group through interaction with the ammonium group. On the other hand Goebel et al.[12] showed that RNA cleavage can also be accomplished without the aid of an internal base. Since the cyclen moiety exists as a bis(ammonium) salt at neutral pH it can be envisioned that the remarkable hydrolysis is the result of a mechanism that involves the formation of a bis-cation on the cyclen moiety. Investigations addressing the role of the cyclen subunit and the mode of action for this cleavage are presently being performed in our laboratories.

The fact that the nonamer–cyclen conjugate cleaves selectively and very efficiently at neutral pH and room temperature and that these cleavage reactions are more efficient in the absence than in the presence of Eu^{III}, makes them a promising tool for a selective interaction with the life cycle of HIV-1.

Experimental Section

All experiments were performed in autoclaved Eppendorf reaction vessels. Extreme precaution has been taken to avoid RNase contamination. Water (Millipore quality) and all equipment had been treated with diethyl pyrocarbonate (DEPC) and then autoclaved prior to used. The RNA cleavage reaction was carried out in pH 7.4 Tris-HCl (20 mM) containing RNA (75.4 nM), peptides (167.2 μM), and NaCl (20 mM). The reaction mixture (5 μL) was incubated for 1 h at room temperature unless otherwise stated. After reaction, fish sperm DNA (1 μg) and formamide gel loading buffer (6 μL) was added to the reaction mixture. The mixture was then heated to 85 °C for 5 min and then cooled in ice. 7 μL of each sample was loaded onto a 20% denaturing polyacrylamide gel. After electrophoresis the RNA was transferred onto a positively charged nylon membrane by electro-blotting. After immobilization at 80 °C for 30 min followed by the wash protocol from Ambion, the RNA was visualized with streptavidin/alkaline phosphatase and CDP-star on Kodak film.

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[6] Spectrometer: Kompakt Maldi 3 from Kratos. The experiments were performed in the positive-linear-high mode with α-cyanocinnamic acid as the matrix. The matrix was saturated with a mixture of MeCN/0.1% TFA (2/1). The calculated mass for compound **6** is 1552, the observed mass was 1552 and corresponds to $[M^+]$ of **6**.

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The First Synthesis of Organic Diselenolates: Application to the Synthesis of Diorganyl Diselenides**

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Dedicated to Professor Léon Ghosez on the occasion of his 65th birthday

There has been a growth in interest[1] of organoselenium chemistry over the last two decades and although several new reactions have been described, a relatively small number of

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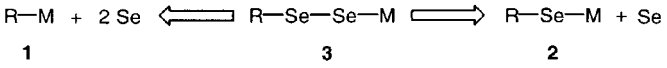
novel functional groups have been discovered in selenium chemistry. Diselenides, the selenium counterpart of organic peroxides, play a key role in organoselenium chemistry since they are stable, easily handled, and reactive enough to produce electrophilic, nucleophilic, and radicophilic species.[1] Related derivatives in which one selenium atom has been replaced by oxygen or sulfur are also known[2] and the latter even play a crucial role in biology for the degradation of hydroperoxides.[2a]

Organic diselenols (RSeSeH **4**) or their salts (RSeSeM **3**), the selenium analogues of hydroperoxides (or their salts (ROOM)),[3] are at present unknown. Except in rare cases, the structurally related selenenic acids (RSeOH) are unstable and exhibit an extremely high tendency to disproportionate to diselenides and seleninic acids.[4] It was therefore of interest to know if these species could exist and what kind of reactivity they possess. This aspect is described herein.

The synthetic approaches planned are straightforward and imply the selective insertion of 1) two selenium atoms into the carbon–metal bonds of organometallic compounds **1** or of 2) one selenium atom into selenolates **2** (Scheme 1).[5]



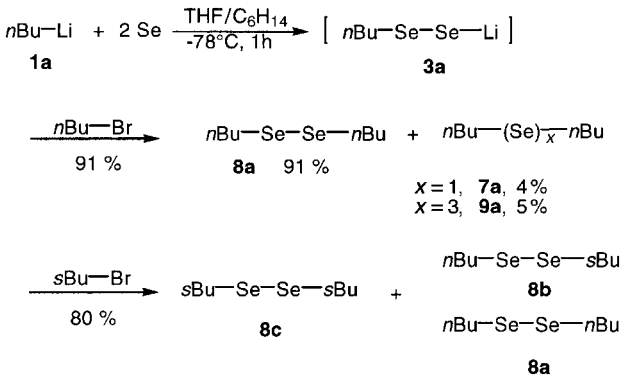
Scheme 1. Retrosynthetic routes to organic diselenolates.

Although the reaction of elemental selenium with organic selenolates has to our knowledge never been reported, the reaction of elemental selenium with various organometallic compounds is one of the most valuable methods to synthesize organic selenolates **2**.[6] In this respect the state of the art is to avoid the production of polyselenolates that result from the introduction of more than one selenium atom into the organometallic compound.

In fact the selenium insertion reaction is poorly described and, up to now, no effort has been made to elucidate it.[7] We decided to investigate it systematically through the use of *n*-butyllithium **1a** and treat it stepwise with one to ten equivalents of elemental selenium. For that purpose we added *n*-butyllithium (1.6 N in hexane) to a suspension of gray metallic elemental selenium (2 equiv) in THF maintained at $-78\,^{\circ}C$ under argon. We were delighted to find that all the selenium disappeared quite rapidly producing a dark-red clear solution (stage A).[8]

The addition of a solution of *n*-butyl bromide in THF to the reaction medium led to the formation in almost quantitative yield of di-*n*-butyl diselenide **8a** contaminated with small amounts (< 5 % each) of di-*n*-butyl selenide **7a** and di-*n*-butyl triselenide **9a** (Scheme 2). We monitored the reaction by GC using an internal standard to confirm that the di-*n*-butyl diselenide is produced by alkylation of *n*-butyl diselenolate, and have observed the disappearance of *n*-butyl bromide after one hour at $-78\,^{\circ}C$.

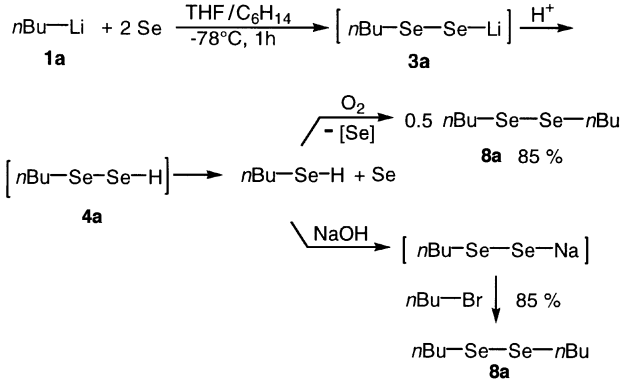
Successful alkylation of *n*-butyl diselenolate **3a** has also been achieved with *sec*-butyl bromide (80 % yield) but this reaction affords a statistical mixture of diselenides: the expected *n*-butyl-*sec*-butyl diselenide **8b** as well as di-*n*-butyl diselenide **8a** and di-*sec*-butyl diselenide **8c**, which results



Scheme 2. Synthesis of di-*n*-butyl diselenides from lithium *n*-butyl diselenolate.

from the diselenide **8b** probably under the influence of the basic medium (**8b**/**8a**/**8c** = 50/25/25).[9]

The synthesis of the as yet unknown *n*-butyldiselenol (*n*BuSeSeH) **4a** was our next objective. We planned to produce it by acidification of the medium (stage A) but attempts to trap this species have been unsuccessful. In fact addition of hydrochloric acid (1 N aq solution) results in the release of almost one equivalent of selenium as a red amorphous species, which rapidly turns gray and shiny (0.5 h, 95 % recovery after centrifugation). ^{77}Se NMR analysis of the remaining solution clearly shows the presence of *n*-butylselenol, which is quantitatively oxidized to di-*n*-butyl diselenide **8a** on further reaction with oxygen (Scheme 3).



Scheme 3. On the way to *n*-butyldiselenol.

We have subsequently found that sequential addition of hydrochloric acid, sodium hydroxide, and *n*-butyl bromide to the solution of lithium *n*-butyl diselenolate in THF (stage A) produces di-*n*-butyl diselenide (85 % yield, complete consumption of *n*-butyl bromide by GC).

This led us to suspect that *n*-butyl selenolates **2** are able to react with elemental selenium to produce the *n*-butyl diselenolates **3**. We indeed found in separate experiments that lithium *n*-butyl selenolate **2a** and its magnesium, sodium, and potassium analogues react at room temperature with gray metallic elemental selenium in THF or DMF to provide the corresponding *n*-butyl diselenolates **3**, which on further reaction with *n*-butyl iodide provides di-*n*-butyl diselenide **8a** (Table 1).

We have shown in a separate experiment that lithium *n*-butyl diselenolate **3a**, prepared from butyllithium and sele-

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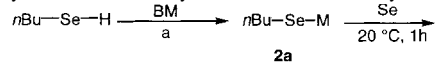
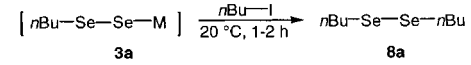
Table 1. Synthesis of di-*n*-butyl diselenide from *n*-butylselenol.



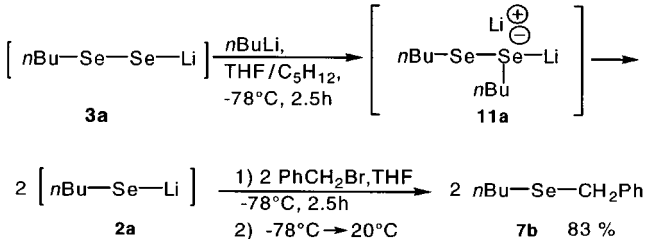


Entry	Base BM	Conditions a	Yield [%]
a	*n*BuLi	DMF, 0 °C, 0.5 h	93[a]
b	*n*BuMgCl	THF, 0 °C, 0.5 h	80[b]
c	LiH	DMF, 20 °C, 1 h	85[a]
d	NaH	THF, 20 °C, 1 h	95[a]
e	KH	DMF, 20 °C, 1 h	85[a]

[a] Trace amounts of di-*n*-butyl selenide and di-*n*-butyl triselenide are also observed. [b] 18% of di-*n*-butyl selenide is formed beside the diselenide.

nium in THF, is quite stable under argon; the di-*n*-butyl diselenide is formed in almost quantitative yield even if *n*-butyl diselenolate is heated at reflux for one hour prior to the addition of *n*-butyl bromide (1 equiv, reflux, 2 h, **7a**/**8a**/**9a** = 6/92/2). These results suggest that either lithium *n*-butyl diselenolate **3a** is not in equilibrium with lithium *n*-butyl selenolate **2a** and elemental selenium or it is so nucleophilic that the equilibrium is shifted towards the formation of di-*n*-butyl diselenide **8a**.

Finally we have found that *n*-butyllithium reacts with *n*-butyl diselenolate **3a** to produce **11a**, which releases one of its selenium atom very smoothly to produce two equivalents of lithium *n*-butyl selenolate **2a**. This compound has been quenched with benzyl bromide to deliver benzyl *n*-butyl selenide **7b** in very high yield (Scheme 4). This may be



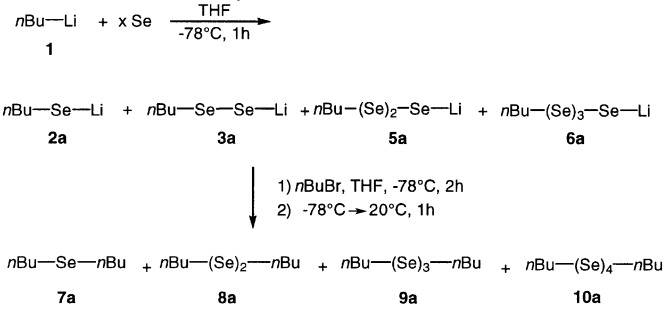
Scheme 4. Reaction of lithium *n*-butyl diselenolate with *n*-butyllithium.

explained by assuming that *n*-butyllithium selectively reacts with the selenium atom bearing the lithium counter ion (Scheme 4).

In conclusion we have presented some evidence that *n*-butyl diselenolates **3** exist and that the lithium derivative can be prepared quite easily and in almost pure form from *n*-butyllithium and two equivalents of elemental selenium. It was therefore tempting to try to get the higher homologues by simply adding increasing amounts of selenium to the medium. This procedure however leads to the formation of an intractable mixture of di-*n*-butyl selenide **7a**, di-*n*-butyl diselenide **8a** (major product), and higher homologues **9a** and **10a** (Table 2).

The reactions disclosed here are not limited to *n*-butyllithium. Other organolithium compounds such as *sec*-butyllithium or *tert*-butyllithium also produce the corresponding diselenolates **3**. The scope and limitation of this reaction will be

Table 2. Reaction of *n*-butyllithium and various amount of selenium.



Entry	*x* equiv Se^0	**7a**	**8a**	**9a**	**10a**
a	1	94	6	–	–
b	2	5	92	6	2
c	3	9	68	19	4
d	5	21	62	14	3
e	10	20	64	13	3

reported in due course. We are presently attempting to learn more about the reactivity of organic diselenolates.

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Keywords: diselenolate · Grignard reactions · organometallic compounds · selenium · selenolates

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Surface-Confined Metallodendrimers: Isolated Nanosize Molecules**

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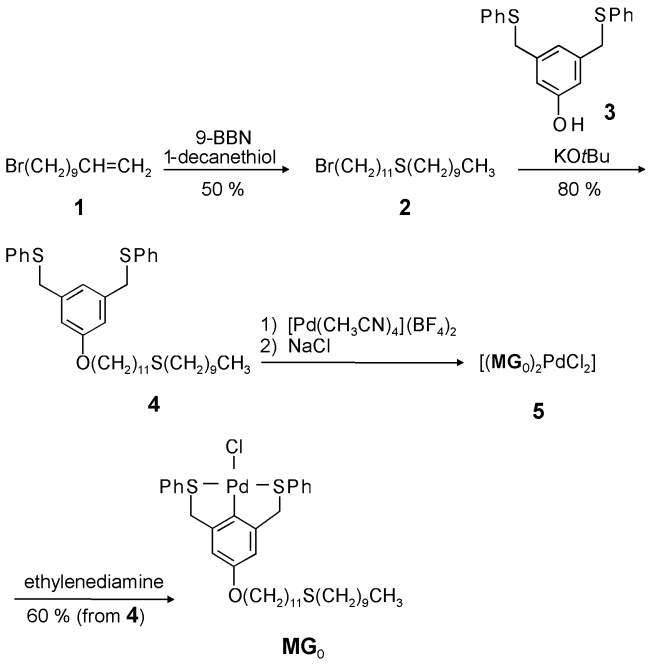
Current technology allows the patterning of surfaces at the submicrometer level but there is a considerable tendency to decrease this to the nanometer level.[1] Nanotechnology aims at the design and manufacture of nanosize devices,[2] and ultimately the components may consist of single molecules. However, there are two minimal requirements that need to be fulfilled: the simple synthesis of functional nanosize structures and the controlled positioning of these molecules. Dendrimers are a class of molecules of nanometer-size dimensions and they can be synthesized in a limited number of steps. They have a well-defined treelike architecture, and a wide variety of dendrimers with various cores, monomeric units, and functional groups have been reported.[3, 4] Self-assembly, in particular self-assembly of sulfur-containing compounds on gold surfaces, is a versatile method to order and orient molecules at an interface.[5, 6] Scanning tunneling microscopy (STM) showed that individual thiols can be isolated by insertion into a dodecanethiol monolayer on a gold surface.[7] Therefore, modification of a gold surface by the self-assembly of dendrimers seems to be a promising strategy to achieve surface-confined nanosize structures. Only a few studies have addressed dendrimers in spin-cast films.[8] Spontaneous surface confinement has been achieved by Crooks et al., who attached dendrimers to gold surfaces through the dendrimer end groups.[9, 10]

Our strategy to construct structures with nanometer dimensions relies on the self-assembly of specifically designed dendritic wedges that are easy to synthesize and to functionalize. Potentially, they can be chemically modified after surface confinement. The dendritic wedges are prepared by noncovalent synthesis, analogous to the controlled assembly of metallodendrimers previously developed in our group.[11]

A dendritic wedge was grown from a sulfide-derived core (→**MG$_0$**) for the attachment of metallodendrimers to the gold surface. Sulfide **2** was prepared by reaction of 11-bromo-1-undecene (**1**)[12] with 9-borabicyclo[3.3.1]nonane (9-BBN) and 1-decanethiol (Scheme 1). The ^{1}H NMR spectrum of **2** showed the complete conversion of the double bond. The pincer ligand was introduced by reaction of **2** with **3**[13] in the presence of a base. Ligand **4** was subsequently treated with two equivalents of $[Pd(CH_3CN)_4](BF_4)_2$ and converted into the overall neutral Pd-Cl complex by stirring with aqueous NaCl. More than one equivalent of $[Pd(CH_3CN)_4](BF_4)_2$ is needed for complete cyclopalladation, because the sulfide in the long alkyl chain also forms a complex with the Pd^{II} reagent. Initially, the trinuclear complex **5** was formed, which was isolated and subsequently converted into **MG$_0$** by addition of the chelating ethylenediamine. Formation of **MG$_0$** was confirmed by an intense signal at m/z=769.2 ($[M^+ - Cl]$, calcd: 769.3) in the FAB-MS spectrum.



Scheme 1. Synthesis of the dendrimer core.

Higher generation metallodendritic wedges with a sulfide chain were synthesized by controlled assembly. **MG$_0$** was activated by reaction with one equivalent of $AgBF_4$ and subsequently one equivalent of **BB$_{py}$-Cl**[14] was added (Scheme 2). This complex coordinates to the palladium center through the pyridine moiety. The first generation dendrimer (**MG$_1$**) was formed in essentially quantitative yield. This procedure was repeated with **MG$_1$** and two equivalents each of $AgBF_4$ and **BB$_{py}$-Cl** to afford the second-generation dendrimer **MG$_2$**. The ^{1}H NMR spectra of both **MG$_1$** and **MG$_2$** clearly reflected the coordination of the pyridine ligands with a shift of the α-pyridine protons from $\delta = 8.54$ to $\delta = 8.30$, similar to the previously reported metallodendrimer synthesis.[14] The successful formation of the dendritic assemblies was further confirmed by the presence of the molecular peaks minus a BF_4^- counter ion at $m/z = 1962.0$ (calcd: 1961.3) and

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