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Synthesis and Characterization of Organochalcogens (S, Te, Se)

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Abstract

Organochalcogen compounds, which include sulfur (S), tellurium (Te), and selenium (Se), constitute a diverse and highly functional class of molecules with broad applications in medicinal chemistry, materials science, and catalysis. This review explores various synthetic approaches for organochalcogens, such as nucleophilic substitution, transition metal-catalyzed coupling, and cyclization reactions, aiming to enhance reaction efficiency and sustainability. Notably, microwave-assisted synthesis has emerged as an eco-friendly alternative, significantly reducing reaction time while enhancing product selectivity. The preparation of organotellurium(IV) diiodides and dithiocarbamate-functionalized tellurium compounds underscores their structural flexibility and potential in coordination chemistry. In organosulfur chemistry, the development of dithiocarbamate-functionalized organotellurium(IV) derivatives has demonstrated high reaction efficiency, achieving yields of up to 93%, with monodentate ligand coordination confirmed by spectroscopic techniques. Additionally, selenium-containing heterocycles, including selenenophenes, selenochromenes, and quinoxalines, have been synthesized via photoinduced cyclization and radical pathways, often facilitated by copper(I) iodide, iron(III) chloride, or dialkyl diselenides. Despite these advancements, challenges persist in improving compound stability, optimizing green synthetic methods, and broadening their functional applications. Furthermore, the study confirms the formation of stable organochalcogen derivatives with distinct structural and electronic properties, demonstrating potential applications in antioxidant, anticancer, and photovoltaic technologies.

Keywords

Organochalcogens, Sulfur, Selenium, Tellurium, Synthesis, Characterization, Spectroscopic Techniques.

1. Introduction

Organochalcogen compounds, incorporating sulfur (S), selenium (Se), and tellurium (Te), have gained significant attention in synthetic chemistry due to their diverse structural frameworks and wide-ranging applications in medicinal chemistry, materials science, and catalysis.

Compounds like dithiocarbamates are organosulfur, monoanionic, chelating ligands that can act as bridging ligands and form stable complexes with main group, d-block, and f-block elements [1–3]. Based on the type of amines used in synthesis, they are classified as monoalkyl dithiocarbamates and dialkyl dithiocarbamates [3–5]. Rubber, agriculture, organic synthesis, analytical chemistry, photo-stabilizing polymers, material science, radiator protection, medicine, pharmaceuticals, and sulfide film semiconductor precursors are a few of these applications [5–13]. They are reported for their modulation of key proteins, possible treatment of AIDS and cancer, and antibacterial, antifungal, and antioxidant activities [14–20]. They exhibit antibacterial, antifungal, anti-inflammatory, antitumor, and antileishmanial properties [30–37].

The study of metal-containing macrocyclic compounds is very important because they have many useful properties. Because of their structural similarities, manmade macrocyclic compounds can exhibit characteristics of natural macrocycles, including metalloproteins and metalloenzymes. The study of metal-containing macrocyclic compounds is an important area of research [39]. Given that of their structural resemblance, these artificial macrocyclic complexes can mimic naturally occurring macrocycles, which included metalloenzymes and metalloproteins. [40,41]. These complexes have various applications, including biometallic activation and catalysis [42], medical uses [43], and the paint industry [44]. These substances are also well-known for their exceptional capacity to build complexes selectively [45,46], which makes them valuable catalysts [47–49]. Macrocyclic complexes with both soft (tellurium) and hard (nitrogen and oxygen) donor atoms play an important part in MOCVD processes [50-52], also in asymmetric synthesis catalyzed by transition metals synthesis [53,54]. Scientists are investigating the chemical characteristics of metal-macrocyclic ligand complexes that contain Te, which have attracted a lot of attention [55–57]. A template technique has been employed to investigate the synthesis of transition complexes of metals with tellurium-based tetraazamacrocycles [58,59]. Kumari et al. reported synthesizing divalent complexes of transition metals of ten membered dithiadiazamacrocycles containing tellurium. [60]. Furthermore, scientists have created physiologically active Ni, Pd, and Pt compounds of tetraazamacrocycles based on Te each with oxidation state two. [61]

In organochalcogen chemistry, microwave (MW) irradiation has drawn a lot of interest because of its capacity to increase reaction speeds, boost product yields, and reduce the production of byproducts. MW approaches employ localized superheating effects, which result in rapid molecular activation, in contrast to conventional heating methods that depend on external heat transfer. As a result, reaction times are lowered; frequently, hours-long reactions are reduced to a few minutes. Furthermore, softer reaction conditions are made possible by MW synthesis, which is especially advantageous for applications involving green chemistry. MW irradiation is an environmentally benign method in synthetic chemistry since it can carry out reactions without the use of solvents or with very little solvent [63-67]). Compared to traditional heating techniques, microwave-assisted synthesis has a number of benefits. First off, MW synthesis is very efficient due to its reduced reaction times; reactions can be finished in minutes as opposed to hours [68-70].

Second, MW heating encourages uniform energy distribution, which lowers side reactions and undesirable by-products, leading to improved yields and selectivity [71-76]. Finally, MW approaches reduce the amount of solvent used, which is consistent with the concepts of green and sustainable chemistry. MW irradiation is an environmentally benign synthetic tool since it can execute several reactions without the need of harmful organic solvents.

2. Synthesis of Organochalcogens

Three novel organotellurium (IV) derivatives of diphenyl dithiocarbamate—$C_4H_8Te[(CS_2)N(C_6H_5)_2]_2$ (1), $(C_2H_5)_2Te[(CS_2)N(C_6H_5)_2]_2$ (2), and $C_4H_7(CH_3)Te[(CS_2)N(C_6H_5)_2]_2$ (3)—were successfully synthesized through the reaction of sodium diphenyldithiocarbamate with $C_4H_8TeI_2$, $(C_2H_5)_2TeI_2$, and $C_4H_7(CH_3)TeI_2$, respectively. The structures of these compounds were confirmed using elemental analysis and spectroscopic techniques, including IR, ^1H NMR, and $^{13}C{^1H}$ NMR. Their antimicrobial properties were evaluated against selected bacterial and fungal strains using broth microdilution and disc diffusion methods, with penicillin and amphotericin B serving as positive controls for strains of bacteria and fungi, respectively. Within the bacterial strains, complex 3 displayed the strongest activity, whereas compound 1 demonstrated the highest antifungal potency with a 6.25 µg/ml MIC value. Overall, all three complexes displayed moderate to high antimicrobial activity. [77]

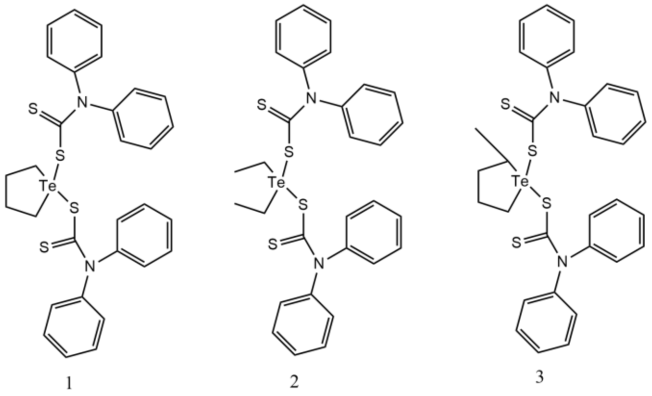


Figure 1. Molecular structures of $C_4H_8Te[S_2CN(C_6H_5)_2]_2$ **(1)**, $(C_2H_5)_2Te[S_2CN(C_6H_5)_2]_2$ **(2)**, and $C_4H_7(CH_3)Te[S_2CN(C_6H_5)_2]_2$ **(3)**.[78]

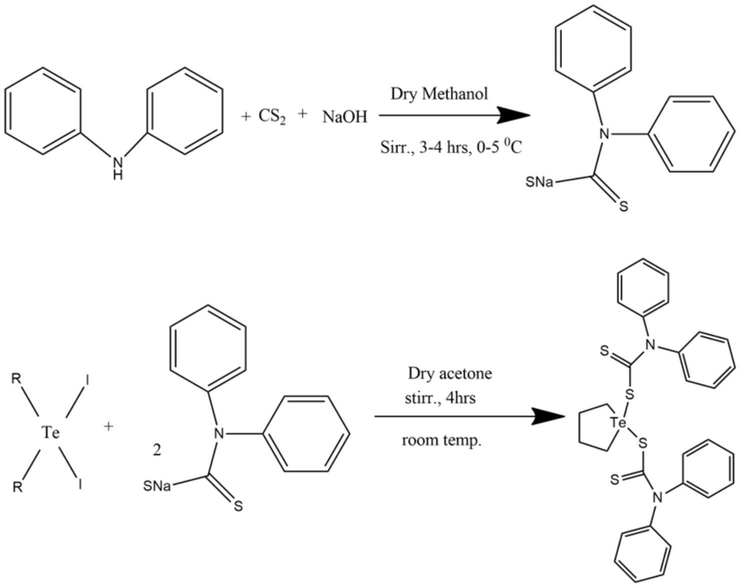


Figure 2. Synthesis of $C_4H_8Te[S_2CN(C_6H_5)_2]_2$ **(1)**, $(C_2H_5)_2Te[S_2CN(C_6H_5)_2]_2$ **(2)**, and $C_4H_7(CH_3)Te[S_2CN(C_6H_5)_2]_2$ **(3)**.[78]

Three novel 10-membered tetraazamacrocyclic Co(II) complexes featuring organotellurium groups were synthesized through template condensation. Using 1,2-diaminopropane and cobalt dichloride hexahydrate in dry methanol, 1,1-diiodo-1-telluracyclopentane, 1,1-diiodo-2-methyl-1-telluracyclopentane, and 1,1-diiodo-1,1-diethyltellurium(IV) were involved in the reaction in a 2:2:1 molar ratio. The resulting cobalt(II) complexes were characterized using elemental analysis, spectroscopic techniques, and molar conductance measurements. Additionally, agar disc diffusion and broth microdilution were used to evaluate the synthesized complexes' antibacterial properties. [78]

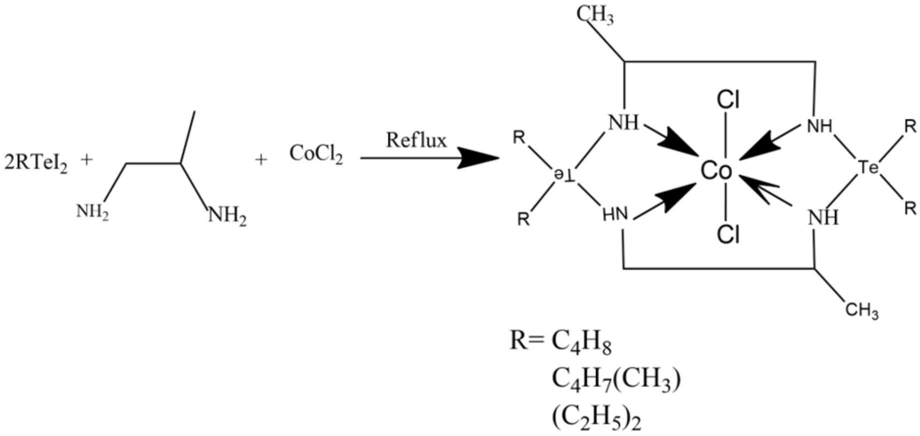


Figure 3. Co in +2 oxidation state compounds of Te with tetra-aza compounds are synthesized.

Seven novel organotellurium derivatives of carboxylic acids were synthesized through the reaction of 1,1-diiodo-1-telluracyclopentane, 1,1-diiodo-2-methyl-1-telluracyclopentane, and 1,1-diiodo-1,1-diethyltellurium(IV) with silver salts of the corresponding carboxylic acids in either a 1:1 or 1:2 molar ratio. The resulting organotellurium carboxylates have been described using IR spectroscopy, ^1H NMR ($^{13}C\{^1H\}$), and elemental analysis. Based on these studies, the compounds were assigned the general formula [$R_2Te(OCOR')_2$] and [$R_2Te(OCO)_2R''$]. The antibacterial properties of these compounds were evaluated on P. aeruginosa, E. coli, S. aureus, and K. pneumoniae implementing the broth microdilution and agar disc diffusion procedures. Among them, two compounds exhibited significant antibacterial activity at lower concentrations against all tested strains, while others demonstrated strong activity against specific bacterial species.[79]

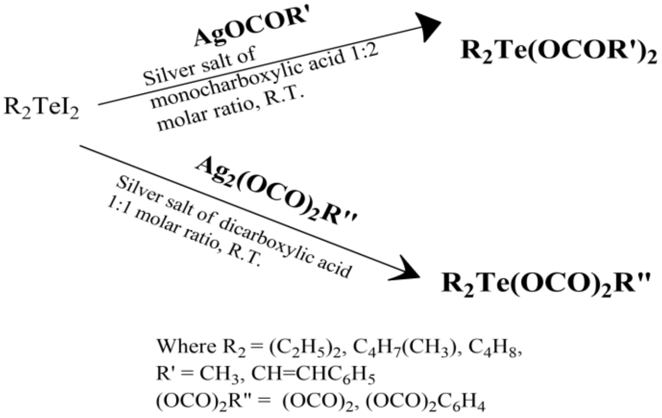


Figure 4. Synthesis of novel organotellurium derivatives of carboxylic acids

A number of new di-alkyl dithiocarbamate compounds, specifically $C_4H_8Te(S_2CNR)_2$ and $C_4H_8TeI(S_2CNR)$, were synthesized through the reaction of the corresponding silver salts of dithiocarbamates with $C_4H_8TeI_2$ and DCM(dichloromethane) at 25℃. The use of silver salts facilitated metal displacement and ensured a straightforward synthesis. IR, ^1H NMR, ^{13}C NMR, UV spectroscopy, elemental analysis was used to describe the produced compounds. The molar conductance measurements confirmed that all complexes were non-electrolytic. Spectroscopic analysis revealed that the dithiocarbamate moiety and tellurium were monodentately coordinated.

The binding of these developed complexes (ligands) to the crystal structures of Staphylococcus aureus alpha-hemolysin and cyclooxygenase-2 (prostaglandin synthase-2) at their unique active sites was examined using molecular docking. PyMOL and Protein-Ligand Interaction Profiler were used in order to visualize the protein-ligand interactions. [80]

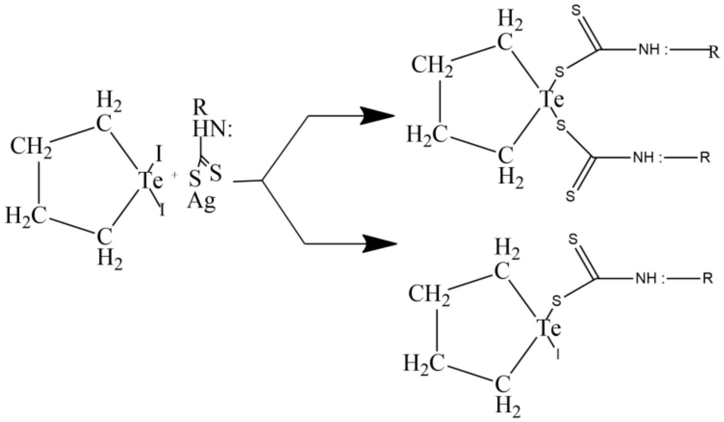


Figure 5. Synthesis of di-alkyl dithiocarbamate compounds.

The precursor 2-methyl-1,1-diiodo telluracyclopentane was used in this study to synthesize a novel series of compounds of dithiocarbamate telluracyclopentane. The synthesis, characterization, and in vitro antibacterial activity of these compounds were all thoroughly examined. The findings indicated that anisobidentate coordination modes were the most common. Metal thiocarbamates have demonstrated remarkable applications in coordination chemistry, supramolecular science, and sensor development for detecting guest substrates. Previous research has also reported the biological significance of organotellurium compounds, highlighting their antibacterial, antifungal, anti-inflammatory, and germicidal properties. AutoDock 4.2 was used to examine important metrics such hydrogen bond interactions, binding energy, and root mean square deviation (RMSD). The findings showed that the chosen ligands had binding energies that ranged from -3.66 kcal/mol to -6.14 kcal/mol for alpha-hemolysin and from -4.31 kcal/mol to -8.06 kcal/mol for cyclooxygenase-2. PyMOL software, the Protein-Ligand Interaction Profiler were used to further visualize the protein-ligand interactions. [81]

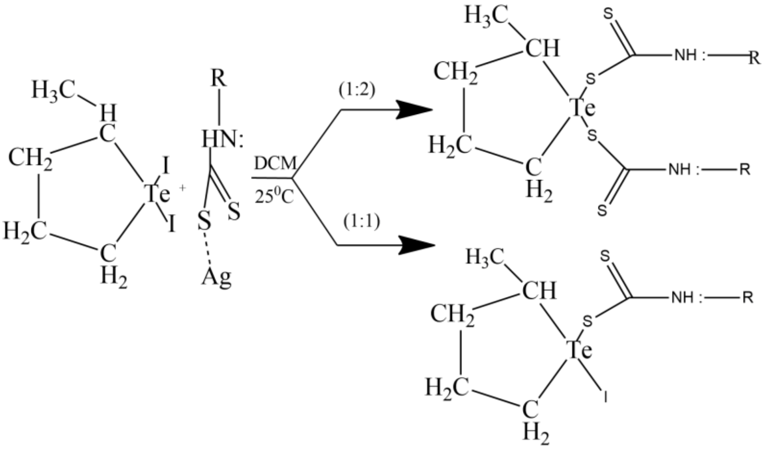


Figure 6. Synthesis of organotellurium dithiocarbamate (R=C_8H_{18}, C_7H_8, C_6H_{14}, C_4H_{10} and C_8H_{10}, DCM= dichloromethane)

A series of 1,1,2,3,4,5,6-heptahydro-1,1-di(carboxylato)telluranes, $C_5H_{10}Te(OCOR)_2$ (where R represents OCO, C_6H_5, 4-$NO_2C_6H_4$, 3,5-$(NO_2)_2C_6H_3$, or 4-$OCH_3C_6H_4$), along with 1,3-dihydro-2λ^4-benzotellurole-2,2-diyl dicarboxylates, $C_8H_8Te(OCOR)_2$ (where R includes CH_3, C_6H_5, 4-$NO_2C_6H_4$, 3,5-$(NO_2)_2C_6H_3$, 4-$OCH_3C_6H_4$, or 4-$NH_2C_6H_4$), were synthesized through the reaction of 1,1,2,3,4,5,6-heptahydro-1,1-diiodotellurane and 1,3-dihydro-2λ^4-benzotellurole-2,2-diyl diiodide with silver carboxylates.

The synthesized compounds were characterized using IR spectroscopy, as well as ^1H, ^{13}C, and ^{125}Te NMR spectroscopy. Additional analyses, includes thermogravimetric analyses, $^{13}C/^{125}Te$ CP/MAS spectrum, and UV spectroscopy., were conducted for selected complexes. The structures of $C_5H_{10}Te(OCOC_6H_5)_2$ and $C_8H_8Te(OCOC_6H_5)_2$ were ascertained using X-ray diffraction on a single crystal.

Two –CH_2 – atoms (from C_5H_{10} and C_8H_8) and A lone pair with stereochemical activity were positioned equatorially, while two O atoms from monodentate benzoate groups occupied axial positions in the tellurium center, which displayed a pseudo-trigonal bipyramidal coordination geometry in both instances. Unlike its progenitor, $C_5H_{10}TeI_2$, which has a trimeric structure, the crystal structure of $C_5H_{10}Te(OCOC_6H_5)_2$ showed polymeric zig-zag chain associations. Meanwhile, $C_8H_8Te(OCOC_6H_5)_2$ exhibited tetrameric stair-like associations (as opposed to the polymeric arrangement found in its precursor, $C_8H_8TeI_2$).[82]

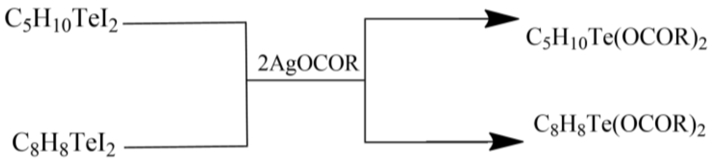
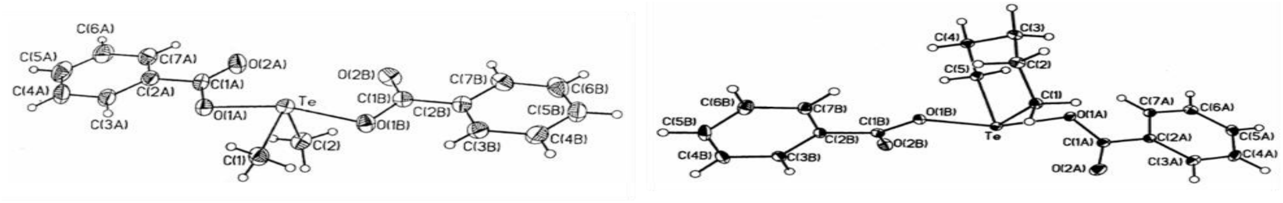


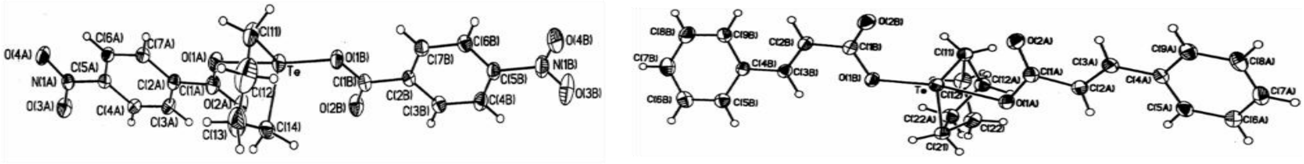
Figure 7. Synthesis of derivatives of $C_5H_{10}Te(OCOR)_2$ and $C_8H_8Te(OCOR)_2$[82]

The reactions of R_2TeI_2 (where R_2 = $(CH_3)_2$, C_4H_8, C_5H_{10}) with silver carboxylates AgOCOR (where R = C_6H_5, 4-$NO_2C_6H_4$, CH=CHC_6H_5) in a 1:2 molar ratio resulted in the formation of diorganotellurium dicarboxylates: $(CH_3)_2Te(OCOC_6H_5)_2$ (1), $C_5H_{10}Te(OCOC_6H_5)_2$ (2), $C_4H_8Te(OCO\text{-}4\text{-}NO_2C_6H_4)_2$ (3), and $C_4H_8Te(OCOCH{=}CHC_6H_5)_2$ (4).

In addition to solution-phase (^1H, ^{13}C, and ^{125}Te) and solid-state (^{13}C and ^{125}Te) NMR spectroscopy, these compounds were investigated by IR spectroscopy. The tellurium center of compounds 1–4 exhibits a trigonal bipyramidal shape that is deformed and has a stereochemically active lone pair, based on an X-ray diffraction examination of a single crystal. These complexes' function in the creation of supramolecular assemblies was further highlighted by a study of their capacity to create intermolecular C-H-O hydrogen bonds. [83]



Crystal structure of 1 Crystal structure of 2

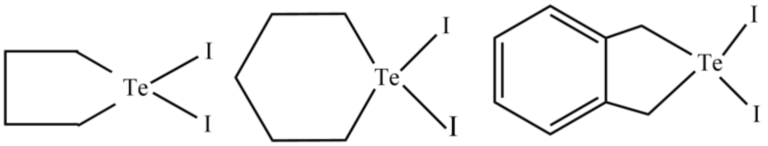


Crystal structure of 3 Crystal structure of 4

Figure 8. Crystal structure of 1, 2, 3, and 4.[83]

A comparative analysis of heterocyclic organotellurium diiodide crystal structures, namely 1,1,2,3,4,5-hexahydro-1,1-diiodotellurophene ($C_4H_8TeI_2$) (1), 1,1,2,3,4,5,6-heptahydro-1,1-diiodotellurane ($C_5H_{10}TeI_2$) (2), and 1,3-dihydro-2λ⁴-benzotellurole-2,2-diyl diiodide ($C_8H_8TeI_2$) (3), has been carried out concerning bond lengths, bond angles, and polymorphism. The parameters analyzed include (a) Tellurium–I, (b) Tellurium –C, (c) I– Tellurium –I, (d) C– Tellurium–C, (e) C– Tellurium –I, as well as intermolecular secondary bonding interactions such as (f) Tellurium-I, (g) I– Tellurium -I, and (h) C– Tellurium-I.

By employing X-ray diffraction research on a single crystal, the crystal structures of the three heterocyclic organotellurium diiodides were found. The tellurium atom takes on a distorted octahedral shape in each case., with Te···I secondary interactions giving rise to different supramolecular architectures: compound 1 forms two-dimensional zigzag ribbons, compound 2 exhibits trimeric molecular aggregates, while compound 3 adopts a three-dimensional polymeric structure. [84]



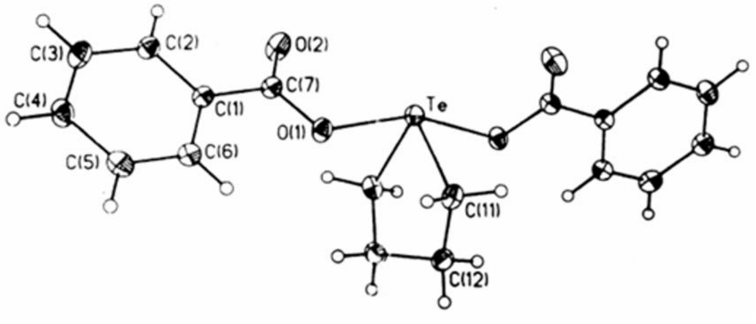
1 2 3

Figure 9. Structures of heterocyclic organotellurium diiodides

1,1,2,3,4,5-Hexahydro-1,1-dicarboxylatotellurophenes, $C_4H_8Te(OCOR)_2$, were synthesized through the reaction of 1,1,2,3,4,5-hexahydro-1,1-diiodotellurophene with silver oxide and carboxylic acids or silver carboxylates. The resulting compounds were characterized using IR, as well as (^1H, ^{13}C, ^{125}Te)-NMR spectroscopy. Using single-crystal X-ray diffraction analysis, the structures of $C_4H_8Te(OCOC_6H_5)_2$ and $C_4H_8Te(4\text{-}NO_2C_6H_4OCO)_2$ were identified. With two O atoms from the unidentate carboxylate groups occupying axial positions and two –CH_2- atoms from the C_4H_8 group and a stereochemically active lone electron pair occupying equatorially, tellurium center displays a pseudotrigonal bipyramidal coordination geometry in both instances. Additionally, the molecular packing within the unit cells is influenced by weakly bridged dimeric associations through intermolecular Te···O secondary bonding interactions. [85]

$$C4H8TeI2 + 2AgOCOR \rightarrow C4H8Te(OCOR)2 + 2AgI$$

Where R = C_6H_5, 4-$NO_2C_6H_4$, 4-$OCH_3C_6H_4$, 3,5-$(NO_2)_2C_6H_3$,



Crystal structure

Figure 10. Structure of $C_4H_8Te(OCOC_6H_5)_2$ crystal[85]

The comparative ^{1}H-NMR spectra in the solid-state crystal formations and in solution, dimethyltellurium dihalides ($Methyl_2TeX_2$, where X = Cl, Br, I) are discussed. The dimethyltellurium dicarboxylates ($Me_2Te(OCOC_6H_5)_2$, $Me_2Te(OCOCH_3)_2$, $Me_2Te(OCO)_2$) were synthesized through the reaction of Me_2TeI_2 with silver carboxylates. These chemicals were characterized by ^{1}H-NMR, ^{13}C-NMR, and IR spectroscopy. The structures for Me_2TeBr_2 and $Me_2Te(OCOC_6H_5)_2$ were determined by single crystal X-ray diffraction studies. The tellurium atom with the stereochemically active electron lone pair is surrounded by a deformed trigonal bipyramidal shape in both. Additionally, the dicarboxylate derivative features weak intermolecular C−H···O interactions. [86]

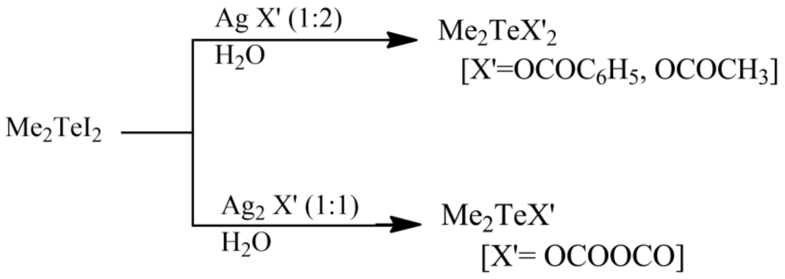


Figure 11. Synthesis of dimethyltellurium dihalides

In addition to an improved approach for preparing $[(C_2H_5)_4N]_2TeI_6$, $[(CH_3)_4N]_4TeI_8$, $[(C_6H_5)_4As]_2TeI_4$, and $[(C_6H_5)_4Sb]_2TeI_4$ are synthesized. Furthermore, novel complexes of anionic $(CH_3)_2TeI_2$ of general formula $[R_4M][(CH_3)_2TeI_2X]$ (R = C_2H_5, C_6H_5; M = N, P, Sb; X = Cl, Br, I) are synthesized and characterized. IR spectroscopy, ^{1}H-NMR spectroscopy, molecular weight determination, elemental analysis, and molar conductance were used in the characterisation process. [87]

$$(CH_3)_2TeI_2 + R_4MX \rightarrow [R_4M]\,[(CH_3)_2TeI_2X]$$

The highest yield of 80.60% was observed for $[(C_2H_5)_4P]\,[(CH_3)_2TeI_2Cl]$, with a melting point of 308°C.

2.2. Selenium-based Organocompounds

In order to create benzo[b]selenophenes, Perin et al. discovered that Oxone®, a green oxidant, stimulated electrophilic cyclization processes. In the presence of ethanol and Oxone®, diorganyl diselenide was refluxed with alkyne (2-butylselanyl functionalized phenylselenoalkyne) for two to three hours under an inert atmosphere of argon. While unsubstituted or electron-deficient diphenyl diselenide produced up to 75% product, the diselenide's reactivity was increased by the electron-releasing group in the phenyl ring, which produced a 94% yield. [88]

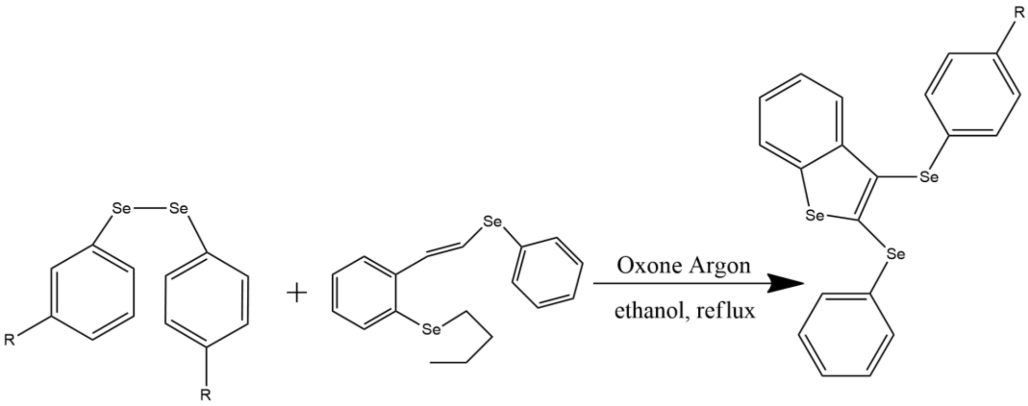


Figure 12. Synthesis of benzo[b]selenophenes using Oxone (where R=H, Me, Cl, F)

The 2-aryl-4-phenyl-5,6,7,8-tetrahydro-4H-selenochromenes synthesized and has been reported using an in-situ generated selenium reagent. In this method, hydrogen selenide (H_2Se) is produced from the reaction of zinc selenide (ZnSe) with phosphorus trichloride (PCl_3) in methanol, serving as the selenium source. The reaction proceeds under continuous stirring for 30–40 hours, leading to the formation of the desired selenochromenes in 71–88% yield. This approach provides an efficient route to selenium-containing heterocycles with potential biological and material applications. [89]

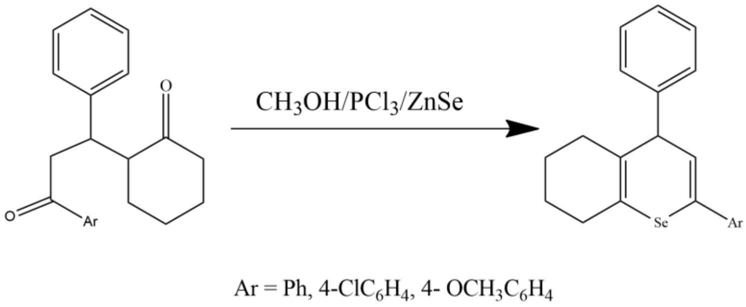


Figure 13. Synthesis of 2-aryl-4-phenyl5,6,7,8-tetrahydro-4H-selenochromenes

An increased quantity of hexahydrate iron(III) chloride (3 eq.) and dialkyl diselenides (2.5 eq.) were needed to optimize reaction conditions for the production of quinoline-based, five-cyclic selenophene-condensed heteroacenes utilizing 3-(1,3,5-triynyl) quinolines. With yields ranging from 75 to 84%, Thieno[2,3-b]quinolines condensed with diselenophene and diselenopheno[2,3-b]quinolines were effectively produced under these circumstances. [90]

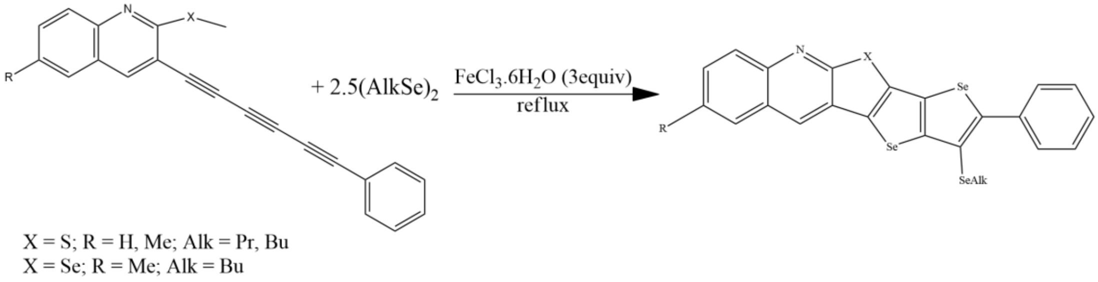


Figure 14. The preparation of diselenophene-bridged quinolines and diselenophene-fused thieno[2,3-b]quinolines.

An effective photoinduced cyclization process involving o-diisocyanoarenes and organic diselenides or thiols produced chalcogenated quinoxalines. It is believed that a radical mechanism underlies the reaction with organic diselenides. An extensive collection of quinoxalines that have been replaced with organylselanyl and are recognized for their abilities as oxidants with remarkable biological activity, can be developed by this method. [91]

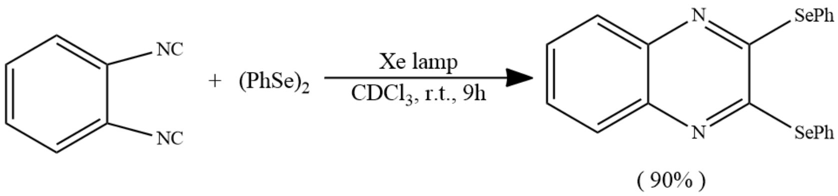


Figure 15. The preparation of quinoxalines with organylselanyl groups.

Condensed 3-butylselanyl-selenophene was produced from 3-butylselanyl-2-alkynylindoles indoles using a simple process that produced yields of 55–70%. Using an Fe(III) Cl/dibutyl diselenide system, the reaction was conducted in CH_2Cl_2 at room temp., successfully promoting the heterocyclic framework's cyclization and functionalization. [92]

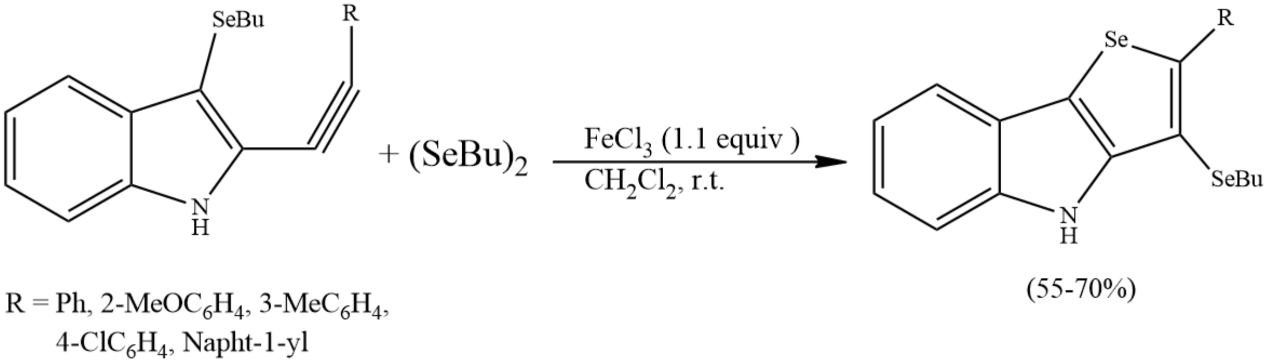


Figure 16. The $FeCl_3$/dibutyl diselenide-mediated 3-butylselanyl-2-alkynylindoles, leading to formation of 3-butylselanyl-selenophene-fused indoles.

2.3 Sulfur-based Organocompounds

Dithiocarbamate-Functionalized Organotellurium(IV) Compounds (Ref. [38]): 1,1-Diiodotelluracyclopentane, diethyltellurium-1,1-diiodide, and 2-methyl-1,1-diiodotelluracyclopentane reacted with sodium diphenyl dithiocarbamate in dry acetone under MW conditions. This approach led to enhanced reaction efficiency and high product purity compared to conventional reflux methods with 93% yield. The monodentate coordination of dithiocarbamate ligands was confirmed via IR, ^{1}H NMR, and ^{13}C NMR spectroscopy, highlighting their structural integrity and stability.

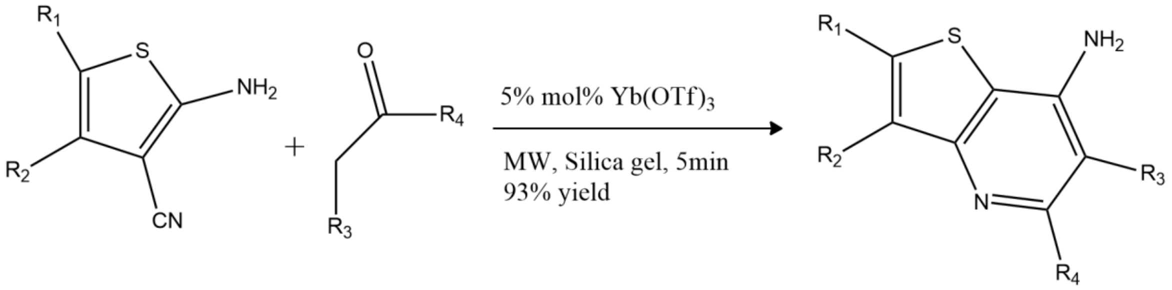


Figure 17. Synthesis of amino-thieno[2,3-b]pyridine derivatives catalyzed by $Yb(OTf)_3$.

3. Conclusion

The synthesis and characterization of organochalcogens (S, Se, Te) continue to be a focal point in materials science, medicinal chemistry, and catalysis. Advances in microwave-assisted synthesis have enhanced reaction efficiency while promoting eco-friendly, solvent-free approaches. The integration of macrocyclic and dithiocarbamate-functionalized frameworks has led to materials with promising electronic and biological properties. Additionally, the green synthesis of selenium nanoparticles (SeNPs) offers a sustainable route for biomedical applications. Future research should focus on enhancing stability, scalability, and functionalization to expand their industrial and medical potential.

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