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# Selenium and Tellurium Derivatives of Corannulene: Serendipitous Finding of a One-Dimensional Stereoregular Coordination Polymer Crystal Based on Te-O Backbone and Side-Chain Aromatic Array

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Abstract: Monobromo-, tetrabromo-, and pentachloro-corannulene are subjected to nucleophilic substitution reactions with toluyl selenide and phenyl telluride-based nucleophiles generated in situ from the corresponding dichalcogenides. In the case of selenium nucleophile, the reaction provides moderate yields (52-77%) of the targeted corannulene selenoethers. A subsequent oxidation of the rim selenium atoms proceeds smoothly to furnish corannulene seleneoxides in 81-93% yield. In the case of tellurides, only monosubstitution of the corannulene scaffold could be achieved albeit with concomitant oxidation of the tellerium atom. Unexpectedly, this monotelluroxide derivative of corannulene (RR'Te=O, R = Ph, R' = corannulene) is observed to form a linear coordination polymer chain in the crystalline state. In this chain, Te-O constitutes the polymer backbone around which the aromatic groups (R and R') arrange as polymer side-chains. The polymer crystal is stabilized through intramolecular  $\pi$ - $\pi$  stacking interactions of the side-chains and intermolecular hydrogen and halogen bonding interactions with the solvent (chloroform) molecules. Interestingly, each diad of the polymer chain is racemic. Therefore, in terms of stereoregularity, the polymer chain can be described as syndiotactic.

Corannulene is a bowl-shaped polycyclic aromatic hydrocarbon ( $C_{20}H_{10}$ ), which can be envisaged as a fragment of fullerene  $C_{60}$ . Interestingly, however, Barth and Lawton created this molecule some two decades earlier than the discovery of buckminsterfullerene.<sup>1</sup> With some period of dormancy, Scott's gas-phase synthesis rejuvenated this area of research.<sup>2</sup> Later, Siegel re-introduced the concept of solution-phase synthesis.<sup>3</sup> In 2000, Sygula and Rabideau enhanced the practicality of the solution-phase synthesis.<sup>4</sup> Currently, Siegel's kilogram-scale synthesis is the last chapter in the illustrious synthetic history of this unique molecule.<sup>5</sup>

The practical access to corannulene has also led to a flurry of activity in studying its various properties and creating new molecules and functional materials.<sup>6</sup> Two aspects, however, still remain underexplored. 1) Despite a large number of studies on lighter chalcogen derivatives of corannulene,<sup>7</sup> stable heavier analogues with direct attachment of the heteroatom to the

aromatic scaffold remain unknown.8-9 2) Although Baldridge and coworkers have recognized that columnar packing of corannulene is essential in harnessing its electronic properties,<sup>10</sup> long-range organization of corannulene remains a challenging goal.<sup>11</sup> The primary aim of the present study was to investigate synthesis of heavier chalcogen derivatives of corannulene. However, during the course of this investigation we unexpectedly found that a tellurium derivative (RR'Te=O, R = Ph, R' = corannulene), which remains monomeric in solution, gives rise to linear polymer chains in the crystal in which the corannulene side-chains orient themselves into one column and the polymer backbone is formed by the tellurium oxygen bond. The tellurium atom is tetracoordinated. However, it does not display any secondary bonding interactions which are normally observed for organotellurium species.<sup>12</sup> In coordination polymers,<sup>13-14</sup> having a tellurium atom in the polymer backbone is also a rarity.<sup>15-18</sup> Overall, therefore, the work described here led to some planned and unplanned discoveries which are of relevance to the three broad areas of research: the chemistry of corannulene, organotellurium chemistry, and coordination polymers.

The synthesis of lighter corannulene chalcogenides is relatively simple as a large variety of the required hydroxyl or thiol reagents are commercially available and bench-stable compounds. The selenium and tellurium analogues, however, have limited commercial availability and stability. They are prone to a facile oxidative dimerization process to diselenides or ditellurides under ambient conditions. Therefore, the most practical route is to generate the selenide and telluride nucleophiles in situ from the corresponding dichalcogenides. For the synthesis of selenoethers, we have prepared di-(p-tolyl)diselenide (1) according to a modified literature procedure.<sup>19</sup> The required arylselenide nucleophile was generated in the presence of K2CO3 and dithiothreitol according to another modified procedure.<sup>20</sup> After addition of the respective halocorannulene starting material, the substitution occurred at 60 °C over 1-4 days. In this way, the reactions with monobromo- (2),<sup>21</sup> tetrabromo- (3),<sup>22</sup> and pentachlorocorannulene<sup>23</sup> (4) furnished the desired selenoethers 5, 6, and 7 in moderate to good yields (52-77%) (Scheme 1).

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Scheme 1. Synthesis of selenium derivatives of corannulene.



Scheme 2. Synthesis of a tellurium derivative of corannulene.

In the case of sulfur nucleophile, the reaction proceeds at room temperature over several days or overnight when heated and gives isolated yields of 38-80%.7i In the case of alkoxide nucleophile, a reaction temperature of 180 °C over a 2-day period is necessary (yield = 41%).7b The prepared corannulene-based selenoethers are white (5), yellow (6) or orange (7) bench stable solid compounds. Oxidation of these ethers was attempted using an excess of *m*-CPBA at room temperature. The oxidations were performed in CDCl<sub>3</sub> and we were able to monitor the conversion and byproduct profile with <sup>1</sup>H NMR by taking aliquots of the reaction mixture. Compounds 5 and 7 successfully underwent the oxidation process. However, the oxidized products (8 and 9) exhibited sensitivity towards acidic conditions (even on silica gel used for column chromatography) probably due to protodeselenation. To overcome this problem, we had to

investigate methods that do not involve silica gel for purification purposes. In addition, 8 was also found to be sensitive to an aqueous workup procedure. Therefore, 8 was purified by column chromatography on Florisil, on which no decomposition was observed, and it was obtained in a 93% yield. Due to the highly polar nature of the compound 9, chromatography on Florisil was not feasible. Exploiting the good solubility of m-CPBA and the corresponding *m*-chlorobenzoic acid in diethyl ether, we were able to precipitate 9 from the crude mixture and isolate it by a simple filtration with a yield of 81%. We were unable to isolate the tetra-selenoyl corannulene compound from ether 6, even when attempting the precipitation-filtration strategy. A decomposed mixture was obtained, possibly due to the large steric clash of the ortho arylselenoyl groups causing chemical instability. We are unable to find any examples in the literature of aromatic compounds bearing an ortho-diselencyl substitution pattern.8

For the synthesis of a corannulene-based tellurium ether, we used commercially available diphenyl ditelluride (10) as the starting material, but the target product was not formed using the same conditions as above (Scheme 2). A harsher method<sup>24</sup> yielded the product in 11% yield, using KOH and DMSO at 100 °C. However, during the routine characterization of 11. ESI-HRMS identified a monooxidized tellurium species. The oxidation occurred probably due to the presence of an oxidative (DMSO/KOH) reaction mixture as the reaction was carried out under an atmosphere of argon. However, an aerobic process cannot be ruled out as the cause for oxidation. The structure of this compound was subsequently determined by single crystal X-ray crystallography and proven to be the corannulene telluroxide.<sup>25</sup> Further oxidation of this compound with *m*-CPBA yielded an insoluble black solid, which was most likely some form of tellurium oxide arising from the decomposition of the desired corannulene tellurone. Due to the product being possibly unstable under the harsh reaction conditions and the low yield, we have decided not to optimize the synthesis any further. We have attempted to synthesize the corresponding corannulene-based penta-telluroxide but the reaction failed to provide any product.



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Figure 2. Inter- and intra-molecular interactions in the crystal structure of 11.



Figure 3. Tacticity of racemic polymer diad shown with the help of chemical (top) and crystal (bottom) structures.

The backbone of the crystal structure of 11 consists of alternating Te-O bonds with bond lengths of 2.146 Å and 2.006 Å, indicating the presence of Te-O sigma bonds, which are slightly longer than the typically reported Te-O-Te linkages of polymeric telluroxides (Figures 1-3 and Figures S1-S4).<sup>12,26</sup> One of the Te-O bonds is covalent, while the other - slightly longer one - is coordinate and a result of chalcogen bonding between the tellurium  $\sigma$ -hole and the oxygen lone pair.<sup>27</sup> The molecule adapts a disphenoidal geometry where the two oxygen atoms occupy the axial positions, while the aryl substituents are located in an equatorial orientation. This geometry is responsible for the slightly distorted axial-axial bond angle of 170.08° and the equatorial-equatorial bond angle of 104.75° due to the large steric demand of the tellurium lone pair located in the equatorial position. There are no secondary bonding interactions observed besides the sole Te-O chalcogen bonding per telluroxide molecule, which is a rare case amongst these types of molecules as the tellurium atom rarely exists in a tetracoordinated form as either a rectangular Te-O dimer is formed with additional Te-O coordination from neighbouring molecules<sup>26</sup> or intramolecular stabilization occurs via adjacent aminoalkyl functionalities.<sup>28</sup> The lack of additional secondary bonding interactions can be rationalized by the large degree of π- $\pi$  stacking interactions of the aromatic side-chains in the crystal. The phenyl groups are organized in two non-overlapping columns, in an alternating fashion; while the corannulenes are packing in a single slip-stacked column. The phenyl rings interact with one (3.384 Å) and the corannulenes with two (3.351 Å and 3.347 Å)

aromatic interactions. All these stabilizing effects are observed within a single chain of the polymer, and two of these chains are found in the unit cell oriented along the c-axis. Intermolecular interactions rely upon solvent (CHCl<sub>3</sub>) molecules. A hydrogen bond exists between the oxygen atom of the backbone and CH of chloroform (2.074 Å). A bifurcated Cl-π (3.375 Å and 3.342 Å) interaction exists between the halogen atom and the corannulene nucleus. Finally, the phenyl ring interacts with the solvent molecule through a CI-H interaction (2.942 Å). Out of the 4 chloroform molecules shown in the Figure 2, only two interact with the polymer strands. In terms of stereochemistry, each polymer chain is composed of alternating R and S configurations at the tellurium atom (Figure 3). The racemic nature of the adjacent repeating units, known as racemo diads in polymer chemistry, helps to classify the polymer tacticity as syndiotactic. It has to be noted that this type of polymer structure is only found in the crystalline state, as the compound remains monomeric in solution (Figure S5).



Figure 4. UV/Vis absorption spectra for **5** (0.012 mM), **6** (0.017 mM), **7** (0.015 mM), **8** (0.007 mM), **9** (0.017 mM), and **11** (0.017 mM), in dichloromethane at 25 °C.

Finally, a comparison can be made with the reported pentasubstituted corannulene ethers and thioethers,7b and the present selenium ether 7 in terms of their electronic properties. The  $\pi$ - $\pi$ \* absorption band in the UV/Vis spectra shifts from 308 to 349 nm as we move from ethers to thioethers.7b In the case of corannulene selenoether, this red shift continues and the  $\lambda_{max}$  is located at 360 nm for compound 7 (Figure 4 and Figure S6). In summary, this work shows that mono, tetra, and pentaselenide derivatives of corannulene can be prepared through nucleophilic substitution reaction with an aryl selenide at 60 °C in 1-4 days. In comparison, the sulfur analogues can be accessed overnight and the oxygen analogues require much higher temperature (140 °C). The oxidation of the selenium atoms can be achieved as easily as the known oxidation of the sulfur atoms present on the rim of corannulene.7i However. unlike corannulene sulfones. corannulene selenoxides are prone to decomposition in aqueous and acidic conditions. However, once purified, they can be stored for long periods of times. In the case of corannulene tellurides, only monosubstitution can be achieved albeit with concomitant

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oxidation of the tellurium atom. This derivative gives rise to diorganotelluroxide-based linear polymer chains in which the lack of secondary bonding interactions has been rationalized by a large degree of  $\pi$ - $\pi$  stacking interactions in the corannulene and phenyl side-chains as the major organizing factor in the polymer crystal. Since the presence of tellurium in the molecular structure enables long exciton diffusion lengths through the formation of triplet excitons,<sup>29</sup> and corannulene is a known electron acceptor,<sup>30</sup> the polymer crystal reported here presents an enticing material for studying charge transport properties.

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This work enhances the repertoire of chalcogen derivatives of corannulene and introduces the first example of a one-dimensional coordination polymer with a tellurium oxide backbone and organized corannulene side-chains.