings could be proposed on the basis of the NMR spectroscopic and mass spectrometric data. The selective formation of the nine-membered rings was ultimately confirmed by an X-ray diffraction study of **5a** (Figure 1).

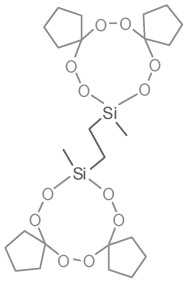
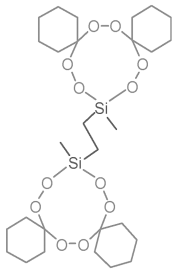
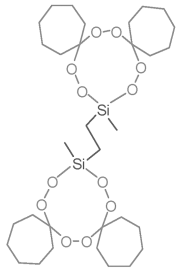
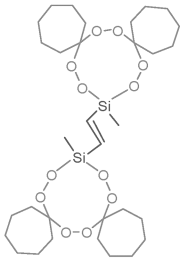
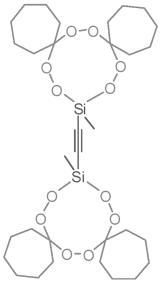
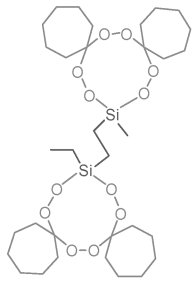
In the crystal structure of **5a**, the molecules are centrosymmetric with an inversion center located at the midpoint of the ethylene group that bridges the two Si atoms. All bond lengths and bond angles have standard values and are in good agreement with those observed in the crystal structures of two related 1,2,4,5,7,8-hexaoxa-3-silonanes.[25] Each Si atom has a distorted tetrahedral environment formed by two O atoms [Si–O 1.674(9) and 1.691(10) Å] and two C atoms [Si–C 1.875(14) and 1.897(15) Å]. Two independent cyclopentane rings adopt an envelope conformation with the C-5 and C-10 atoms as flaps. The crystal packing is typical of branched hydrocarbons with hydrophobic space-filling contacts.

A surprising fact was the instability of **5a**–**5c**, which are explosive compounds sensitive to friction, impact, and heat. We failed to determine their melting points; the heating of these compounds led to their extensive decomposition with destruction of equipment, which made it very difficult to perform elemental analysis. Nine-membered monocyclic peroxides containing three peroxide groups synthesized in our previous work[25] were stable compounds. The ratio of the C, H, O, and Si atoms in the stable monocyclic perox-



Scheme 1. Synthesis of nine-membered bicyclic silyl peroxides **5a**–**5c**, **6**, **7**, and **8**.

Table 1. Synthesis of nine-membered bicyclic silyl peroxides **5a**–**5c**, **6**, **7**, and **8** (structures; yields).

Structures of nine-membered dicyclic peroxides **5a–c**, **6**, **7**, **8**; % yield		
		
5a; 52[a]	**5b**; 73[a]	**5c**; 78[a], 95[b]
		
6; 90[b]	**7**; 78[b]	**8**; 76[a], 95[b]

[a] A solution of tetrachlorodisilane **1** (0.150 g, 0.586 mmol) in Et_2O (1.5 mL) was added to a mixture of 1,1′-dihydroperoxydicycloalkyl peroxide **4a**–**4c** (0.329–0.408 g, 1.406 mmol) and imidazole (0.199 g, 2.930 mmol) in Et_2O (9 mL) at 20–25 °C for 3–4 min, and then the reaction mixture was stirred for 3 h. Compounds **5a**–**5c** and **8** were isolated by crystallization. [b] A solution of tetrachlorodisilane **1** (0.150 g, 0.586 mmol), **2** (0.149 g, 0.586 mmol), or **3** (0.148 g, 0.586 mmol) in Et_2O (1.5 mL) was added to a mixture of 1,1′-dihydroperoxydicycloalkyl peroxide **4a**–**4c** (0.329–0.408 g, 1.406 mmol) and imidazole (0.199 g, 2.930 mmol) in Et_2O (9 mL) at 20–25 °C for 3–4 min, and then the reaction mixture was stirred for 3 h. Compounds **5c** and **6**–**8** were isolated by silica gel filtration.

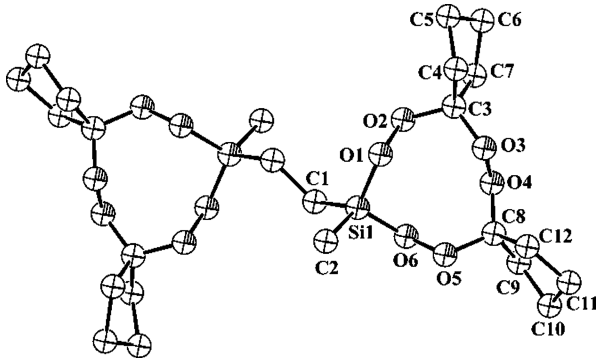
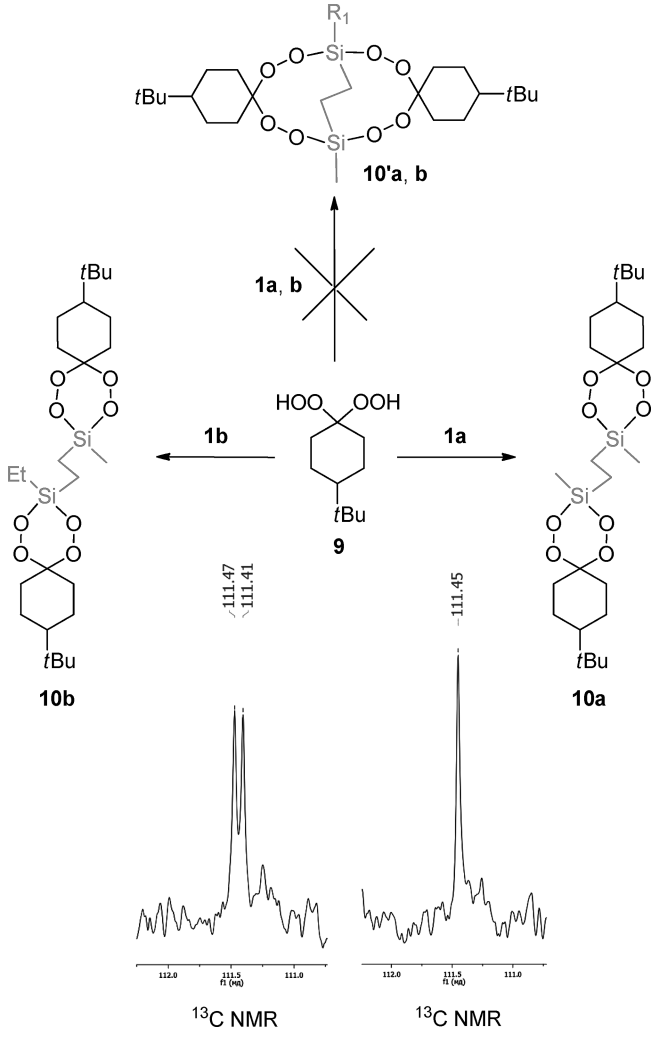


Figure 1. The molecular structure of **5a** showing the atomic numbering scheme and 50% probability displacement ellipsoids. The unlabeled atoms are related to the labeled atoms by the symmetry operation (1 – *x*, 1 – *y*, 1 – *z*). Hydrogen atoms are omitted for clarity.

ides[25] is the same as that in the bicyclic peroxides **5a**–**5c**. Therefore, the explosive properties of **5a**–**5c** are attributed to the presence of six peroxide groups in one molecule and the consequential higher probability of cleavage of one O–O bond to initiate the intramolecular decomposition of **5a**–**5c**.

Synthesis of Six-Membered Bicyclic Silyl Peroxides from 1,2-Bis[dichloro(alkyl)silyl]ethanes and 1,1-Bis(hydroperoxy)-4-*tert*-butylcyclohexane

In the next stage of our work, we investigated the reaction of the 1,2-bis(alkyldichlorosilyl)ethanes **1a** and **1b** with 1,1-bis(hydroperoxy)-4-*tert*-butylcyclohexane (**9**, Scheme 2).



Scheme 2. Synthesis of the six-membered bicyclic silyl peroxides **10a** and **10b** and parts of the ^{13}C NMR spectra with the resonances of the OOCOO fragments.

In this case, the formation of nine-membered cyclic silyl peroxides **10′a** and **10′b** as more-stable compounds would be expected by analogy with the data on the formation of cyclic structures according to Scheme 1.[25,26]

However, the reaction produced exclusively the six-membered cyclic silyl peroxides **10a** and **10b** rather than the

nine-membered peroxides, that is, two geminal chlorine atoms at the silicon atom are replaced by two geminal hydroperoxide groups of diperoxide **9** (Scheme 2).

Unlike the nine-membered peroxides **5a**–**5c**, the six-membered bicyclic peroxides **10a** and **10b** are unstable. They were not isolated in the individual state, because these compounds did not withstand purification on alumina and different types of silica gel. The NMR monitoring of the reaction presented in Scheme 2 showed that the reaction afforded six-membered bicyclic peroxides as the only products, but these compounds completely decomposed in the reaction mixtures within 24 h.

The formation of the six-membered bicyclic peroxides **10a** and **10b** rather than the possible nine-membered bicyclic peroxides **10′a** and **10′b** was confirmed by the NMR spectroscopic monitoring of the reactions of **9** with 1,2-bis(dimethylchlorosilyl)ethane (**1a**) and 2-dichloroethylsilyl-1-methyldichlorosilylethane (**1b**) in the presence of pyridine in $CDCl_3$ (Scheme 2). For the reaction of the diperoxide **9** with tetrachlorodisilane **1a**, the ^{13}C NMR spectrum (Scheme 2) shows one signal of the OOCOO group at δ = 111.45 ppm, which is indicative of the formation of two identical rings, that is, of the equally probable peroxides **10a** or **10′a**. The key data in support of the formation of the six-membered bicyclic peroxides **10a** and**10b** were obtained by NMR spectroscopic monitoring of the reaction of **9** with **1b**. Thus, the ^{13}C NMR spectrum shows two signals at δ = 111.41 and 111.47 ppm (Scheme 2), which belong to two nonequivalent OOCOO groups of six-membered rings **10b** and by no means correspond to two identical OOCOO groups of bicyclic peroxide **10′b**.

The structure of the six-membered bicyclic peroxide **10b** was ultimately established by ^1H, ^{13}C, and ^{29}Si NMR spectroscopy through the 2D correlation techniques COSY, heteronuclear single quantum coherence (HSQC), and HMBC and by diffusion-ordered spectroscopy (DOSY). The DOSY NMR spectroscopic data provided an estimate of the molecular weight of this compound and, consequently, the number of residual dihydroperoxide and disilane moieties in the molecule.

Conclusions

Compounds containing two nine-membered silyl peroxide rings bridged by an ethane, ethene, or ethyne group were synthesized by the reaction of 1,2-bis[dichloro(alkyl)silyl]-ethanes, (*E*)-1,2-bis[dichloro(methyl)silyl]ethene, or 1,2-bis-[dichloro(methyl)silyl]ethyne with 1,1′-dihydroperoxyperoxides and were then isolated in the individual state. Only bicyclic peroxides containing two six-membered rings were detected in the reactions of 1,2-bis[dichloro(alkyl)silyl]ethanes with 1,1-bis(hydroperoxy)-*tert*-butylcyclohexane. In both cases, the ring is formed by the replacement of the geminal chlorine atoms at the silicon atom by two hydroperoxide groups of one peroxide molecule. The nine-membered bicyclic peroxides containing six peroxide groups in one molecule are extremely explosive. However, they can be isolated, used in reactions, and stored at 0–10 °C without clear signs of decomposition over several months. After further investigations in the field of explosives, the synthesized Si–O–O peroxides could find use as such compounds along with nitro compounds, alkyl peroxides, and azides, for example.

Experimental Section

Caution: *When working with peroxides, precautions, such as the use of shields, fume hoods, and the avoidance of transition metal salts, heating, and shaking, should be taken whenever possible.*

The ^1H and ^{13}C NMR spectra were recorded with 400 (400.1 and 100.6 MHz, respectively) and 300 MHz (300.1 and 75.5 MHz, respectively) spectrometers with samples in $CDCl_3$. The assignments of ^1H and ^{13}C NMR signals were made with the aid of 2D COSY, HSQC, and HMBC spectra when necessary. The ^{29}Si NMR spectra were recorded with a 300 MHz spectrometer (59.6 MHz; 300.1 MHz for ^1H) with samples in $CDCl_3$ by using the insensitive nuclei enhanced by polarization transfer (INEPT) sequence and Me_4Si as the standard. The assignments of ^{29}Si NMR signals were made with the aid of 2D HMBC spectra when necessary. As some chemical shifts in the ^{29}Si NMR spectra could not be directly detected, they were taken from the corresponding two-dimensional spectra. The reactions in $CDCl_3$ solutions were monitored in NMR tubes. The diffusion coefficients were measured by 2D ^1H DOSY NMR spectroscopy with samples in $CDCl_3$ with a 300 MHz spectrometer (300.1 MHz for ^1H). The BPP-LED pulse sequence was used with Δ = 100 ms and T_e = 5 ms. The DOSY spectra were processed by monoexponential fitting and the SCORE algorithm by using the Bruker TopSpin and DOSY Toolbox software. Dibenzo-18-crown-6 was used as an internal and external calibrant for the molecular weights in a molar ratio of ca. 1:1 to the compounds under study in the DOSY spectra.

MeCN (HPLC grade) for ESI-HRMS experiments was purchased from Merck and used as supplied. All samples for ESI-HRMS experiments were prepared in 1.5 mL Eppendorf tubes. All plastic disposables (Eppendorf tubes and tips) used in sample preparation were washed with MeCN before use. High-resolution mass spectra were recorded with a Bruker maXis instrument equipped with an electrospray ionization (ESI) ion source.[28] All measurements were performed in positive (+MS) ion mode (interface capillary voltage: 4500 V) with a scan range m/z = 50–3000. External calibration of the mass spectrometer was performed with Electrospray Calibrant Solution (Fluka). A direct syringe injection was used for all of the analyzed solutions in MeCN (flow rate: 3 μL/min). Nitrogen was used as the nebulizer gas (0.4 bar) and dry gas (4.0 L/min); the interface temperature was set at 180 °C. All of the spectra were processed by using the Bruker DataAnalysis 4.0 software package.

The TLC analysis was performed with standard silica gel chromatography plates. The melting points were determined with a Kofler hot-stage apparatus. Chromatography was performed with silica gel (63–200 mesh). Dichloromethane, Et_2O, petroleum ether (PE; 40/70), MeCN, tetrahydrofuran (THF), ethyl acetate, and imidazole were purchased from Acros. Silica fume (particle size 0.007 mm, S5130) was purchased from Sigma. 1,1′-Dihydroperoxyperoxides **4a**–**4c** and 1,1-bis(hydroperoxy)-4-*tert*-butylcyclohexane (**9**) were prepared as described previously.[29]

CCDC-1012863 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The

Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

General Procedure for the Synthesis of 5a–c and 8: A solution of silane **1a** (0.150 g, 0.586 mmol) in Et_2O (1.5 mL) was added with vigorous stirring to a mixture of 1,1′-dihydroperoxydicycloalkyl peroxide **4a**–**4c** (0.329–0.408 g, 1.406 mmol) and imidazole (0.199 g, 2.930 mmol) in Et_2O (9 mL) at 20–25 °C over 3–4 min. The reaction mixture was stirred for 3 h. Then, petroleum ether (40 mL) was added. The mixture was successively washed with a 5% NaOH solution (2× 10 mL) and water (2× 10 mL). The organic phase was separated and dried with $MgSO_4$. The solvent was evaporated by using a water-jet vacuum pump. The products **5a**–**5c** and **8** were isolated by crystallization from MeOH (Table 1, note [a]).

Synthesis of 3,3′-Ethane-(or ethene or ethyne)-1,2-diylbis-1,2,4,5,7,8,3-hexaoxasilonanones 5c and 6–8: A solution of silane **1** (0.150 g, 0.586 mmol), **2** (0.149 g, 0.586 mmol), or **3** (0.148 g, 0.586 mmol) in Et_2O (1.5 mL) was added with vigorous stirring to a mixture of **4c** (0.408 g, 1.406 mmol) and imidazole (0.199 g, 2.930 mmol) in Et_2O (9 mL) at 20–25 °C over 3–4 min. The reaction mixture was stirred for 3 h, petroleum ether (40 mL) was added, and the reaction mixture was filtered through a short layer (2 cm) of silica gel (5–40 μm). The solvent was evaporated by using a water-jet vacuum pump (Table 1, note [b]).

15,15′-Ethane-1,2-diylbis(15-methyl-6,7,13,14,16,17-hexaoxa-15-siladispiro[4.2.4.5]heptadecane) (5a): White crystals; explosive on heating above 152 °C. R_f = 0.57 (TLC, hexane/ethyl acetate, 10:1). ^{1}H NMR (400.1 MHz, $CDCl_3$): δ = 0.29 (s, 6 H), 0.75–2.35 (m, 36 H) ppm. ^{13}C NMR (100.6 MHz, $CDCl_3$): δ = –7.30, 2.69, 2.79, 24.57, 24.64, 24.92, 33.20, 33.29, 33.41, 33.57, 120.23 ppm. ^{29}Si NMR (59.6 MHz, $CDCl_3$): δ = 7.52 ppm. HRMS (ESI): calcd. for $C_{24}H_{42}NaO_{12}Si_2$ [M + Na]$^+$ 601.2107; found 601.2107.

17,17′-Ethane-1,2-diylbis(17-methyl-7,8,15,16,18,19-hexaoxa-17-siladispiro[5.2.5.5]nonadecane) (5b): White crystals, m.p. 132–135 °C with explosive decomposition. R_f = 0.58 (TLC, hexane/ethyl acetate, 10:1). ^{1}H NMR (400.1 MHz, $CDCl_3$): δ = 0.29 (s, 6 H), 0.75–2.35 (m, 44 H) ppm. ^{13}C NMR (100.6 MHz, $CDCl_3$): δ = –7.22, 2.81, 22.56, 22.80, 25.64, 29.92, 30.26, 30.76, 108.74 ppm. ^{29}Si NMR (59.6 MHz, $CDCl_3$): δ = 7.05 ppm. HRMS (ESI): calcd. for $C_{28}H_{50}NaO_{12}Si_2$ [M + Na]$^+$ 657.2733; found 657.2739.

19,19′-Ethane-1,2-diylbis(19-methyl-8,9,17,18,20,21-hexaoxa-19-siladispiro[6.2.6.5]heneicosane) (5c): White crystals; M.p. 134–137 °C; explosive on heating above 163 °C. R_f = 0.6 (TLC, hexane/ethyl acetate, 10:1). ^{1}H NMR (400.1 MHz, $CDCl_3$): δ = 0.27 (s, 6 H), 0.75–2.21 (m, 52 H) ppm. ^{13}C NMR (100.6 MHz, $CDCl_3$): δ = –7.21, 2.83, 23.01, 30.00, 30.07, 32.62, 32.86, 33.00, 113.73 ppm. ^{29}Si NMR (59.6 MHz, $CDCl_3$): δ = 6.58 ppm. HRMS (ESI): calcd. for $C_{32}H_{58}NaO_{12}Si_2$ [M + Na]$^+$ 713.3359; found 713.3354.

19,19′-Ethene-1,2-diylbis(19-methyl-8,9,17,18,20,21-hexaoxa-19-siladispiro[6.2.6.5]heneicosane) (6): White crystals, explosive on heating above 110–120 °C. R_f = 0.6 (TLC, hexane/ethyl acetate, 10:1). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.37 (s, 6 H), 0.75–2.3 (m, 48 H), 6.85 (s, 2 H) ppm. ^{13}C NMR (75.5 MHz, $CDCl_3$): δ = –7.34, 22.90, 29.88, 29.97, 32.49, 32.64, 32.89, 32.99, 113.93, 146.44 ppm. ^{29}Si NMR (59.6 MHz, $CDCl_3$): δ = –10.12 ppm. HRMS (ESI): calcd. for $C_{32}H_{56}NaO_{12}Si_2$ [M + Na]$^+$ 711.3203; found 711.3198.

19,19′-Ethyne-1,2-diylbis(19-methyl-8,9,17,18,20,21-hexaoxa-19-siladispiro[6.2.6.5]heneicosane) (7): White crystals, explosive on heating above 120 °C. R_f = 0.6 (TLC, hexane/ethyl acetate, 10:1). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.38 (s, 6 H), 0.75–2.25 (m, 48 H) ppm. ^{13}C NMR (75.5 MHz, $CDCl_3$): δ = –4.88, 22.79, 22.88, 29.07, 32.64, 32.89, 104.98, 114.27 ppm. ^{29}Si NMR (59.6 MHz, $CDCl_3$): δ = –28.31 ppm. HRMS (ESI): calcd. for $C_{32}H_{54}NaO_{12}Si_2$ [M + Na]$^+$ 709.3046; found 709.3068.

19-Ethyl-19-[2-(19-methyl-8,9,17,18,20,21-hexaoxa-19-siladispiro-[6.2.6.5]heneicos-19-yl)ethyl]-8,9,17,18,20,21-hexaoxa-19-siladispiro[6.2.6.5]heneicosane (8): White crystals, explosive on heating above 135 °C. R_f = 0.6 (TLC, hexane/ethyl acetate, 10:1). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.26 (s, 3 H), 0.70–2.23 (m, 57 H) ppm. ^{13}C NMR (75.5 MHz, $CDCl_3$): δ = –7.34, 1.34, 2.25, 2.66, 6.49, 22.76, 22.91, 29.79, 29.93, 32.99 113.60 ppm. ^{29}Si NMR (59.6 MHz, $CDCl_3$): δ = 4.91, 6.57 ppm. HRMS (ESI): calcd. for $C_{33}H_{60}NaO_{12}Si_2$ [M + Na]$^+$ 727.3516; found 727.3521.

Compound 10a: 1,1-Bis(hydroperoxy)-4-*tert*-butylcyclohexane (**9**; 0.040 g, 0.1961 mmol) and pyridine (0.035 g, 0.446 mmol) in a molar ratio of 2.2:5 were dissolved in $CDCl_3$ (0.6 mL) in an NMR tube filled with argon. Then, bis(dimethylchlorosilyl)ethane (**1a**; 0.023 g, 0.0891 mmol) was added. After 1 h, ^{1}H, ^{13}C, and ^{29}Si NMR spectra were recorded by using the 2D correlation techniques COSY, HSQC, and HMBC.

Compound 10b: 1,1-Bis(hydroperoxy)-4-*tert*-butylcyclohexane (**9**; 0.040 g, 0.1961 mmol) and pyridine (0.035 g, 0.446 mmol) in a molar ratio of 2.2:5 were dissolved in $CDCl_3$ (0.6 mL) in an NMR tube filled with argon. Then, 2-dichloroethylsilyl-1-methyldichlorosilylethane (**1b**; 0.024 g, 0.0891 mmol) was added. After 1 h, ^{1}H, ^{13}C, and ^{29}Si NMR spectra were recorded by using the 2D correlation techniques COSY, HSQC, and HMBC.

2D ^{1}H DOSY of Peroxide 10a: 1,1-Bis(hydroperoxy)-4-*tert*-butylcyclohexane (**9**; 0.040 g, 0.1961 mmol) and pyridine (0.035 g, 0.446 mmol) in a molar ratio of 2.2:5 were dissolved in $CDCl_3$ (0.6 mL) in an NMR tube filled with argon, and then bis(dimethylchlorosilyl)ethane (**1a**; 0.023 g, 0.0891 mmol) was added. After 1 h, dibenzo-18-crown-6 (0.0250 g, 0.0694 mmol) was added as the standard. Silica fume (1 wt.-%) was added to the NMR tube, the tube was shaken, and the 2D ^{1}H DOSY NMR spectrum was recorded.

2D ^{1}H DOSY of Peroxide 10b: 1,1-Bis(hydroperoxy)-4-*tert*-butylcyclohexane (**9**; 0.040 g, 0.1961 mmol) and pyridine (0.035 g, 0.446 mmol) in a molar ratio of 2.2:5 were dissolved in $CDCl_3$ (0.6 mL) in an NMR tube filled with argon, and then 2-dichloroethylsilyl-1-methyldichlorosilylethane (**1b**; 0.024 g, 0.0891 mmol) was added. After 1 h, dibenzo-18-crown-6 (0.0250 g, 0.0694 mmol) was added as the standard. Silica fume (1 wt.-%) was added to the NMR tube, the tube was shaken, and the 2D ^{1}H DOSY NMR spectrum was recorded.

Supporting Information (see footnote on the first page of this article): ^{1}H and ^{13}C NMR spectra, 2D and 3D NMR spectra, mass spectra, and details of X-ray crystallography data.

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