2-Pyridylselenenyl versus 2-Pyridyltellurenyl Halides: Symmetrical Chalcogen Bonding in the Solid State and Reactivity towards Nitriles

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Abstract: The synthesis of 2-pyridyltellurenyl bromide via Br2 oxidative cleavage of the Te–Te bond of dipyridylditelluride is reported. Single-crystal X-ray diffraction analysis of 2-pyridyltellurenyl bromide demonstrated that the Te atom of 2-pyridyltellurenyl bromide was involved in four different noncovalent contacts: Te⋯Te interactions, two Te⋯Br ChB, and one Te⋯N ChB contact forming 3D supramolecular symmetrical framework. In contrast to 2-pyridylselenenyl halides, the Te con-
genner does not react with nitriles furnishing cyclization products. 2-Pyridylselenenyl chloride was demonstrated to easily form the corresponding adduct with benzonitrile. The cyclization product was studied by the single-crystal X-ray diffraction analysis, which revealed that in contrast to earlier studied cationic 1,2,4-selenadiazoles, here we observed that the adduct with benzonitrile formed supramolecular dimers via Se⋯Se interactions in the solid state, which were never observed before for 1,2,4-selenadiazoles.

Keywords: nitriles; noncovalent interactions; chalcogen heterocycles; chalcogen bonding; tellurium

1. Introduction

The field of noncovalent interactions has experienced rapid growth and constitutes one of the most intensely studied areas of current chemistry. Noncovalent interactions allow the design and construction of supramolecular materials and control of their ultimate architectures and symmetry [1,2]. Importantly, the properties of supramolecular aggregates are different from the sum of the constituent molecules [1,3–9]. Recently, chalcogen bonding (ChB) has emerged as a powerful tool for the creation of such materials. In contrast to halogen bonding (XB) or hydrogen bonding (HB), usage of ChB in crystal engineering, preparative chemistry, sensing, etc., is still emerging [10].
We have recently showcased that the addition of 2-pyridylselenenyl halides to a triple CN bond of inactivated nitriles resulted in the formation of novel cationic 1,2,4-selenadiazoles [11]. Moreover, we showed that the Se atom in the adducts of 2-pyridylselenenyl halides and nitriles could provide two σ-holes and act as a donor ChB (Figure 1).

![Figure 1](image-url)

**Figure 1.** Schematic representation of adducts of 2-pyridylselenenyl halides and nitriles showing the position of the Se-centered σ-holes and the N atom lone pair.

It is worth mentioning that chalcogen diazoles are appealing research objects due to their utilization in the preparation of soft materials with tunable physical parameters [12–15]. ChB allows modulation of the self-assembly and, therefore, fine-tuning of charge transport within these heterocycles.

Here we describe the synthesis and crystal structure of 2-pyridyltelluranyl bromide, compare its self-assembly in the solid-state with structurally similar selenium congeners, 2-pyridylselenenyl chloride, and compare the reactivity of these two 2-pyridylchalcogenenyl halides towards benzonitrile. Interestingly, while 2-pyridylselenenyl chloride readily forms an adduct with PhCN, the Te analog does not react with benzonitrile or any other simple nitrile tested in the framework of the current study.

2. Materials and Methods

**General remarks.** All manipulations were carried out in air, unless specified. Unless specified, chemicals were purchased from the commercial sources. NMR data was obtained on a Bruker Avance neo 700; chemical shifts are given in ppm, coupling constants in Hz. C, H, S, and N elemental analyses were performed on Euro EA 3028HT CHNS/O. Py2Se2 was prepared as reported earlier [16].

**X-ray crystal structure determination.**

The single-crystal X-ray diffraction data for 15 and 16 were obtained on a three-circle Bruker D8 Venture (Kurnakov Institute of General and Inorganic Chemistry, RAS, Bremen, Germany) or Bruker D8 QUEST PHOTON-III CCD (Zelinsky Institute of Organic Chemistry, RAS, Bremen, Germany) diffractometers using ϕ and ω scan mode. The diffraction data were processed using the SAINT program [17] and an absorption correction based on equivalent reflections was applied with the SADABS program [18]. Crystal data, details of data collection, and results of structure refinement are summarized in Table S1. The structures were solved by the direct method and refined on F2 with anisotropic displacement parameters for non-hydrogen atoms. The hydrogen atoms in all compounds were placed in calculated positions and refined within the riding model with fixed isotropic displacement parameters (Uiso(H) = 1.5Ueq(C) for the CH3-groups and 1.2Ueq(C) for the other groups). All calculations were carried out using the SHELXTL program [19] and OLEX2 program package [20].

Crystallographic data for all investigated compounds have been deposited with the Cambridge Crystallographic Data Center, CCDC 2113480 and 2113481. Copies of this information may be obtained free of charge from the Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or www.ccdc.cam.ac.uk accessed on 2 October 2021).

**Computational details.**

The single point calculations based on the experimental X-ray geometries of 1, 15, and 16 have been carried out at the ωB97X-D3/Sapporo-DZP-2012 level of theory [21–24].
The single point calculations based on the experimental X-ray geometries of 16 have been carried out at the B97X-D3/Sapporo-DZP-2012 level of theory [21–24]. The RIJCOSX approximation [26] and the ORCA 4.2.1 program package [25] have been utilized. The QTAIM analysis [27] has been performed by using the Multiwfn program (version 3.7) [28]. The Cartesian atomic coordinates for model supramolecular associates are presented in Table S1 and in attached xyz-files, Supplementary Materials.

**Synthesis of 15.** 2-Pyridylselenenyl chloride (1 eq, 234 µmol, 45 mg) and PhCN (4.14 eq, 970 µmol, 100 µL) were stirred in Et₂O (3 mL) at ambient temperature for 3 h. White solid precipitated, which was decanted, quickly washed with CH₂Cl₂ (1 mL), Et₂O (3 × 3 mL), and dried under vacuum. Yield: 60 mg (87%). Anal. Calcd for C₁₀H₈ClN₂Se: C, 48.75; H, 3.07; N, 9.48. Found: C, 48.91; H, 3.16; N, 9.39. ¹H NMR (700 MHz, D₂O) δ 6.8 Hz, H₁₀ and H₁₁). ¹³C {¹H} NMR δ 168.25 (C₃), 157.28 (C₉), 139.73 (C₅), 137.14 (C₈), 132.61 (C₁₅), 129.71 (C₁₀ and C₁₄), 129.63 (C₁₁ and C₁₃), 127.29 (C₁₂), 125.80 (C₇), 122.89 (C₆).

**Synthesis of 16.** A solution of bromine (200 µmol, 32 mg) in CH₂Cl₂ (10 mL) was added to a solution of di(2-pyridyl)ditelluride (200 µmol, 82 mg) in CH₂Cl₂ (10 mL), and the mixture was stirred at room temperature for 30 min. Then the solvent was removed in vacuo, the precipitate was washed with Et₂O (3 × 5 mL) and recrystallized from CH₂Cl₂. Yield: 112 mg (98%). Anal. Calcd for C₅H₄NTeBr: C, 20.99; H, 1.39; N, 4.88. Found: C, 21.02; H, 1.41; N, 4.90.

### 3. Results and Discussion

Recently we demonstrated that 2-pyridylselenenyl halides selectively react with a broad scope of nitriles to form adducts 3–14 in excellent yields (Scheme 1) [11,29].

![Scheme 1. Synthesis of 3–15.](image)

Within this work, following our interest in CN and NN triple bond activation [30–43], we attempted to prepare analogous Te derivatives. 2-Pyridyltellurenyl bromide (16) was easily synthesized by the oxidation of di(2-pyridyl)ditelluride with molecular bromine. Single crystals of 16 were grown from CH₂Cl₂ solution, and X-ray analysis demonstrated the formation of 2-pyridyltellurenyl bromide (Figure 2). Overall, metrical parameters for 16 are similar to those of earlier reported 2-pyridyltellurenyl halides [44,45].

Interestingly, in the crystal packing of 16 each Te atom is involved in four different noncovalent contacts: Te···Te interactions, two Te···Br ChB and one Te···N ChB contacts forming 3D supramolecular symmetrical framework.

In contrast, the Se analog 1, which we described earlier, does not exhibit Se···Se interactions in the crystal but features analogous Se···N ChB and terminal Se···Cl ChB forming supramolecular dimers (Figure 3) [46].
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Figure 2. Ball-and-stick representations of the crystal structures of 16, demonstrating symmetrical Te⋯Br and Te⋯N chalcogen bonding and Te⋯Te interactions. Grey and light-grey, blue, dark-green, and brown spheres represent carbon, hydrogen, nitrogen, tellurium, and bromine atoms, respectively.

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Further, we were interested whether 2-pyridyltellurenyl bromide 16 would react with nitriles in a similar fashion as the Se analogs 1 or 2, which we showed to easily react with a broad scope of nitriles. Surprisingly, 16 turned to be inert towards nitriles. 16 did not react with PhCN, CCl₃CN, or MeCN in CH₂Cl₂ at room temperature or at slight heating (60 °C).

Thus, switching from the Te to Se in 2-pyridyltellurenyl halides results in a dramatic impact on its reactivity towards nitriles.

In an extension of our earlier works, here we demonstrate that benzonitrile also easily reacts with 1 forming cationic 1,2,4-selenadiazole 15 in excellent yield (Scheme 1). Single
crystals of 15 were obtained from CH₂Cl₂, and X-ray analysis pointed to the generation of the adduct with PhCN (Figure 4).

Interestingly, the adduct 15 formed supramolecular dimers via Se⋯Se interactions in the solid-state, which we never observed before. For the earlier studied cationic 1,2,4-selenadiazoles we observed supramolecular dimerization via four-center Se⋯N ChB, Se⋯Cl ChB and H⋯Cl interactions (Figure 5), but never via Se⋯Se contacts.

Table 1. Electron densities–ρ(r), electron density Laplacians–∇²ρ(r) and appropriate λ₂ values, densities of energy–Hₐ, potential energy densities–V(r), and Lagrangian kinetic energies–G(r) (a.u.) at the bond critical points (3, −1), associated with various nontrivial noncovalent interactions in the model supramolecular associates 1, 15, and 16, and estimated strength for these contacts Eₐ₂ (kcal/mol).

| Contact * | ρ(r)  | ∇²ρ(r) | λ₂   | Hₐ   | V(r)   | G(r)   | Eₐ₂ ** |
|-----------|-------|--------|------|------|--------|--------|--------|
| Se₂5⋯Cl₂ 3.660 Å | 0.007 | 0.021  | −0.007 | 0.001 | −0.003 | 0.004  | 0.9    |
| 16        |       |        |       |      |        |        |        |
| Te₄9⋯Br₄ 3.606 Å | 0.011 | 0.027  | −0.011 | 0.001 | −0.005 | 0.006  | 1.6    |
| Te₂⋯Br₂ 3.541 Å | 0.012 | 0.031  | −0.012 | 0.001 | −0.006 | 0.007  | 1.9    |
| Te₁⋯Te₁ 3.707 Å | 0.012 | 0.028  | −0.012 | 0.000 | −0.006 | 0.006  | 1.9    |
| 15        |       |        |       |      |        |        |        |
| Se₁⋯Se₂ 3.936 Å | 0.006 | 0.018  | −0.006 | 0.001 | −0.003 | 0.004  | 0.9    |
| Se₂7⋯Cl₂ 3.900 Å | 0.006 | 0.017  | −0.006 | 0.001 | −0.003 | 0.004  | 0.9    |

* The Bondi’s (shortest) van der Waals radii for Se, Te, Cl, and Br atoms are 1.90, 2.00, 1.75, and 1.83 Å, respectively [47]. See xyz-files of model structures in Supplementary Materials for atoms numeration. ** Eₐ₂ ≈ −V(r)/2 [48].

Figure 4. Ball-and-stick representations of the crystal structures of 15, demonstrating symmetrical Se⋯Cl chalcogen bonding and Se⋯Se interactions. Grey and light-grey spheres represent carbon and hydrogen, respectively.
Figure 5. Ball-and-stick representations of the crystal structures of 6–12, demonstrating supramolecular dimerization via ChB or HB. Grey and light-grey spheres represent carbon and hydrogen, respectively.
The QTAIM analysis of model supramolecular associates 1, 15, and 16 reveals the existence of critical bond points (3, −1) for noncovalent interactions listed in Table 1 and shown in Figures 6–8. The low magnitude of the electron density (0.006–0.012 a.u.), positive values of the Laplacian of electron density (0.017–0.031 a.u.), and very close to zero energy density (0.000–0.001 a.u.) in appropriate bond critical points (3, −1) and energies for these short contacts (0.9–1.9 kcal/mol) are typical for weak noncovalent interactions involving halogen and chalcogen atoms in similar chemical systems [6,7,9,49–52]. The ratio −G(r)/V(r) > 1 at the bond critical points (3, −1) reveals that the nature of appropriate interaction is purely noncovalent [53]. The sign of λ2 can be used to distinguish bonding (λ2 < 0, attractive) weak contacts from nonbonding ones (λ2 > 0, repulsive) [54,55]. Thus, discussed weak noncovalent interactions in 1, 15 and 16 are attractive (Table 1).
In conclusion, we described the synthesis and characterization of 2-pyridyltellurenyl bromide and attempted to perform its cyclization with nitriles. In contrast to the Se analogs, 2-pyridyltellurenyl bromide does not react with nitriles. We also performed structural characterization of 2-pyridyltellurenyl bromide by the single-crystal X-ray diffraction. Interestingly, the Te atom of 2-pyridyltellurenyl bromide was involved in four different noncovalent contacts: Te···Te interactions, two Te···Br ChB and one Te···N ChB contacts forming 3D supramolecular symmetrical framework. In contrast, 1 did not exhibit Se···Se interactions in the crystal but featured similar Se···N ChB and terminal Se···Cl ChB forming supramolecular dimers. Within this study, we also prepared and performed a structural investigation of the adduct of 2-pyridylselenenyl chloride with benzonitrile. In contrast to earlier studied cationic 1,2,4-selenadiazoles here we observed that the adduct 15 formed supramolecular dimers via Se···Se interactions in the solid-state, which we never observed before.

Supplementary Materials: The following are available online at https://www.mdpi.com/article/10.3390/sym13122350/s1, Table S1: Cartesian atomic coordinates for model supramolecular associates, Table S2: Crystallographic parameters, data collection, and structure refinement details for 15 and 16.

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