Bis(vinylenedithio)tetrathiafulvalene analogues of BEDT-TTF

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Abstract
This review aims to give an overview of the current status of our research on the synthesis of π-electron donor bis(ethylenedithio)tetrathiafulvalene (BEDT-TTF, ET) analogues prepared from 1,8-diketones via a ring forming reaction. The new synthesized π-electron donors have vinyl moieties producing extended π-electron delocalization over the substituent phenyl rings at the peripheries.

Introduction
Tetrathiafulvalene (TTF, 1, Figure 1) was first synthesized in 1960s by Klingsberg’s method [1]. Shortly after this, it was synthesized by other research groups and used as a donor molecule in 1970 [2]. Although, in 1972, 1 was demonstrated to be an organic material conductor in the form of its chloride salt [3]. The interest in the chemistry of 1 began with the discovery of the salt of 1 with 7,7,8,8-tetracyanoquinodimethane (2, TTF-TCNQ) in 1973 [4]. Since then, studies have been focused on the syntheses of donor TTF analogues and investigations of the physical properties of their charge-transfer (CT) salts with various acceptors for applications such as electrically conductive materials, super conductive materials, magnetic substances, electrochromic materials, electroluminescent materials, etc. [5-16]. TTF-TCNQ, which is metallic under 54 K and known to be the first true one-dimensional synthetic metal, led to the production of superconducting salts based on TTF type donors containing a heteroatom such as sulfur, selenium, oxygen, etc. [17-20]. Among a large number of tetrathiafulvalene analogues, bis(ethylenedithio)tetrathiafulvalene (BEDT-TTF, 3), also known as ET, has been the most studied and has had the largest number of radical cation salts of its CT materials investigated at very low temperature [12,21-24].

In order to improve the properties of TTF type materials, various methods have been applied, including extension of π-conjugation through double bonds [25-30] and fused aromatic
rings [31-34] and the construction of molecules having acceptor substituents [35-37]. Generally, all these modifications served to produce molecules with better conjugation and improved S⋯S intermolecular and C–H⋯anion interactions in determining the solid state properties [35-37]. Bis(vinylenedithio)tetrathiafulvalene (BVDT-TTF) 4 (R = Ph, 4-CH$_3$OC$_6$H$_4$, 4-BrC$_6$H$_4$, 4-CH$_3$C$_6$H$_4$, 4-O$_2$NC$_6$H$_4$, 2-thienyl) is a BEDT-TTF analogue possessing π-bonds with aromatic groups on the outer rings (Figure 1) [26,38-41]. Since BEDT-TTF has two ethylene units at the both ends of the molecule, it has a non-planar structure [42]. π-Extended molecules such as 4 with a vinylene group at the end of the BEDT-TTF unit have more planar structures [41,43]. Further, a tetrathiafulvalene with a fused aromatic heterocycle was synthesized as a π-extended donor molecule [28,40]. The most notable superconductivity was observed with the radical cation salts derived from the electron-donor molecule bis(ethylenedithio)tetrathiafulvalene (BEDT-TTF) as a (BEDT-TTF)$_2$Cu[N(CN)$_2$]Br salt at 12.5 K (resistive onset) [24].

The tetrathiafulvalene (TTF) ring system is one of the most intensively studied redox-active organic molecules. It has two easily accessible oxidized states, TTF$^+$ and TTF$^{2+}$ with potentials of $E_{1/2} = +0.34$ and $E_{2/2} = +0.78$V, respectively, using Ag/AgCl in acetonitrile, (Figure 2) [5,6,44].

TTF analogues have been synthesized by coupling and without coupling methods [45,46]. Depending on the presence of electron-withdrawing groups on the TTF, they exhibit various oxidation potential ranges [15,26]. Recently, TTF and analogues have received widespread attention involving the development of new materials by using various anions to form different charge transfer salts. The physical and electronic properties of their solid states were investigated [13,25,47-49].

We attempt here to provide a summary of the synthesis of differently functionalized and extensively π-electron delocalized conjugated TTF core dithiin- and thiophene-fused donor molecules, obtained from 1,8-diketone ring closure reactions, and coupling reactions, published by our group.

**Review**

**BVDT-TTF analogues from 1,8-diketones**

Bis(vinylenedithio)tetrathiafulvalene (BVDT-TTF) 4 (R = Ph, 4-CH$_3$OC$_6$H$_4$, 4-BrC$_6$H$_4$, 4-CH$_3$C$_6$H$_4$, 4-O$_2$NC$_6$H$_4$, 2-thienyl) is a fully unsaturated analogue of BEDT-TTF (ET)$_3$. It possesses a vinyl moiety at the peripheries in place of the ethylene group of ET. It can also be considered as a tetrathiafulvalene analogue having fused 1,4-dithiin rings as its peripheries. The synthesis was achieved through the reaction of a 1,8-diketone with Lawesson’s reagent (LR) [50] or tetraphosphorus decasulfide (P$_4$S$_{10}$) [51] Although, in most cases, formation of 1,4-dithiins is the only result, or the major one, a thiophene formation can also take place [46]. So far, eighteen BVDT-TTF analogues have been synthesized (Figure 3).

In 1996, we reported a convenient method of synthesizing fused 1,4-dithiin and thiophene ring systems, possessing functional groups such as Ph 4-MeOC$_6$H$_4$ and 4-O$_2$NC$_6$H$_4$ (Scheme 1) [46]. The synthesis involved treatment of the diketone 6, produced through the reaction of the readily available dianion 5 with α-haloketones, with Lawesson’s reagent 15 to obtain [1,3-dithiolo[4,5-b][1,4]dithiin-2-thione 11, which is an analogue of half ET, as a major product, and the thiophene 13 as a minor product.

After employing different reaction conditions and an in depth study, we suggested that the reaction mechanism involves interaction of 6 with LR 15 (refluxing toluene) initially leading to
the formation of enethiols 8, a tautomer of 7, then nine-membered ring 9, rearrangement of which produces 10. Lastly, the reaction of 10 with fragment 14 of LR would give 11 as a major product (Scheme 1). Rearrangement of the 1,4-dithiin unit of 11 would produce 13 as a minor product through the intermediate 12 by the loss of elemental sulfur. The reaction of a series of 1,8-diketones with LR 15 or P₄S₁₀ was further explored in 2003 [53]. With both reactants, 1,4-dithiin 11 was obtained as a major and thiophene 13 as a minor product along with the side products 16–19 (Scheme 2).

Depending on the electron-releasing or electron-withdrawing nature of the groups on 6, the yields for 11 and 13, with LR varied between 35–52% and from not detected (n.d.) to 18%,
Scheme 1: Reaction mechanism of fused 1,4-dithiin and thiophene ring systems.

Scheme 2: Reaction conditions (i) LR, toluene, reflux, overnight; (ii) \( \text{P}_4\text{S}_{10} \), toluene, reflux, 3 h.

respectively, \( \text{P}_4\text{S}_{10} \) gave yields for 11 and 13 from 5 to 49% and n.d. to 27%, respectively. Both of the reagents produced the dithiin as a major product. Compound 13d, with a thiophene ring, was not obtained with either of the two reagents (Table 1).

A possible reaction mechanism for the formation of 16–19 was suggested to involve the intermediate 10 (Scheme 3) [46-53]. A detailed semi-empirical PM3 calculation indicated that the formation of the intermediate 10 is an endothermic process with

Table 1: Ring closure methods and product yields.

| Starting material | LR | \( \text{P}_4\text{S}_{10} \) |
|-------------------|----|--------------------------|
|                   | dithiin | thiophene | dithiin | thiophene |
| 6a R = Ph         | 11a (40%) | 13a (17%) | 11a (49%) | 13a (n.d.) |
| 6b R = 4-BrC_6H_4 | 11b (35%) | 13b (18%) | 11b (40%) | 13b (2%) |
| 6c R = 4-CH_3OC_6H_4 | 11c (45%) | 13c (15%) | 11c (30%) | 13c (27%) |
| 6d R = CH_3C_6H_4 | 11d (52%) | 13d (n.d.) | 11d (5%) | 13d (n.d.) |

n.d.: not detected.
$\Delta H_{\text{rxn}} = 29.435$ kcal/mol. The reaction of the intermediate 10 with itself could produce the 1,4-dithiin ring 11 and the side product thiophene 17 through the intermediates 21 and 22 by removal of elemental sulfur (Scheme 3). The other side products 18 and 19 were possibly formed from the reaction of 10 with 20, leading to formation of 23 and 24, rearrangement of which would then produce 18 and 19. Moreover, rearrangement of 10 via 25 would result in the formation of 16. The structures of the side products 16–19 can be taken as evidence for the proposed reaction mechanism.

The thione sulfur atoms of 11a–d and 13a–c were converted into their corresponding oxo forms 26a–d and 27a–c, respectively, using mercury acetate (Scheme 4) [46,53,54]. These were then subjected to cross coupling reactions. While the cross couplings of 26a–d with 28 and 31 [53] led to the formation of 29a–d and 32a–d, respectively, along with the self coupling products 4, 30 and 3, coupling of 27a–c with 28 gave 33a–c and the self coupling products 34a–c and 30. The cross coupled product 35 from 27c and 31 was obtained in a similar manner.

The redox properties of the donor molecules 29a–d, 32a–d, 33a–c and ET 3 were studied by cyclic voltammetry in solution in acetonitrile, containing NaClO$_4$ and dichloromethane, containing tetrabutylammonium tetrafluoroborate (TBABF$_4$) (Table 2 and Table 3). Measurements were performed under a nitrogen atmosphere at room temperature using Pt as working and counter electrodes and Ag/AgCl reference electrode. The oxidation potentials of the coupled products were compared with ET 3.

The measurements indicated that as the first oxidation potential of ET was higher than the first oxidation potential of 29a, 29c and 29d, the oxidation potential of 29b was equal to that of
Scheme 4: Reaction conditions (i) Hg(OAc)$_2$, AcOH/CHCl$_3$, rt, 1 h; (ii) (EtO)$_3$P, N$_2$, 3 h, 110 °C.

Table 2: Redox potential of 29 and ET 3. in 1 mM MeCN solution, NaClO$_4$ (0.1 M) vs Ag/AgCl, 100 mVs$^{-1}$.

| Donor | $E_{ox}^1$ (V) | $E_{ox}^2$ (V) | $\Delta E_{ox}$ (V) |
|-------|---------------|---------------|--------------------|
| 29a   | 0.49          | 0.63          | 0.14               |
| 29b   | 0.50          | 0.63          | 0.13               |
| 29c   | 0.47          | 0.72          | 0.25               |
| 29d   | 0.42          | 0.66          | 0.24               |
| 3 ET  | 0.50          | 0.77          | 0.27               |

Table 3: Redox potential of 32a–d and 33a–c. and ET 3 in 1 mM CH$_2$Cl$_2$ solution, TBABF$_4$ (0.1 M) vs Ag/AgCl, 115 mVs$^{-1}$.

| Donor | $E_{ox}^1$ (V) | $E_{ox}^2$ (V) | $\Delta E_{ox}$ (V) |
|-------|---------------|---------------|--------------------|
| 32a   | 0.66          | 0.96          | 0.30               |
| 32b   | 0.60          | 0.95          | 0.35               |
| 32c   | 0.68          | 1.00          | 0.32               |
| 32d   | 0.64          | 0.99          | 0.24               |
| 33a   | 0.59          | 0.86          | 0.27               |
| 33b   | 0.51          | 0.83          | 0.32               |
| 33c   | 0.62          | 0.94          | 0.32               |
| 3 ET  | 0.51          | 0.85          | 0.34               |

ET 3 and the second oxidation potentials of 29a–d were found to be lower than for ET. On the other hand, the first and second oxidation potentials of the donors 33a–c were slightly higher than the oxidation potentials of ET. The oxidation potentials of the donor molecules 32a–d were higher than the ET 3 oxidation potential.

A BEDT-TTF analogue containing phenyl-l,4-dithiin and 2,3-dihydroxybutane-l,4-dithio at the periphery, 43, was reported as a new highly functionalized donor molecule (Scheme 5) [54].
Scheme 5: Reaction conditions (i) iPr₂NEt, MEMCl, THF, rt, 12 h; (ii) LiAlH₄, dry ether, rt, 24 h; (iii) tosyl chloride, dry pyridine, 0 °C, 4 h; (iv) KI, dry acetone, N₂, reflux, overnight; (v) dry THF, 75 °C, N₂, 48 h; (vi) P₄S₁₀, toluene, reflux, 3 h; (vii) Hg(OAc)₂, AcOH/CHCl₃, rt, 2 h; (viii) neat (EtO)₃P, N₂, 3 h; (ix) sample in THF at 0 °C, add 20% HCl, then rt., overnight.

The reaction of diketone 6a, with LR 15 in refluxing toluene for 3 h gave the dithin 11a, which was converted into the oxo form 26a with mercuric acetate in CHCl₃/glacial acetic acid at room temperature in 2 h. Synthesis of 41 was completed in five steps, starting from dimethyl L-tartrate 36, the hydroxy groups of which were protected by reaction with methoxyethoxymethyl chloride (MEMCl) and then the ester groups of 37 were reduced to alcohols with LiAlH₄ to obtain the diol 38. This was converted into 39 through tosylation of the hydroxy groups with tosyl chloride and then conversion into iodides 40 using potassium iodide. Treatment of 40 with the dianionic salt 5 in dry ethanol at room temperature produced 41 in 90% yield (Scheme 6) [40].

In 2000, syntheses of 5,6-diphenyl[1,3]dithiolato[4,5-b][1,4]dithiin-2-thione 48 and its coupling product 52, which is a fully unsaturated analogue of BEDT-TTF, were achieved. The 1,8-diketone 47 was easily obtained from the reaction of the dianion 5 (1 equiv) and desyl chloride 46 (2 equiv) in dry ethanol at room temperature for 3 h in 90% yield (Scheme 6) [40].

The ring closure reaction of 47 was performed initially using LR, which produced only the thiophene 50, similar to the result obtained by another research group [28]. Next, the reaction was conducted with P₄S₁₀, which gave benzylphenyldithiole 49 and the thiophene 50 in 25 and 30% yields. Considering that the reaction could take place through a radical mechanism, it was repeated in the dark using P₄S₁₀. After 3 h of reflux in toluene, the dithin 48 was successfully obtained in 65% along with a trace of benzylphenyldithiole 49 and the thiophene 50 in 20% yields. The fully unsaturated tetraphenyl analogue 52 of ET was obtained in 90% yield by a coupling reaction of 51, which was obtained by converting the thione group of 48 to its corres-
ponding oxo form in 85% yield, in hot triethyl phosphite, yielding 52 in 90% yield (Scheme 7).

A charge transfer salt 54 of 52 was prepared with the acceptor 2,3-dichloro-5,6-dicyano-p-benzoquinone (DDQ) 53 (1:1) in dichloromethane at room temperature to investigate the optical constant and optical band gap of the complex (Scheme 8) [56]. A solution of the salt was evaporated on a quartz substrate until \( \approx 110 \) nm thickness of the film was obtained. The film was annealed at 25, 55, 85, 115 and finally at 145 °C for 30 min.
Electronic transitions of the complex 54, i.e. n–π and π–π transitions, led to the formation of radical ion pairs 55. The refractive index dispersion and optical constant of the annealed film were examined for each temperature. The absorbance, refractive index, reflectance and transmittance values of the material were found to be between 0.16–0.32, 2.3–2.7, 16–20% and 46–66%, respectively at 400–800 nm wavelength range which clearly indicated that the refractive index, absorbance and reflectance of the complex decreased while transmittance increased with increased annealing temperature.

Our easy synthesis of dithiin-containing compounds led to the production of various BEDT-TTF analogues, comprising monophenylidithiin, diphenylidithiin, diphenylthiophene and diols [40,46,53]. While coupling of 28 with 51 smoothly gave the corresponding ET analogue 56, its reaction with 57 did not produce any result (Scheme 9) [57]. This could be due to the reaction of the benzylphenylidithiolate moiety with triethyl phosphite.

Analogues of ET, having dithiin and thiophene rings along with hydroxy groups were synthesized to provide the possibility of intramolecular hydrogen bonding through the hydroxy groups [57]. The half ET analogue 61 was obtained from the reactions of either the diion 5 or the zinc-complex 59 with 2-bis(bromomethyl)propane-1,3-diol (60, Scheme 10). As the hydroxy groups could lead to side products during the coupling reaction, performed using triethyl phosphite, and the reaction for conversion of the thione group to a keto group with mercury acetate and acetic acid, they were protected by reaction with methoxyethoxymethylchloride (CH₃OCH₂CH₂OCH₂Cl, MEMCl) to obtain 62. The thione group of this compound, was then converted into a keto group to give 63. Its reaction with 26a–c in triethyl phosphite led to the formation of cross-coupled product 64a–c, along with the self coupled one. Following the same procedure, the half ET analogue 65, possessing a fused diphenylthiophene ring was coupled with 63 to produce 66, along with self coupled products like 67. Removal of the MEM groups of both 64 and 66 in dilute HCl/THF mixture resulted in the formation of the target analogues 68a–c and 69, having two hydroxy groups. Coupling of the dithione 51 with 63 gave 70 and its hydrolysis yielded the ET analogue 71, possessing diphenylidithiin and two hydroxy groups. Following the same strategy, an ET analogue 72, having half ET and two hydroxy groups was synthesized to compare the oxidation and reduction potentials of the analogues. The yields of the resultant products are given in Table 4.

### Table 4: Yields of the products 64, 65, 68–71.

| Product | Yield (%) | Diol | Yield (%) |
|---------|-----------|------|-----------|
| 70      | 30        | 71   | 75        |
| 65      | 30        | 69   | 75        |
| 64a     | 27        | 68a  | 43        |
| 64b     | 29        | 68b  | 35        |
| 64c     | 40        | 68c  | 34        |

The oxidation and reduction properties of the diphenyl analogues 56, 69 and 71 and monophenyl analogues 68a–c, 31a and 29a were investigated and compared by cyclic voltammetry (CV) (Table 5) with ET 3 and its fully unsaturated tetraphenyl analogue 52. The CV measurement of the donors was performed in 0.1 M NaClO₄/ACN with a scan rate of 100 mVS⁻¹ at room temperature, using Pt working and Ag/Ag⁺ reference electrodes. The spectroelectrochemical studies were carried out in CH₂Cl₂ containing 0.1 M TBABF₄ at room temperature.

The CV studies indicated that while the fully unsaturated 52 and diphenylthiophenedimethylthio 67 had the highest
Scheme 10: Reaction conditions (i) EtOH, reflux, overnight; (ii) diisopropylethylamine in CH$_2$Cl$_2$, room temperature, overnight; (iii) (AcO)$_2$Hg/AcOH, CHCl$_3$, rt, 3 h; (iv) (EtO)$_3$P, 110 °C, N$_2$, 2 h; (v) sample in THF at 0 °C, add 20% HCl, then rt, overnight.

Table 5: Redox potential of ET$_3$ and its analogues, ACN solution of 0.1 M NaClO$_4$.

| Sample | $E^1_{\text{OX}}$ (V) | $E^2_{\text{OX}}$ (V) | $\Delta E$ (V) |
|--------|-----------------------|-----------------------|----------------|
| 52     | 0.72                  | 1.03                  | 0.31           |
| 70     | 0.44                  | 0.70                  | 0.26           |
| 66     | 0.60                  | 0.84                  | 0.24           |
| 71     | 0.41                  | 0.63                  | 0.22           |
| 69     | 0.57                  | 0.80                  | 0.23           |
| 56     | 0.36                  | 0.59                  | 0.23           |
| 68a    | 0.49                  | 0.74                  | 0.25           |
| 68b    | 0.50                  | 0.76                  | 0.26           |
| 68c    | 0.42                  | 0.70                  | 0.28           |
| 67     | 0.72                  | 1.06                  | 0.34           |
| 31a    | 0.49                  | 0.63                  | 0.14           |
| 72     | 0.42                  | 0.70                  | 0.28           |
| 29a    | 0.66                  | 0.96                  | 0.30           |
| 3ET    | 0.46                  | 0.71                  | 0.25           |

oxidation potentials, diphenyldithiindimethylthio 56 displayed the lowest oxidation potential and combination of dithien and diol groups led to oxidation potentials comparable with ET$_3$.

BEDT-TTF analogues possessing thiophene substituted 1,4-dithien and thiophene rings were reported in 2013 [58]. Their syntheses began with our standard synthesis of a 1,8-diketone 74 having a thiophene in place of a benzenoid aromatic group (Scheme 11). Reaction of the zinc-complex 59 with four mol equivalents of $\alpha$-bromoketone 72 gave the diketone 74 in 80% yield, subsequent ring closure of which with P$_4$S$_{10}$ in acidic and basic conditions produced 1,4-dithien 75 (75%) and thiophene 76 (57%) rings, respectively. They were then converted into their corresponding oxo forms 77 (65%) and 78 (77%), respectively, with mercury acetate and subjected to the coupling reaction with triethyl phosphite to produce the ET analogues having 1,4-dithien rings 79 (80%) and thiophene rings 80 and 81 (75%) as inseparable isomers.
Unfortunately, all attempts to electropolymerize the analogues failed. Computational studies indicated that the α-carbons of the peripheral thiophenes, where the polymerization was expected to take place, did not exhibit enough spin density.

Conclusion
Bis(ethylenedithio)tetrathiafulvalene (BEDT-TTF, ET) is a unique molecule which has been successfully used as an electronic material that challenges the creativity and inventiveness of chemists in areas such as organic chemistry, materials chemistry, supramolecular chemistry and polymer chemistry. 1,8-Diketones have been demonstrated to be versatile starting materials for the synthesis of various challenging analogues of ET, possessing dithiin and thiophene moieties. This chemistry not only led to the production of the target materials, having interesting electronic properties, but also illustrated challenging synthetic heterocyclic chemistry.

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