

into 100 mL of saturated NaCl solution. The white precipitate was filtered off and washed thoroughly with Milli-Q-Water until no chloride could be detected by silver nitrate.

The wet precipitate was redispersed in toluene, dried over sodium sulfate and filtered. The remaining water and alcohols were evaporated and additional 2.5 g of trimethylethoxysilane were added. After 24 h the solvent was evaporated at 40 °C and 64 mbar vacuum and the endcapped microgels were freeze-dried in benzene (yield: 55–80 %).

TEM measurements were performed by a Philips EM420 (120KV) or by a Philips EM300 (80KV) microscope. The samples were prepared from a 0.01 wt.-% solution in cyclohexane or toluene and measured on carbon-coated copper grids.

Light scattering measurements were performed by an ALV goniometer SP 125 equipped with Spectra Physics 2060-4S argon ion laser (514 nm) and ALV-5000 digital correlator.

UV/Vis spectra were measured on a Perkin-Elmer Lambda 17 spectrometer equipped with digital data handling (Perkin Elmer CSS-Software). The spectra were corrected for solvent absorption and  $\mu$ -gel scattering.

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## Covalent Linking of $\beta$ -Slabs of EDT-TTF Moieties: Bis(ethylenedithiotetrathiafulvalenyl)-ethane and Its 1:1 Radical Cation Salt with $\text{Au}(\text{CN})_2^{-**}$

By Marc Fourmigué,\* Cécile Mézière, Enric Canadell, David Zitoun, Klaus Bechgaard, and Pascale Auban-Senzier

Increasing the dimensionality has been one of the main goals in the search for highly conducting metallic and superconducting organic charge transfer salts. Appropriate modification of the strongly one-dimensional (1D) TMTTF<sup>[1]</sup> salts led to TMTSF<sup>[1]</sup> (by introduction of selenium atoms) or BEDT-TTF<sup>[1]</sup> (by introduction of extra sulfur atoms) salts, which were shown to have pseudo-1D or 2D electronic band structures while several of them exhibited superconductivity.<sup>[2,3]</sup> Later, C<sub>60</sub>, with its spherical  $\pi$ -type electronic distribution, gave rise to 3D superconducting salts with higher critical temperatures.<sup>[4]</sup> Looking for a different strategy towards an increase of dimensionality, we were attracted by the possibility, offered by dimeric TTFs, to couple together stacks or slabs of partially oxidized donors along a second or third direction through the bridging group. Depending upon the nature of this bridge, the intramolecular interaction between the two redox moieties, and hence between the stacks or slabs, could be modified in a controlled way. Furthermore, in 1D two-band systems<sup>[5]</sup> with a small degeneracy between the bands, the Fermi level is expected to cut several bands, giving rise to an equal number of Fermi vectors, as observed for example in [TTF][Ni(dmit)<sub>2</sub>]<sub>2</sub>, where dmit is 2-thioxo-1,3-dithiole-4,5-dithiolate.<sup>[5]</sup> In this case, the possible occurrence of a 2k<sub>F</sub> charge density wave (CDW) transition at one Fermi vector should not destroy the whole Fermi surface.

Several dimeric TTFs have been reported, and this topic was recently reviewed.<sup>[6,7]</sup> One of the main problems en-

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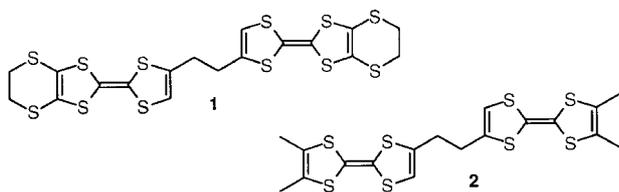
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countered with those large donor molecules is their very limited solubility in the usual solvents. This has been often circumvented by substituting the molecule with alkyl chains<sup>[6,8]</sup> or by using long, flexible spacers,<sup>[9]</sup> at the expense of sizable, delocalized intermolecular interactions. It was also shown that, regardless of the nature of the bridge between two TTF moieties, the intramolecular interaction is expected to remain weak<sup>[10]</sup> since the coefficients of the highest occupied molecular orbital (HOMO) of each individual TTF are small at the bridging carbon atoms, the HOMO of TTF being mainly concentrated on the central C<sub>2</sub>S<sub>4</sub> core. Besides, the nature of the bridge is expected to play a crucial role in the relative orientation of the two TTF moieties with the possibility of different conformations, as exemplified for instance by the different solid state arrangements of TTF–Te–TTF and TTF–Te–Te–TTF.<sup>[11]</sup>

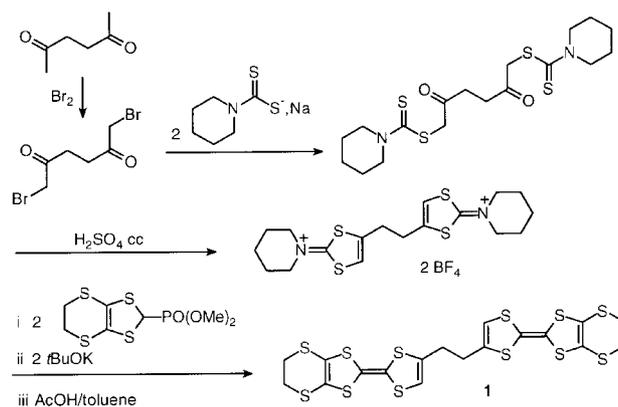
Cation radical salts of such a dimeric molecule incorporating two dimethyltetrafulvalene (Me<sub>2</sub>TTF) moieties linked together through an ethylenic (–CH<sub>2</sub>–CH<sub>2</sub>–) bridge, (Me<sub>2</sub>TTF)<sub>2</sub>C<sub>2</sub>H<sub>4</sub>, **2** (Scheme 1) were recently described.<sup>[12,13]</sup> In its salts with the mono- and dianionic rhenium cluster anions [Re<sub>5</sub>Q<sub>5</sub>Cl<sub>9</sub>]<sup>–</sup> and [Re<sub>6</sub>Q<sub>6</sub>Cl<sub>8</sub>]<sup>2–</sup> (Q = S, Se), molecules were found to stack on top of each other into strongly dimerized or trimerized units, giving rise to 1D structures with semiconducting character.<sup>[13]</sup> Note also that in those salts the ethylenic bridge adopts the most stable, fully staggered (antiperiplanar) conformation, with the two TTF moieties lying in the same plane. It was hoped that similar twin molecules, now incorporating the EDT–TTF<sup>[1]</sup> moiety, would lead to the characteristic  $\alpha$ ,  $\beta$  or  $\kappa$  phases encountered in the BEDT–TTF or EDT–TTF radical cation salts, therefore with a potential for interconnecting those slabs through the covalent ethylenic bridge. This proved to be the case indeed, as will be shown in this paper.



Scheme 1. Chemical structures of **1** and **2**.

The target molecule was prepared following an experimental procedure already used for the synthesis of the Me<sub>2</sub>TTF analog **2** (Scheme 2).<sup>[12,13]</sup>

The twin donor molecule **1** was isolated as needle-like crystals after recrystallization from tetrahydrofuran (THF). Only two reversible oxidation waves are observed by cyclic voltammetry, at 0.45 and 0.85 V, vs saturated calomel electrode (SCE). This result confirms a very weak interaction, if any, between the two EDT–TTF moieties, mainly attributable to the small coefficient of the EDT–TTF core HOMO on the bridging carbon atom, as already discussed above. Electrocrystallization of **1** in 1,1,2-trichloroethane



Scheme 2. Synthesis of **1**.

at 25 °C afforded black elongated parallelepipeds which, from the X-ray crystal structure determination, analyze as (**1**)[Au(CN)<sub>2</sub>]. Because of the dimeric character of the donor, this 1:1 stoichiometry actually implies a partial charge transfer on the organic part, identical to the one observed in the 2:1 salts of TMTSF, BEDT–TTF, and EDT–TTF.

The salt crystallizes in the triclinic system, space group *P*-1.<sup>[14]</sup> One organic donor molecule and one Au(CN)<sub>2</sub><sup>–</sup> anion are located in general position in the unit cell (see Fig. 1). Note that one of the two ethylenic end groups of the EDT–TTF moieties is found disordered on two positions, as commonly observed in EDT–TTF and BEDT–TTF salts. The organic molecule is almost planar, the most significant bond lengths within the TTF core, i.e., the central C<sub>c</sub>=C<sub>c</sub> and the C<sub>c</sub>–S bonds, are comparable in the two EDT–TTF moieties. These values are collected in Table 1 together with data concerning the neutral EDT–TTF molecule.<sup>[15]</sup>

Table 1. Significant bond lengths in (**1**)[Au(CN)<sub>2</sub>].

(EDT–TTF) <sub>2</sub> X	Central C <sub>c</sub> =C <sub>c</sub> bond [Å]	C <sub>c</sub> –S bonds [Å]
Fragment S1–S6 in <b>1</b>	1.341(12)	1.75(1)
Fragment S7–S12 in <b>1</b>	1.357(11)	1.74(1)
EDT–TTF <sup>0</sup>	1.335(4)	1.758(3)

The similarity of the two EDT–TTF moieties in (**1**)[Au(CN)<sub>2</sub>] indicates that the charge is evenly distributed between the two fragments. This is further confirmed by extended Hückel calculations<sup>[16]</sup> on **1** which show that the two highest occupied orbitals, at –8.57 and –8.61 eV, are nearly degenerated. In addition, these calculated energy values also confirm that the HOMOs of the two EDT–TTF moieties are not coupled through the ethylenic bridge. As shown in Figure 1, (**1**)[Au(CN)<sub>2</sub>] has a layered structure exhibiting a segregation between the EDT–TTF moieties in one hand, the ethylenic bridges and the Au(CN)<sub>2</sub><sup>–</sup> anions in the other hand. Thus, each molecule takes part in two adjacent slabs, while each slab is covalently linked to the

two neighboring ones through the ethylenic bridges which cross the anion layers. This structure brings about a covalent interaction between the slabs, across the anion layer, a novel 3D structure in this class of materials.

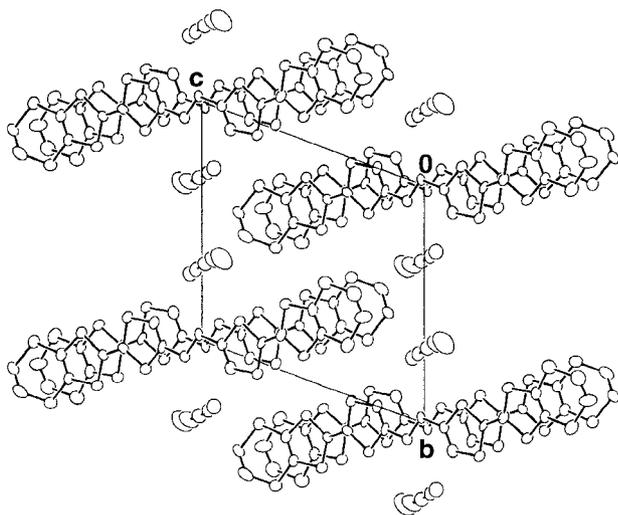


Fig. 1. A view of the unit cell of  $(1)[Au(CN)_2]$ .

Within each layer, the EDT-TTF moieties adopt the so-called  $\beta$ -type structure (Fig. 2). Molecules stack along the  $\langle 100 \rangle$  axis, also parallel to the long axis of the  $Au(CN)_2^-$  anion. There are eight different intermolecular interactions within these EDT-TTF type slabs. Two of them (I–II) are interactions along the stacks parallel to the  $a$ -direction and six are lateral interstack interactions (III–VIII). The associated  $\beta_{HOMO-HOMO}$  interaction energies<sup>[17]</sup> are quite strong for the intrastack interactions (0.439 and 0.334 eV), but considerably weaker for the interstack interactions (between 0.058 and 0.013 eV). Hence, it is expected that  $(1)[Au(CN)_2]$  will exhibit a pseudo-1D HOMO band structure. Since the unit cell contains two molecules, each having two almost uncoupled EDT-TTF HOMO levels, the

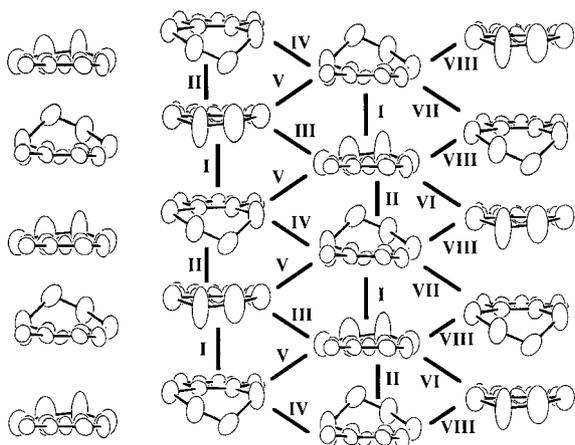
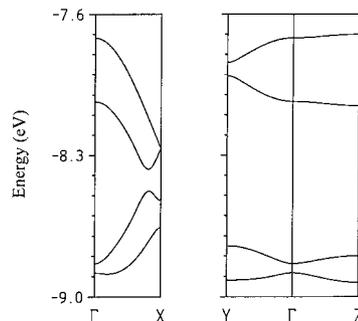
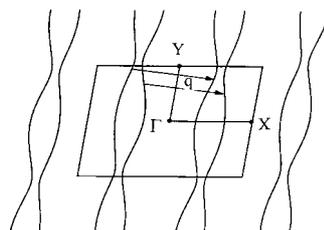


Fig. 2. A side view of one  $\beta$  slab with the corresponding intermolecular interactions (I–VIII).

band structure in the region of the partially filled bands (see Fig. 3) contains four HOMO bands. In agreement with our qualitative analysis, there is virtually no dispersion along  $c^*$ , a moderate although non-negligible dispersion along  $b^*$ , and a strong dispersion along  $a^*$ . Thus, as far as the HOMO band structure is concerned,  $(1)[Au(CN)_2]$  should be seen as a series of uncoupled pseudo-1D EDT-TTF slabs.



(a)



(b)

Fig. 3. a) Calculated dispersion relations for the HOMO bands in  $(1)[Au(CN)_2]$ .  $\Gamma = (0,0,0)$ ,  $X = (a^*/2,0,0)$ ,  $Y = 0,b^*/2,0$ , and  $Z = (0,0,c^*/2)$ . b) Calculated section of the Fermi surface assuming a metallic filling of the bands in (a).

$(1)[Au(CN)_2]$  is a semiconductor exhibiting a high room-temperature conductivity ( $\sigma_{RT} = 12 \text{ Scm}^{-1}$ ) with a very small activation energy (0.06 eV). Because of the 1:1 stoichiometry, there are six electrons to fill the four bands of Figure 3. Since the upper two bands overlap and have quite a sizable dispersion, the salt could have been expected to be metallic, in contradiction to the observed semiconducting behavior. However, we believe that there is a simple reason behind this apparently puzzling observation. As mentioned, with regard to the HOMO band structure,  $(1)[Au(CN)_2]$  can be described as a series of weakly coupled 1D stacks. Since the EDT-TTF moieties of these stacks adopt a zigzag structure, every chain has two EDT-TTF fragments and three electrons to fill the HOMO levels. Thus, from a purely electron counting point of view, every chain has a  $3/4$ -filled dispersive band, so that tetramerization of these chains (i.e., a dimerization of the real zigzag type chains) is expected, thereby opening a gap at the Fermi level and leading to the semi-conducting behavior. This means that the reported crystal structure is really

an average structure (see below for further discussion). However, one must be careful with this kind of qualitative argument because there is some coupling between the chains of the same layer. In order to be correct, the Fermi surface calculated on the basis of this presumably average structure should exhibit nesting vectors with a  $a^*/2$  component. The calculated Fermi surface is shown in Figure 3b and indeed shows that even if two bands are cut by the Fermi level, a single nesting vector ( $q$ ) with the  $a^*/2$  component should allow the simultaneous destruction of all the Fermi surface.<sup>[18]</sup> Why then does our X-ray structure not lead to a unit cell twice longer along the  $a$ -axis? We must remind the reader that every molecule belongs simultaneously to two different slabs. Thus, there is a certain constraint in the possible movements of the two different EDT-TTF moieties of the same molecule so that it is very likely that the tetramerization in the different stacks (or even in different regions of the same stack) have only a short range order. Under such circumstances only the average structure will be observed. X-ray diffuse scattering measurements would be very important in order to confirm the short range tetramerization as the origin of the semiconducting behavior of **(1)**[Au(CN)<sub>2</sub>].

In conclusion, the very specific donor network adopted in this salt with the linear Au(CN)<sub>2</sub><sup>-</sup> anion points out the potential of **1** in leading to a wide variety of solid state arrangements with other anions of different size and shape. Furthermore, the intramolecular interaction between the two EDT-TTF moieties could possibly be enhanced by introducing  $\pi$ -bonding in the bridging group, as in (EDT-TTF)-CH=CH-(EDT-TTF) or (EDT-TTF)-C $\equiv$ C-(EDT-TTF), as already described with TTF itself.<sup>[19]</sup>

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8.897(1),  $b = 12.954(2)$ ,  $c = 13.358(1)$  Å,  $\alpha = 107.01(1)$ ,  $\beta = 106.06(1)$ ,  
 $\gamma = 99.42(3)^\circ$ ;  $V = 1367.7(3)$  Å<sup>3</sup>,  $Z = 2$ ,  $\rho_{\text{calc}} = 2.104$  g cm<sup>-3</sup>,  $\mu =$   
6.33 mm<sup>-1</sup>, crystal size:  $0.26 \times 0.10 \times 0.03$ ; Stoe Imaging Plate Diffrac-  
tometer with Mo K $\alpha$  radiation,  $\lambda = 0.71073$  Å. The structure was solved  
by direct methods using SHELXS-86 and refined with SHELXL-93.  
Refinement (on  $F^2$ ) of 314 variables with anisotropic thermal param-  
eters (except for the disordered carbon atoms of one ethylenic group,  
i.e., C7A, C7B, C8A, C8B) gave  $R(F) = 0.039$  (with  $F^2 > 2\sigma(F^2)$ ) and  
 $wR(F^2) = 0.072$  using 4002 absorption corrected (multi-scan,  $T_{\text{max}} =$   
0.733,  $T_{\text{min}} = 0.621$ ) reflections. H atoms were introduced at calculated  
positions (riding model). Crystallographic data (excluding structure  
factors) have been deposited with the Cambridge Crystallographic Da-  
ta Centre as supplementary publication no. CCDC-102945. Copies of  
the data can be obtained free of charge on application to The Director,  
CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (fax: (+44)1223-  
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## Parallel Patterning of Mesoscopic Ring Arrays Using Nanochannel Glass Replica Masks\*\*

By Douglas H. Pearson,\* Ronald J. Tonucci,  
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There is widespread technological and scientific interest in materials engineered on the mesoscopic scale (feature sizes  $\sim 100$  nm to 1  $\mu\text{m}$ ). The applications of such materials span a variety of disciplines, which includes electronic and optical devices,<sup>[1–8]</sup> filtration<sup>[9]</sup> and biomedical uses.<sup>[10,11]</sup> In addition, such materials provide opportunities to study interesting effects such as persistent currents in superconductors and quantum-confinement effects in semiconductors.

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