© 1983 The Chemical Society of Japan *Bull. Chem. Soc. Jpn.*, **56**, 2894—2898 (1983) [Vol. 56, No. 10

The Crystal Structure of Tetramethyltetraselenafulvalenium Perrhenate, (TMTSF)$(ReO_4)(C_2H_3Cl_3)_{0.25}$

Hayao Kobayashi,* Akiko Kobayashi,† Yukiyoshi Sasaki,† Gunzi Saito,†† and Hiroo Inokuchi††
Department of Chemistry, Faculty of Science, Toho University, Funabashi, Chiba 274
†*Department of Chemistry, Faculty of Science, The University of Tokyo, Hongo, Bunkyo-ku, Tokyo 113*
††*Institute for Molecular Science, Okazaki 444*

(Received February 7, 1983)

The crystal structure of (TMTSF)$(ReO_4)(C_2H_3Cl_3)_{2.25}$ was determined. The crystals are monoclinic P2/c, a=18.573(3), b=7.418(2), c=26.210(6)Å, β=107.85(2)°, Z=8. TMTSF molecules are stacked to form diadic columns along the b axis. The mode of intermolecular overlapping between adjacent TMTSF molecules shows a new mode of overlapping. The strong alternation of the intermolecular Se–Se distances along the TMTSF columns indicates that this compound is a Peierls insulator, which is consistent with the semiconducting behavior. The Peierls instability is accompanied by a bending distortion of the planar TMTSF molecules. The room-temperature conductivity is 3×10^{-4} $(\Omega\ \mathrm{cm})^{-1}$, and the activation energy is 0.18 eV. There are some short Se–O contacts between ReO_4 anions and TMTSF stacks. The solvent molecnles $(C_2H_3Cl_3)$ are located in the channel formed along the b axis.

Since the discovery of the organic superconductors, $(TMTSF)_2X(X{=}ClO_4, PF_6\cdots)$, a number of works have been undertaken to elucidate various aspects of the organic metal systems (TMTSF: tetramethyltetraselenafulvalene). The crystal structure analyses of $(TMTSF)_2X$ compounds have revealed that these compounds have both one-dimensional (1-D) and two-dimensional (2-D) characters.[1] The TMTSF molecules are stacked face-to-face to form columns typically observed in 1-D organic metal systems. The interstack Se–Se contacts indicate the two-dimensionality of the crystals. Unlike the complex 2 : 1 salts of TMTSF, though no crystal-structure determination has been made on the simple 1 : 1 salts. In this paper, we report on the crystal structure of (TMTSF) (ReO_4) $(C_2H_3Cl_3)_{0.25}$.

Experimental

The black needle crystals were grown electrochemically from 1,1,2-trichloroethane by using twice-crystallized Bu_4NReO_4 as the electrolyte. The crystal data are: $(C_{10}H_{12}Se_4)$ (ReO_4) $(C_2H_3Cl_3)_{0.25}$, a=18.573(3), b=7.418(2), c=26.210(6) Å, β=107.85(2)°, V=3489.7 Å^3; monoclinic, space group P2/c, Z=8. The intensities were measured on a Rigaku automated four-circle diffractometer with Mo $K\alpha$ radiation (λ=0.7107 Å). The size of the crystal used was 0.04×0.10×0.30 mm. The structure was solved by the direct method and has been refined to R=0.089, using 2742 independent reflections for which $|F|>3\sigma(|F|)$. The hydrogen atoms were not included in the calculations. The atomic coordinates are given in Table 1.[2]**

TABLE 1. ATOMIC COORDINATES OF (TMTSF) $(ReO_4)(C_2H_3Cl_3)_{0.25}$

Atom	x	y	z
Re(1)	0.2996 (1)	0.4890 (4)	0.0457 (1)
Re(2)	0.1916 (1)	0.0831 (4)	0.2630 (1)
Se(1)	0.0457 (2)	0.1889 (8)	0.0805 (2)
Se(2)	−0.1205 (2)	0.2189 (7)	0.0056 (2)
Se(3)	0.1084 (2)	0.3085 (7)	−0.0234 (2)
Se(4)	−0.0601 (2)	0.3351 (8)	−0.0953 (2)
Se(5)	0.4906 (2)	0.0543 (9)	0.3344 (2)
Se(6)	0.6291 (2)	0.0563 (8)	0.2907 (2)
Se(7)	0.3770 (2)	0.5263 (8)	0.1948 (2)
Se(8)	0.4463 (2)	0.5256 (9)	0.3173 (2)
Cl(1)	0.4212 (17)	0.3103 (71)	0.4484 (15)
Cl(2)	0.4260 (19)	0.7012 (73)	0.4519 (15)
C(1)	−0.0124 (25)	0.2315 (83)	0.0160 (19)
C(2)	0.0016 (23)	0.2789 (77)	−0.0305 (21)
C(3)	−0.0321 (32)	0.1368 (81)	0.1120 (15)
C(4)	−0.1080 (26)	0.1470 (80)	0.0695 (28)
C(5)	0.0921 (33)	0.3603 (79)	−0.0912 (22)
C(6)	0.0129 (26)	0.3678 (77)	−0.1313 (18)
C(7)	−0.0071 (29)	0.0820 (100)	0.1639 (20)
C(8)	−0.1741 (25)	0.0965 (82)	0.0916 (22)
C(9)	0.1564 (32)	0.3941 (102)	−0.1156 (25)
C(10)	−0.0069 (33)	0.4026 (77)	−0.1843 (18)
C(11)	0.5172 (23)	0.0501 (84)	0.2761 (19)
C(12)	0.5897 (30)	0.0155 (82)	0.3846 (25)
C(13)	0.6491 (22)	0.0217 (85)	0.3618 (21)
C(14)	0.5874 (30)	0.0097 (70)	0.4399 (23)
C(15)	0.7267 (26)	0.0015 (91)	0.3962 (26)
C(16)	0.4651 (22)	0.5202 (78)	0.2551 (16)
C(17)	0.3142 (29)	0.5505 (88)	0.2375 (18)
C(18)	0.3393 (26)	0.5620 (60)	0.2841 (19)
C(19)	0.2255 (27)	0.5796 (98)	0.2058 (21)
C(20)	0.2973 (36)	0.5839 (132)	0.3325 (29)
O(1)	0.2538 (20)	0.4967 (74)	0.0917 (13)
O(2)	0.3945 (16)	0.4921 (88)	0.0868 (16)
O(3)	0.2772 (19)	0.3254 (104)	0.0069 (16)
O(4)	0.2808 (22)	0.6591 (89)	0.0046 (17)
O(5)	0.1894 (23)	0.0915 (80)	0.2044 (19)
O(6)	0.1492 (24)	0.2706 (62)	0.2799 (18)
O(7)	0.2745 (36)	0.0774 (134)	0.3122 (21)
O(8)	0.1505 (27)	−0.1015 (72)	0.2784 (19)
C(21)	0.4821 (56)	0.5464 (155)	0.4785 (37)
C(22)	0.4684 (84)	0.4093 (233)	0.4773 (59)

Result and Discussion

The crystal structure is shown in Fig. 1. There are three crystallographically independent TMTSF molecules. One is on the general position, and the others, on the two-fold axis. They will hereafter be named molecules A, B, and B′ respectively (see Fig. 1). The crystal has channels along the b axis. The structure refinements have revealed the existence of solvent molecules in the channels. A similar inclusion of the $C_2H_3Cl_3$ solvent molecules has been observed in (BEDT–TTF)$_2$-$ClO_4(C_2H_3Cl_3)_{0.5}$.[3] The volume of the channel indicates that two solvent molecules can be accommodated in the unit cell. However, the atoms of the solvent molecules are highly disordered and could not be located definitely. Fourier maps gave four large and four small peaks around (1/2, 1/2, 1/2), which were assumed to be the positions of the Cl and C atoms of $C_2H_3Cl_3$.

** A list of the observed and calculated structure amplitudes and a table of the thermal parameters are kept at the Chemical Society of Japan as Document No. 8339.

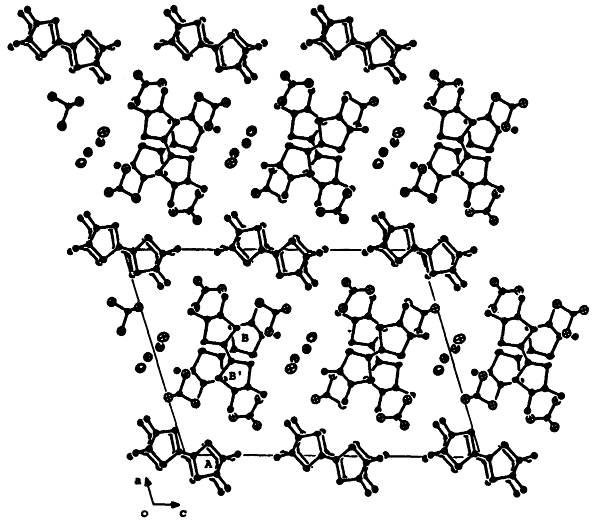


Fig. 1. The b axis projection of the structure of (TMTSF)(ReO_4) $(C_2H_3Cl_3)_{0.25}$.

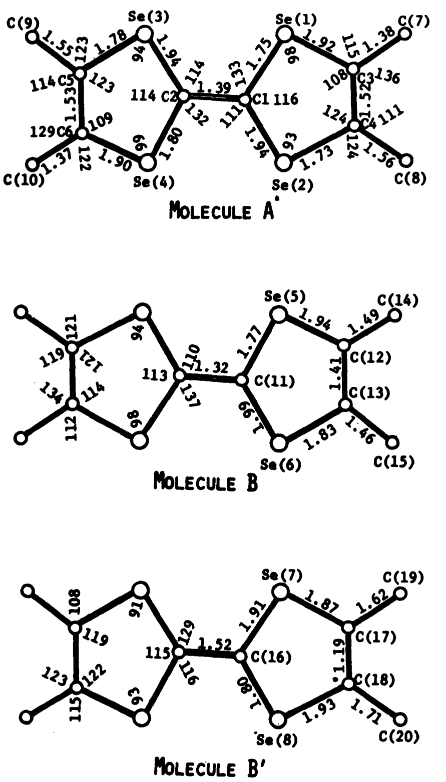


Fig. 2. Bond lengths (Å) and angles (°) of TMTSF. The standard deviations are: 0.06—0.07 Å (Se–C), 0.09—0.10 Å (C–C), 2—3° (C–Se–C), and 5—6° (C–C–Se and C–C–C).

In the least-squares refinements, the atomic populations of Cl and C atoms were taken to be 0.75 and 0.5 respectively.

Unlike $(TMTSF)_2ReO_4$,[4] (TMTSF)$(ReO_4)(C_2H_3Cl_3)_{0.25}$ does not show the orientational disorder of ReO_4. However, the large temperature factors of some oxygen atoms of the ReO_4 anions indicate a large thermal motion. The isotropic thermal parameters (B/Å^2) are: O(1), 5.6; O(2), 6.1; O(3), 6.4; O(4), 6.7; O(5), 7.8; O(6), 6.5; O(7), 10.7; O(8), 7.7; Re(1), 4.6; Re(2), 5.1.[5] It is natural that the ReO_4 anion, with its relatively large thermal parameters (Re(2), O(5), O(6), O(7), and O(8)), shows a loose contact with

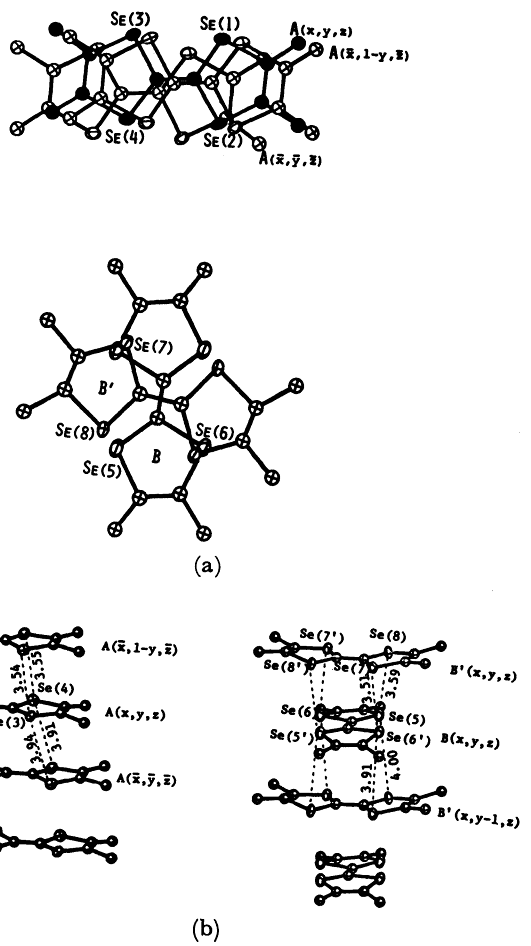


Fig. 3. (a) The modes of intermolecular overlapping. A(x, y, z), B(x, y, z), and B′(x, y, z) indicate three independent TMTSF molecules (see Fig. 1 and Table 1). A(u, v, w) indicates the molecule related to A(x, y, z) by the symmetry operation (u, v, w). (b) The intermolecular short Se–Se distances along the stacks.

Table 2. Bond lengths and angles of ReO_4. the standard deviations are: 0.05—0.10 Å (Re–O) and 2—4° (O–Re–O)

Bond length	l/Å	Bond angle	φ/°
Re(1)···O(1)	1.69	O(1)–Re(1)–O(2)	100
Re(1)···O(2)	1.79	O(1)–Re(1)–O(3)	114
Re(1)···O(3)	1.57	O(1)–Re(1)–O(4)	114
Re(1)···O(4)	1.64	O(2)–Re(1)–O(3)	116
		O(2)–Re(1)–O(4)	112
		O(3)–Re(1)–O(4)	101
Re(2)···O(5)	1.55	O(5)–Re(2)–O(6)	111
Re(2)···O(6)	1.72	O(5)–Re(2)–O(7)	121
Re(2)···O(7)	1.69	O(5)–Re(2)–O(8)	114
Re(2)···O(8)	1.68	O(6)–Re(2)–O(7)	101
		O(6)–Re(2)–O(8)	109
		O(7)–Re(2)–O(9)	100

TABLE 3. THE BEST PLANES OF THE TMTSF MOLECULES

Molecule A

(1) The best plane calculated from the atomic coordinates of the non-hydrogen atoms.
$-2.1187X+7.1702Y+6.7436Z=1.770$ (Å)
(X, Y, and Z are coordinates referred to the direct lattice)
Deviations (Å): Se(1), 0.03; Se(2), 0.09; Se(3), 0.05; Se(4), 0.12; C(1), 0.02; C(2), 0.02; C(3), 0.04; C(5), 0.00; C(6), −0.05; C(7), −0.06; C(8), −0.09; C(9), −0.06; C(10), −0.11.

(2) The best plane calculated from the atomic coordinates of Se(1), Se(2), Se(3), Se(4), C(1), and C(2).
$-1.8954X+7.1483Y+7.0873Z=1.817$
Deviations: Se(1), 0.02; C(1), 0.02; Se(3), 0.02; Se(4), 0.02; C(1), −0.02; C(2), −0.04.

(3) The best plane calculated from the atomic coordinates of Se(1), Se(2), C(3), C(4), C(7), and C(8).
$-2.6425X+7.0702Y+7.9249Z=1.892$
Deviations: Se(1), −0.04; Se(2), 0.02; C(3), 0.05; C(4), −0.02; C(7), 0.01; C(8), −0.02.

(4) The best plane calculated from the atomic coordinates of Se(3), Se(4), C(5), C(6), C(9), and C(10).
$-1.5359X+7.2652Y+5.33982Z=1.978$
Deviations: Se(3), −0.03; Se(4), 0.04; C(5), 0.01; C(6), −0.03; C(9), 0.03, C(10), −0.03.

Molecule B

(1) The plane calculated from the atomic coordinates of Se(5), Se(6), and C(11).
$7.418Y=0.398$
Deviations: Se(5), 0.01; Se(6), 0.02; C(11), −0.03.

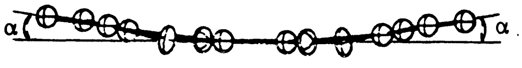
(2) The best plane calculated from the atomic coordinates of Se(5), Se(6), C(12), C(13), C(14), and C(15).
$0.9258X+7.3500Y+2.7742Z=1.783$
Deviations: Se(5), 0.00; Se(6), 0.02; C(12), −0.06; C(13), −0.02; C(14), 0.05; C(15), 0.00.

Molecule B′

(1) The plane calculated from the atomic coordinates of Se(7), Se(8), and C(16).
$7.418Y=3.888$
Deviations: Se(7), 0.02; Se(8), 0.01; C(16), −0.03.

(2) The best plane calculated from the atomic coordinates of Se(7), Se(8), C(18), C(19), and C(20).
$2.6087X+7.3425Y-1.7261Z=4.490$
Deviations: Se(7), 0.02; Se(8), −0.01; C(17), −0.04; C(18), 0.03; C(19), 0.00; C(20), 0.00.

The bending angle α.



Molecule A 2.6, 3.9°
Molecule B 7.8
Molecule B′ 8.2

TABLE 4. INTERMOLECULAR SHORT CONTACTS[a] (l/Å)

$(x, y, z)\cdots(x, y, z)$		$(x, y, z)\cdots(\bar{x}, \bar{y}, \bar{z})$	
Se(5)···Se(8)	3.59 Å	Se(1)···Se(4)	3.55
Se(3)···O(3)	2.99	Se(2)···Se(3)	3.94
Se(7)···O(1)	2.99		
Se(7)···O(2)	3.00	$(x,y,z)\cdots(1-x,y-1,1/2-z)$	
		Se(6)···Se(7)	3.95
$(x, y, z)\cdots(\bar{x}, 1-y, \bar{z})$		Se(5)···Se(8)	4.00
Se(1)···Se(4)	3.55		
Se(2)···Se(3)	3.54	$(x,y, z)\cdots(1-x,y, 1/2-z)$	
Se(2)···O(4)	3.04	Se(6)···Se(7)	3.51
O(4)···C(8)	3.27	Se(6)···O(5)	3.34
$(x,y, z)\cdots(x, 1-y,z-1/2)$		$(x, y, z)\cdots(\bar{x},y, 1/2-z)$	
C(9)···C(20)	3.31	Se(6)···O(5)	3.34

a) The standard deviations are: 0.008 Å (Se···Se), 0.07 (Se···O), and 0.10 (C···O).

surrounding molecules: the (Se(6)···O(5)) contact is the only contact which is slightly shorter than the van der Waals distance (Table 4).

The bond lengths and angles are given in Fig. 2 and Table 2. As a result of the relatively small number of reflections, the standard deviations are fairly large. The least-squares planes of TMTSF molecules are listed in Table 3.

The TMTSF molecules are stacked face-to-face to form diadic columns. The modes of intermolecular overlapping are shown in Fig. 3. The $A(x, y, z)$, $A(\bar{x}, \bar{y}, \bar{z})$, and $A(\bar{x}, 1-y, \bar{z})$ molecules are stacked in a manner similar to that found in usual organic conductors, where $A(u, v, w)$ indicates the molecule related to the $A(=A(x, y, z))$ molecule, by the symmetry operation (u, v, w). The mode of overlapping between $A(x, y, z)$ and $A(\bar{x}, \bar{y}, \bar{z})$ is similar to that observed in $(\mathrm{TMTSF})_2\mathrm{X}$.1) However, the mode between $A(x, y, z)$ and $A(\bar{x}, 1-y, \bar{z})$ is that of a slightly modified direct overlap. The B and B′ molecules have C_2 symmetry, and the mode of intermolecular overlapping is of a new type. The longest axis of the B molecule is approximately perpendicular to that of the B′ molecule. This new overlapping mode appears to be natural, because this structure indicates a large intermolecular Se–Se interaction and a small intermolecular C–C repulsion. Since the symmetry of the highest occupied molecular orbital(HOMO) of TMTSF with ideal D_{2h} symmetry is b_{1u} (the coefficients of the atomic orbitals of the four Se atoms are identical), the magnitude of the intermolecular overlap integral of HOMO for this configulation will be almost equal to that for the direct overlapping. The short intermolecular Se–Se distances are given in Table 4 and Fig. 3. The average Se–Se distance between the $A(x, y, z)$ and $A(\bar{x}, 1-y, \bar{z})$ molecules is 3.55 Å, but that between $A(x, y, z)$ and $A(\bar{x}, \bar{y}, \bar{z})$ is 3.93 Å. Similarly, the Se–Se separation between $B(x, y, z)$ and $B'(x, y, z)$ is 3.55 Å, and that between $B(x, y, z)$ and $B'(x, y-1, z)$ is 3.96 Å. Thus, the positions of the Se atoms are shifted alternately along the b axis from the positions of the regular arrangement, which makes the molecules nonplanar (see Fig. 3 and Table 3). The shortest Se–Se distance of 3.51 Å is much shorter than the van der Waals distance (4.0 Å). In $(TMTSF)_2X$, the Se–Se distance in the TMTSF stack is longer than 3.85 Å.[1,3] Unlike the case in $(TMTSF)_2ReO_4$, the TMTSF columns in (TMTSF) $(ReO_4)(C_2H_3Cl_3)_{0.25}$ have no short Se–Se contact between adjacent columns (see Table 4). The only significantly short contact between TMTSF column can be seen between methyl carbons (C(9)–C(20), 3.31 Å). Thus, the TMTSF columns can be regarded as 1-D systems.

The temperature depence of the electrical conductivity shows that this compound is a semiconductor with the room-temperature conductivity of 3×10^{-4} $(\Omega\ \mathrm{cm})^{-1}$ and the activation energy of 0.18 eV (see Fig. 4).[6] Contrary to the semiconductive behavior of (TMTSF)-$(ReO_4)(C_2H_3Cl_3)_{0.25}$, $(TMTSF)_2ReO_4$ is metallic above 180 K(T_C), where a metal-insulator transition occurs.[7] The superconductivity appears above 9.5 kbar at 1.3 K.[8]

Since the stoichiometry of TMTSF to ReO_4 is 1 : 1, the diadic column of TMTSF indicates that the 1-D band formed along the TMTSF column is filled. Thus, the semiconductive nature of this salt is quite reasonable. The strong alternation of the intermolecular Se–Se distance is consistent with the Peierls instability of the 1-D metallic system. The intermolecular repulsion between the adjacent molecules in the same stack will tend to make the intermolecular spacings regular. On the other hand, the $2k_F$-(Peierls) instability of the 1-D chain tends to produce a diadic distortion in order to to gain the electronic stabilization energy. Since the 1-D band is considered to be made up of atomic orbitals of the Se atoms,[9] the periodical displacements of the other atoms do not contribute to the stabilization of the 1-D electronic system. Therefore, as a result of the Peierls instability, the intermolecular repulsion will make TMTSF molecules nonplanar. Besides the intra-stack interaction, the interaction between TMTSF molecules and ReO_4 anions may also be important. Since only a few short contacts can be seen between oxygen atoms and methyl carbons (Table 4), the steric repulsion between methyl carbons and ReO_4 is not important. However, significantly short contacts can be seen between the Se atoms of TMTSF and the O atoms of ReO_4. Consequently, some interaction must exist between these atoms. Similar short Se–O (or F) contacts have been observed in $(TMTSF)_2X$.[1]

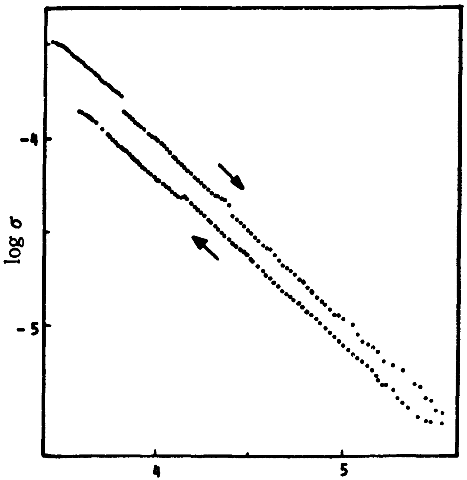


Fig. 4. The electrical conductivity of (TMTSF) (ReO_4) $(C_2H_3Cl_3)_{0.25}$.

A recent X-ray study by Moret, Pouget, Comes, and Bechgaard[10] has revealed that TMTSF columns in $(TMTSF)_2ReO_4$ are strongly modulated by the $2k_F$-wave below $T_c(=180\ \mathrm{K})$, where $(TMTSF)_2ReO_4$ is an insulator. They suggested that the correlation between the TMTSF molecules and the anion displacements can be analyzed as a cooperative phenomenon. The activation energy of about 0.20 eV in $(TMTSF)_2ReO_4$ is approximately equal to that of (TMTSF)$(ReO_4)(C_2H_3Cl_3)_{0.25}$ (0.18 eV).

Since the elctron-phonon coupling in the commensurate system is considered to be strong, the large distortion must be related to that period of the $2k_F$-wave in (TMTSF)$(ReO_4)(C_2H_3Cl_3)_{0.25}$, which is strongly commensurate with the lattice spacing.[11] The structure of this compound shows that the $2k_F$-wave is accompanied by molecular deformation. The bending deformation of the planar molecule has also been found in the partially oxidized platinum compound, $Rb_{1.67}[Pt(C_2O_4)_2]\cdot1.5H_2O$ (RbDOX), which has six-fold commensurate $2k_F$-structure.[14]

The average intermolecular Se–Se distance in the TMTSF stack is much smaller than that of $(TMTSF)_2X$ (≈3.9 Å). Therefore, if the TMTSF stack of (TMTSF) $(ReO_4)(C_2H_3Cl_3)_{0.25}$ were regular, it would be a 1-D metal with a sufficiently large band width. However, the interstack interaction is not large enough to prevent the Peierls instability.

References

1) N. Thorup, G. Rindorf, H. Soling, and K. Bechgaard, *Acta Crystallogr., Sect. B*, **37**, 1236 (1981); F. Wudl, *J. Am. Chem. Soc.*, **103**, 7064 (1981); J. M. Williams, M. A. Beno, E. H. Appelman, and J. M. Capriott, *Mol. Cryst. Liq. Cryst.*, **79**, 319 (1982); H. Kobayashi, A. Kobayashi, G. Saito, and H. Inokuchi, *Chem. Lett.*, **1982**, 245; K. Bechgaard, K. Carneiro, F. B. Rasmussen, M. Olsen, G. Rindorf, C. S. Jacobsen, H. J. Pedersen, and J. C. Scott, *J. Am. Chem. Soc.*, **103**, 2440 (1981); J. M. Williams, M. A. Beno, J. C. Sullivan, L. M. Banovetz, J. M. Braam, G. S. Blackman, C. D. Carlson, D. L. Geer, and D. M. Loesing, *J. Am. Chem. Soc.*, to be published.

2) Adopting anisotropic temperature factors for the Se and Cl atoms and isotropic ones for C and O atoms, the block-

diagonal least-squares refinements gave the R value of 0.093. The subsequent anisotropic refinements of all the non-hydrogen atoms produced only a little improvement (R=0.089), but the temperature factor of C(3) became singular. Therefore, the isotropic temperature factors are adopted for C and O atoms (Table 1).

3) H. Kobayashi, A. Kobayashi, Y. Sasaki, G. Saito, and H. Inokuchi, *J. Am. Chem. Soc.* **105**, 297 (1983).

4) N. Thrup, G. Rindorf, and H. Soling, private communication; H. Kobayashi, unpublished paper.

5) Equivalent isotropic temperature factors as defined by W. C. Hamilton (*Acta Crystallogr.*, **12**, 609 (1959)).

6) The d.c. conductivity was measured along the b axis by using the four-probe method. Gold wires 0.025 mm in diameter were bonded to the specimen with silver paint.

7) C. S. Jacobsen, H. J. Pedersen, K. Mortensen, G. Rindorf, N. Thorup, J. B. Torrance, and K. Bechgaard, *J. Phys. C.*, **15**, 2651 (1982).

8) S. S. Parkin, D. Jerome, and K. Bechgaard, *Mol. Cryst. Liq. Cryst.*, **79**, 213 (1982).

9) T. Mori, A. Kobayashi, Y. Sasaki, and H. Kobayashi, *Chem. Lett.*, **1982**, 1923.

10) R. Moret, J. P. Pouget, R. Comes, and K. Bechgaard, *Phys. Rev. Lett.*, **49**, 1008 (1982).

11) The amplitude of the $2k_F$-wave is considered to be small. However, it has not been determined definitely in any organic conductors. In partially oxidized platinum complexes, the amplitudes of the $2k_F$-wave have been estimated as: 0.025 Å $K_2[Pt(CN)_4]\cdot Br_{0.3}\cdot 3H_2O$ (KCP), 0.025 Å;[12] $K_{1.81}[Pt(C_2O_4)_2]\cdot 1.5H_2O$ (γ-KDOX), 0.17 Å;[13] $Rb_{1.67}[Pt(C_2O_4)_2]\cdot 1.5H_2O$ (RbDOX), 0.17 Å[14]

12) J. M. Lynn, M. Iizumi, and G. Shirane, *Phys. Rev. B*, **12**, 1154 (1975).

13) H. Kobayashi, I. Shirotani, A. Kobayashi, and Y. Sasaki, *Solid State Commun.*, **23**, 409 (1977); A. Kobayashi, Y. Sasaki, I. Shirotani, and H. Kobayashi, *ibid.*, **26**, 653 (1978).

14) A. Kobayashi, Y. Sasaki, and H. Kobayashi, *Bull. Chem. Soc. Jpn.*, **52**, 3682 (1979).