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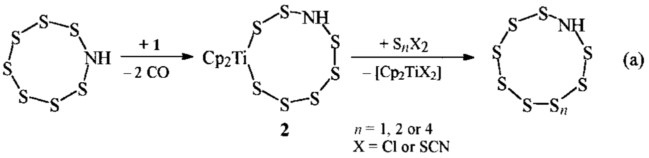
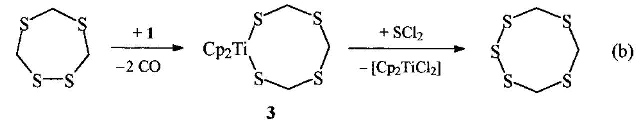
S_4NR (R = Methyl, *n*-Octyl) as Novel Chelating Ligands in Titanocene Complexes and First Synthesis of Small Sulfurimide Heterocycles S_nNR ($n = 5, 6$)[**]

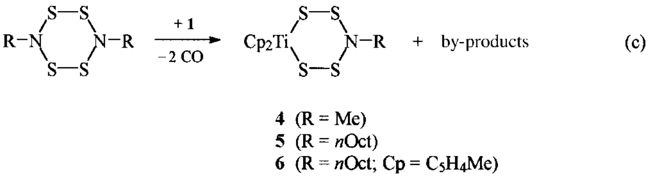
Ralf Steudel,[*] Oliver Schumann, Jürgen Buschmann, and Peter Luger

Recently it has been shown that titanocene dicarbonyl $[Cp_2Ti(CO)_2]$ **1** reacts with the S–S bonds of certain homo- and heterocycles with insertion of the titanocene unit and liberation of the two CO ligands.[1] In this way a number of novel chelate complexes has been prepared that are useful reagents for the synthesis of new chain- and ringlike derivatives by ligand transfer reactions. Examples are the complexes **2** and **3**, which allowed the preparation of the cyclic sulfurimides S_8NH, S_9NH, $S_{11}NH$,[2] and of the natural product pentathiocane 1,3,5-$(CH_2)_3S_5$,[1b] respectively [Eq. (a), (b), $Cp = \eta^5\text{-}C_5H_5$].





While cycloheptasulfurimide S_7NH and its organic derivatives S_7NR have been known for a long time,[3] the six- and seven-membered rings S_5NR and S_6NR have never been observed although the corresponding homocycles S_6 and S_7[4] as well as their carbon-substituted analogues CH_2S_5 and CH_2S_6[5] are well known as pure materials. Herein we show how cyclic penta- and hexasulfurimides may be prepared in very good yield by ligand transfer from the novel chelate complexes **4** and **5**, respectively. Complex **1** reacts in *n*-hexane at 20 °C with *N*-alkyl-substituted derivatives of the cyclic diimide $HN(\mu\text{-}S_2)_2NH$[6] to give titanocene derivatives which, however, do not contain the expected seven-membered but a six-membered metallacycle [Eq. (c), *n*Oct = *n*-octyl].



Contrary to the behavior of S_7NH and S_7NMe[2] there is no insertion into the S–S bond of the diimide, but one NR group is extruded (the fate of this group has not been investigated). Working with two different groups R (Me, *n*Oct) and two different cyclopentadienyl ligands (C_5H_5, C_5H_4Me) only *one* complex was obtained in each case (yields 25–34 %). This contained the ligand SSN(R)SS, which has not been observed before.[7] Compound **4** forms black orthorhombic crystals with a melting point of 134 °C, while **5** and **6** are dark brown oils; all three compounds are stable in air for several days and soluble in *n*-hexane, dichloromethane, and carbondisulfide. Decomposition of the brown solutions occurs after several days at 20 °C.

Complex **4** was characterized by single-crystal X-ray structural analysis,[8] which revealed a metallacycle with a chair conformation (Figure 1) similar to the one in $[Cp_2TiS_5]$[9] and in the analogous complex $[Cp_2Ti(\mu\text{-}S_2)_2AsMe]$.[10] The molecules of **4** occupy general positions but approximately exhibit C_s symmetry. The geometrical parameters show the expected values, for example, $d(S-S) = 206.0$ pm, $d(S-N) =$

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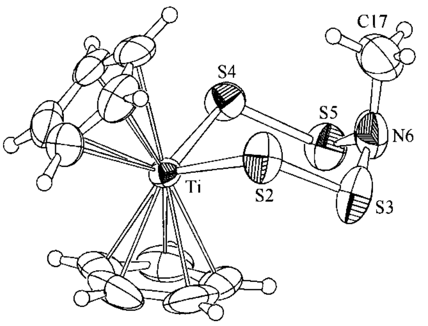

Figure 1. Structure of **4** in the crystal. Selected distances [pm], angles [°], and torsion angles [°]: Ti–S2 243.6(3), S2–S3 206.0(4), S3–N6 169.7(14), N6–S5 169.4(13), S5–S4 206.0(5), S4–Ti 244.4(4), N6–C17 146.0(2); S4-Ti-S2 93.99(13), S3-N6-S5 116.9(7), S3-N6-C17 116.0(11), S5-N6-C17 114.4(10); S4-Ti-S2-S3 53.8(2), Ti-S2-S3-N6 −64.4(5), S2-S3-N6-S5 80.5(7), S3-N6-S5-S4 −82.6(6), N6-S5-S4-Ti 67.2(5), S5-S4-Ti-S2 −54.7(2), S4-S5-N6-C17 57.7(11), S2-S3-N6-C17 −59.1(9).

169.4 and 169.7 pm. The methyl group of **4** is in an axial position. However, the coordination sphere of the nitrogen atom is only slightly pyramidal (sum of bond angles 347°). Similar observations have been made with several cyclic sulfurimides;[11] these findings have been explained by hyperconjugation of the nitrogen lone pair with the unoccupied σ^* orbitals at the neighboring S–S bonds.[12] The ^1H NMR spectra of **4** and **5** demonstrate that the six-membered rings are rigid at 23 °C (two signals for the Cp protons). That the connectivity of **5** is the same as in **4** follows from the ^1H NMR spectrum of the derivative **6**, which was prepared with C_5H_4Me ligands at the titanium atom. In the region of aromatic ring protons the spectrum consists of two triplets and one pseudo-quartet, indicating C_s symmetry; in the case of a lower symmetry (e.g., sequence of ring atoms TiSNSSS) up to eight signals are expected.

Complexes **4**–**6** react rapidly and quantitatively with either SCl_2 or S_2Cl_2 at 20 °C in CH_2Cl_2 to the novel cyclic sulfurimides **7** and **8** [Eq. (d)].

$$[Cp_2Ti(\mu\text{-}S_2)_2NR] + S_mCl_2 \rightarrow S_{4+m}NR + [Cp_2TiCl_2] \qquad \text{(d)}$$

$$R = n\text{Oct} \qquad \mathbf{7}\ (m=1) \qquad \mathbf{8}\ (m=2)$$

Compounds **7** and **8** are pale yellow oils rather than solids as a consequence of the long alkyl chain. They are soluble in *n*-hexane, CH_2Cl_2, and CS_2. In the EI mass spectra the molecular ions were observed with medium intensity; in addition, only signals of fragments of the types S_n^+ and S_nNR^+ were detected. Heterocycle **7** decomposes at 20 °C within a few hours, **8** within one day; however, both compounds may be kept at −25 °C for longer periods of time without decomposition. Decomposition products are S_8 and the homologous molecules $S_nNn\text{Oct}$ with $n \geq 6$. These species can easily be detected by reversed-phase HPLC analysis,[13] since the retention times systematically increase with increasing ring size. For comparison the eight-membered $S_7Nn\text{Oct}$ has been prepared from S_7NH and *n*OctI.[14]

For the pure compounds $S_nNn\text{Oct}$ ($n = 5-7$) only *one* peak was observed in the HPLC analysis. The vibrational and ^1H NMR spectra of the three cyclic octyl-substituted sulfurimides differ markedly, allowing their identification even in mixtures. The chemical shifts of the α- and β-protons of the *n*-octyl groups depend on the ring size (Table 1).

Table 1. ^1H NMR chemical shifts (δ values) and signal multiplicities (200 MHz, $CDCl_3$, 23 °C) of $S_nNn\text{Oct}$ ($n = 5-7$).

$S_5Nn\text{Oct}$	$S_6Nn\text{Oct}$	$S_7Nn\text{Oct}$	
3.90 t (2H)	3.20 t (2H)	3.28 t (2H)	α-CH_2
1.60 q (2H)	1.70 q (2H)	1.69 q (2H)	β-CH_2
1.29 s br (10H)	1.30 s br (10H)	1.30 s br (10H)	-$(CH_2)_5$-
0.89 t (3H)	0.89 t (3H)	0.90 t (3H)	-CH_3

Our findings show that the reactivity of **1** towards sulfur compounds is more versatile than might have been anticipated. Thus, access is provided to novel and unexpected titanocene complexes, which, in turn, may serve as reagents in many new ligand transfer reactions as has been demonstrated by several groups for the now already classical case of $[Cp_2TiS_5]$.[15]

Experimental Section

4: Under an inert atmosphere **1** (304 mg, 1.3 mmol) in *n*-hexane (50 mL) was added dropwise over 2 h to 1,4-$S_4(NMe)_2$ (242 mg, 1.3 mmol) in *n*-hexane (30 mL) at 20 °C and stirred for an additional 15 h. The resulting precipitate was isolated, and the brown-black filtrate reduced in volume to give a dark brown oil, which was purified by column chromatography (SiO_2, *tert*-butyl methyl ether/*n*-hexane 1:2). Yield: 134 mg of needle-shaped black crystals (30 %). ^1H NMR (200 MHz, $CDCl_3$, 23 °C): $\delta = 6.40$ (s, 5H), 6.21 (s, 5H), 2.29 (s, 3H); MS (144 °C, EI): 335 (M^+, 5 %), 242 ($Cp_2TiS_2^+$, 98 %), 209 (Cp_2TiS^+, 15 %), 178 (Cp_2Ti^+, 100 %); UV-Vis (methanol): $\lambda_{max} = 207, 233, 313, 425, 481$ nm (diode array detector).

5: As in the case of **4**, $[Cp_2Ti(CO)_2]$ (609 mg, 2.6 mmol) in *n*-hexane (80 mL) and 1,4-$S_4(Nn\text{Oct})_2$ (995 mg, 2.6 mmol) in *n*-hexane (70 mL). Yield: 627 mg of a dark brown oil (43 %). Correct C,H,N,S analysis; ^1H NMR (200 MHz, $CDCl_3$, 23 °C): $\delta = 6.37$ (s, 5H), 6.22 (s, 5H), 2.25 (t, 2H), 1.47 (q, 2H), 1.25 (br s, 10H), 0.88 (t, 3H); UV-Vis (methanol): $\lambda_{max} = 210$, 233, 314, 428, 490 nm (diode array detector).

6: As in the case of **5**, $[(C_5H_4Me)_2Ti(CO)_2]$ (315 mg, 1.2 mmol) in *n*-hexane (50 mL) and 1,4-$S_4(Nn\text{Oct})_2$ (459 mg, 1.2 mmol) in *n*-hexane (50 mL). Yield: 138 mg of a dark brown oil (25 %). ^1H NMR (200 MHz, $CDCl_3$, 23 °C): $\delta = 6.32$ (t, 2H), 6.25 (t, 2H), 6.07 (q, 4H), 2.39 (s, 3H), 2.39 (t, 2H), 1.97 (s, 3H), 1.49 (q, 2H), 1.25 (br s, 10H), 0.88 (t, 3H); UV-Vis (methanol): $\lambda_{max} = 212, 240, 310, 432, 484$ nm (diode array detector).

7: SCl_2 (0.32 mmol, 10 mL of a 0.032 M solution in CH_2Cl_2) was added dropwise to a solution of **5** (137 mg, 0.32 mmol) in dichloromethane (70 mL) at 20 °C. After the mixture had been stirred for 30 min, the orange-colored solution was filtered through a layer of SiO_2 (10 cm) to remove $[Cp_2TiCl_2]$. The yellow solution was reduced in volume to give **7** (80 mg, 87 %). ^1H NMR (200 MHz, $CDCl_3$, 23 °C): $\delta = 3.90$ (t, 2H), 1.60 (q, 2H), 1.29 (br s, 10H), 0.89 (t, 3H); MS (60 °C, EI): 287 (M^+, 20 %), 255 ($S_4Nn\text{Oct}^+$, 7 %), 223 ($S_3Nn\text{Oct}^+$, 15 %), 191 ($S_2Nn\text{Oct}^+$, 57 %), 160 ($SNn\text{Oct}^+/S_5^+$, 11 %), 126 ($Nn\text{Oct}^+$, 100 %); UV-Vis (methanol): $\lambda_{max} = 220, 288$ nm (diode array detector).

8: As in the case of **7**, S_2Cl_2 (0.056 mL, 0.7 mmol) and **5** (297 mg, 0.7 mmol) in dichloromethane (200 mL). Yield: 204 mg of a pale yellow oil (91 %). ^1H NMR (200 MHz, $CDCl_3$, 23 °C): $\delta = 3.20$ (t, 2H), 1.70 (t, 2H), 1.30 (br s, 10H), 0.89 (t, 3H); MS (69 °C, EI): 319 (M^+, 16 %), 255 ($S_4Nn\text{Oct}^+$, 32 %), 191 ($S_2Nn\text{Oct}^+$, 13 %), 160 ($SNn\text{Oct}^+/S_5^+$, 29 %), 126 ($Nn\text{Oct}^+$, 100 %); UV-Vis (methanol): $\lambda_{max} = 212, 294$ nm (diode array detector).

Received: September 15, 1997 [Z10923IE]
German version: *Angew. Chem.* **1998**, *110*, 515–517

Keywords: heterocycles · nitrogen · sulfur · titanium

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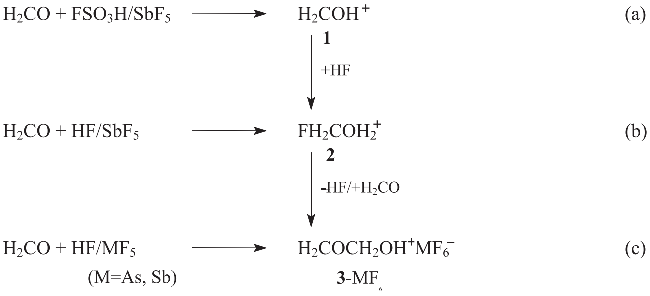
Formaldehyde in Super Acids: A Succession of Products from Carbenium through Oxonium Ion to Hydroxymethyl(methylidene)oxonium Salts

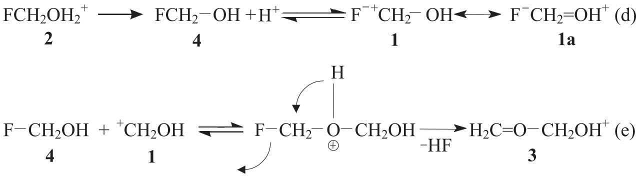
Rolf Minkwitz,* Stefan Schneider, and Hans Preut

The products of the reaction of formaldehyde with super acids were first examined by G. A. Olah et al.[1] The hydroxycarbonium ion **1** was identified in FSO_3H/SbF_5 [Eq. (a)] by NMR spectroscopy, and fluormethyloxonium ions **2** [Eq. (b)] were found after addition of HF or directly in HF/SbF_5.[2, 3] We attempted to isolate salts of monofluoromethanol, which is unknown in pure substance, from the HF solution according to Equation (b). Surprisingly, we obtained hydroxymethyl(methylidene)oxonium hexafluorometalate **3**-MF_6 at −78°C according to Equation (c) but no polymerization product of formaldehyde, as proposed by G. A. Olah et al.[3]



$$\begin{array}{lcll}
\mathrm{H_2CO + FSO_3H/SbF_5} & \longrightarrow & \underset{\mathbf{1}}{\mathrm{H_2COH^+}} & \text{(a)} \\
 & & \big\downarrow {\scriptstyle +HF} & \\
\mathrm{H_2CO + HF/SbF_5} & \longrightarrow & \underset{\mathbf{2}}{\mathrm{FH_2COH_2^+}} & \text{(b)} \\
 & & \big\downarrow {\scriptstyle -HF/+H_2CO} & \\
\underset{(M=As,\ Sb)}{\mathrm{H_2CO + HF/MF_5}} & \longrightarrow & \underset{\mathbf{3}\text{-}\mathrm{MF_6}}{\mathrm{H_2COCH_2OH^+MF_6^-}} & \text{(c)}
\end{array}$$

The formation of cation **3** might proceed in a simple way upon addition of formaldehyde to protonated formaldehyde **1**. However, no indications for **1** were found in the NMR spectra of the HF solution. Only signals for **2**, which exclusively exists in solution, were observed ($\delta(^{13}C) = 100.4$, $^1J_{C,F} = 222$ Hz); this confirms previous results by Olah et al. The stabilization in solution of protonated molecules for which the unprotonated form cannot be found is known and was recently described by Olah et al. for the example of protonated fluoroformic acid.[3, 4] The existence of cation **3** in solution cannot be proved. Considering the NMR spectroscopic results, it seems more likely that **3** is formed from **2** by a reversible electrophilic addition with subsequent elimination of HF [Eqs. (d) and (e)]. With the exception of **2**, no experimental proof was found for the intermediates which are formally required.



$$\underset{\mathbf{2}}{\mathrm{FCH_2OH_2^+}} \longrightarrow \underset{\mathbf{4}}{\mathrm{FCH_2{-}OH}} + \mathrm{H^+} \rightleftharpoons \underset{\mathbf{1}}{\mathrm{F^{-}\,{}^{+}CH_2{-}OH}} \longleftrightarrow \underset{\mathbf{1a}}{\mathrm{F^{-}CH_2{=}OH^+}} \quad \text{(d)}$$

$$\underset{\mathbf{4}}{\mathrm{F{-}CH_2OH}} + \underset{\mathbf{1}}{\mathrm{{}^{+}CH_2OH}} \rightleftharpoons \mathrm{F{-}CH_2{-}\overset{\overset{\displaystyle H}{|}}{\underset{\oplus}{O}}{-}CH_2OH} \xrightarrow[-HF]{} \underset{\mathbf{3}}{\mathrm{H_2C{=}O{-}CH_2OH^+}} \quad \text{(e)}$$

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