

The Structures of Di(2,3,6,7-tetramethyl-1,4,5,8-tetraselenafulvalenium)* Perrhenate, (TMTSF)₂ReO₄, and Perchlorate, (TMTSF)₂ClO₄

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Abstract

The structure of $2C_{10}H_{12}Se_4^{1/2+} \cdot X^-$ with $X = ReO_4$ has been determined at 295 and at 120 K and with $X = ClO_4$ at 295 K. These compounds belong to a series of mixed-valence cation-radical salts (TMTSF)₂X, and are isostructural with (TMTSF)₂PF₆ (space group $P\bar{1}$). $X = ReO_4$ at 295 K: $a = 7.284$ (3), $b = 7.751$ (1), $c = 13.483$ (1) Å, $\alpha = 83.23$ (1), $\beta = 86.56$ (2), $\gamma = 70.08$ (2)°, $Z = 1$, $V = 710.5$ Å³, $D_x = 2.679$, D_m (floatation in CH_2Cl_2/CH_2Br_2) = 2.77 Mg m⁻³, $\mu(Mo K\alpha) = 14.107$ mm⁻¹. Least-squares refinement of the structure gave $R = 0.045$ and $R_w = 0.052$ for 1883 reflections. $X = ReO_4$ at 120 K: $a = 7.154$ (3), $b = 7.710$ (3), $c = 13.310$ (9) Å, $\alpha = 83.88$ (5), $\beta = 88.41$ (5), $\gamma = 69.12$ (4)°, $V = 681.9$ Å³. $R = 0.044$ and $R_w = 0.047$ for 694 reflections. $X = ClO_4$: $a = 7.266$ (1), $b = 7.678$ (1), $c = 13.275$ (2) Å, $\alpha = 84.58$ (1), $\beta = 86.73$ (1), $\gamma = 70.43$ (1)°, $V = 694.4$ Å³, $D_x = 2.380$, $D_m = 2.39$ Mg m⁻³, $\mu(Mo K\alpha) = 10.363$ mm⁻¹. $R = 0.055$ and $R_w = 0.069$ for 2285 reflections. At 295 K the ReO_4^- and ClO_4^- ions are positioned at sites of inversion symmetry, and, therefore, have two different, but symmetrically equivalent, orientations which are randomly occupied. At 120 K a doubling of all three axes indicates an ordering of the ReO_4^- anions with alternating orientations along the axial directions. Only the average structure is reported in this paper.

Introduction

(TMTSF)₂ReO₄ and (TMTSF)₂ClO₄ belong to a series of isostructural, organic mixed-valence cation-radical salts (TMTSF)₂X, $X = ReO_4^-, ClO_4^-, BF_4^-, PF_6^-, AsF_6^-$ etc. (Bechgaard, Jacobsen, Mortensen, Pedersen & Thorup, 1980), the physical properties of which have attracted much attention. (TMTSF)₂ClO₄ becomes superconducting at about 1 K and ambient pressure

* Recommended IUPAC name: di(4,4',5,5'-tetramethyl- $\Delta^{2,2'}$ -bi-1,3-diselenolyliidene)ium.

(Bechgaard, Caneiro, Rasmussen, Olsen, Rindorf, Jacobsen, Pedersen & Scott, 1981). The other salts of this series also exhibit superconductivity around 1 K, but only under a hydrostatic pressure of the order of 1 GPa. (TMTSF)₂ReO₄ also differs from the perchlorate by having a sharp metal-to-insulator transition at 182 K (Jacobsen, Pedersen, Mortensen, Rindorf, Thorup, Torrance & Bechgaard, 1982). These differences in the physical properties between (TMTSF)₂ReO₄ and (TMTSF)₂ClO₄ make it very important to compare carefully the two structures. It is also of interest to investigate the structure of (TMTSF)₂ReO₄ at a temperature below the transition temperature, to find out whether the transition also involves any structural change.

Experimental

(TMTSF)₂X with $X = ReO_4^-$ and ClO_4^- , prepared as described earlier (Bechgaard *et al.*, 1981), were supplied by Dr K. Bechgaard. Unit-cell dimensions were determined (25 centered reflections) and intensity measurements obtained on an Enraf-Nonius CAD-4 diffractometer: graphite-monochromatized Mo $K\alpha$ radiation ($\lambda = 0.71073$ Å); ω -scan technique ($\Delta = 1.00^\circ + 0.35^\circ \tan \theta$) (further details are deposited); space group $P\bar{1}$ rather than $P1$ from intensity statistics and structure refinements; correction for Lorentz, polarization, and absorption effects; correction for the intensity decrease of control reflections by fitting an exponential-type function to the intensity *vs* time observations.

The structures were solved by direct methods and refined by full-matrix minimization of $\sum w(|F_o| - |F_c|)^2$ using the program system *SHELX* (Sheldrick, 1976). Neutral-atom scattering factors and anomalous-dispersion corrections for Se and Re were taken from *International Tables for X-ray Crystallography* (1974); for other atoms the values contained in *SHELX* (Sheldrick, 1976) were used. Difference electron density maps did not reveal satisfactory positions for the methyl H atoms for any of the structures; this is probably due to the high Se content. Therefore, H

Table 1. Fractional coordinates ($\times 10^4$) and equivalent isotropic thermal parameters ($\times 10^3$) for (TMTSF)₂ReO₄ at 295 K
$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j.$$

	x	y	z	U_{eq} (\AA^2)
Se(1)	2988 (2)	3392 (1)	6142 (1)	39 (1)
Se(2)	1640 (2)	7466 (1)	5108 (1)	39 (1)
Se(11)	3711 (2)	1765 (1)	3874 (1)	38 (1)
Se(12)	2276 (2)	5846 (1)	2826 (1)	38 (1)
C(1)	2264 (14)	5347 (14)	6967 (7)	38 (3)
C(2)	1667 (15)	7058 (14)	6531 (7)	35 (3)
C(3)	2502 (15)	4961 (12)	4927 (7)	32 (2)
C(4)	1002 (17)	8844 (14)	7011 (8)	46 (3)
C(5)	2394 (17)	4750 (16)	8060 (8)	48 (3)
C(11)	3693 (16)	2162 (14)	2454 (8)	42 (3)
C(12)	3102 (15)	3871 (15)	2012 (7)	38 (3)
C(13)	2782 (14)	4269 (12)	4048 (7)	31 (2)
C(14)	3015 (18)	4405 (16)	915 (8)	56 (3)
C(15)	4413 (18)	411 (15)	1940 (9)	54 (3)
Re	0	0	0	58 (1)
O(1)	-338 (28)	-1785 (25)	-488 (15)	92 (3)
O(2)	1309 (32)	-1049 (30)	1001 (19)	124 (3)
O(3)	711 (31)	1190 (29)	-869 (20)	126 (3)
O(4)	-2215 (31)	1327 (30)	304 (19)	132 (3)

Table 2. Fractional coordinates ($\times 10^4$) and isotropic thermal parameters ($\times 10^3$) for (TMTSF)₂ReO₄ at 120 KFor Se and Re atoms $U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j.$

	x	y	z	U_{eq} or U (\AA^2)
Se(1)	3084 (4)	3378 (3)	6151 (2)	17 (1)
Se(2)	1579 (4)	7495 (3)	5110 (2)	17 (1)
Se(11)	3733 (4)	1746 (3)	3863 (2)	18 (1)
Se(12)	2145 (4)	5871 (4)	2802 (2)	18 (1)
C(1)	2322 (34)	5370 (29)	6998 (21)	15 (6)
C(2)	1709 (34)	7084 (30)	6553 (20)	12 (5)
C(3)	2475 (35)	4982 (32)	4938 (23)	16 (6)
C(4)	1104 (36)	8851 (32)	7033 (22)	19 (6)
C(5)	2543 (37)	4720 (33)	8045 (24)	24 (6)
C(11)	3622 (36)	2221 (34)	2431 (23)	25 (6)
C(12)	2958 (37)	3924 (34)	1980 (24)	23 (6)
C(13)	2727 (37)	4272 (34)	4090 (24)	25 (6)
C(14)	2859 (38)	4371 (35)	883 (24)	26 (6)
C(15)	4423 (37)	340 (33)	1880 (23)	25 (6)
Re	0	0	0	23 (1)
O(1)	-80 (56)	-1816 (51)	-695 (38)	50 (10)
O(2)	1246 (46)	-1118 (43)	1054 (28)	25 (8)
O(3)	984 (55)	1335 (53)	-730 (37)	54 (10)
O(4)	-2465 (51)	1397 (47)	289 (35)	42 (9)

atoms have been omitted in the present studies. The condition for absorption corrections and the final R values are deposited. Final positional and thermal parameters are given in Tables 1, 2 and 3.*

* Lists of experimental and computational conditions, structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36955 (29 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3. Fractional coordinates ($\times 10^4$) and equivalent isotropic thermal parameters ($\times 10^3$) for (TMTSF)₂ClO₄ at 295 K
$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j.$$

	x	y	z	U_{eq} (\AA^2)
Se(1)	2973 (1)	3426 (1)	6150 (1)	35 (1)
Se(2)	1639 (1)	7496 (1)	5110 (1)	34 (1)
Se(11)	3730 (1)	1707 (1)	3840 (1)	33 (1)
Se(12)	2295 (1)	5784 (1)	2792 (1)	33 (1)
C(1)	2225 (11)	5430 (10)	6997 (6)	31 (2)
C(2)	1653 (11)	7164 (11)	6529 (7)	34 (2)
C(3)	2527 (12)	4942 (10)	4944 (7)	36 (2)
C(4)	1003 (13)	8928 (11)	7033 (8)	44 (2)
C(5)	2338 (14)	4876 (12)	8087 (7)	46 (2)
C(11)	3710 (12)	2036 (11)	2420 (7)	35 (2)
C(12)	3118 (12)	3785 (11)	1957 (6)	34 (2)
C(13)	2814 (11)	4262 (10)	3986 (7)	32 (2)
C(14)	2986 (15)	4271 (13)	841 (7)	48 (2)
C(15)	4448 (14)	280 (12)	1878 (7)	47 (2)
Cl	0	0	0	80 (2)
O(1)	-397 (23)	-1426 (21)	-457 (13)	89 (2)
O(2)	1018 (30)	-876 (29)	907 (20)	173 (3)
O(3)	687 (31)	1054 (28)	-640 (24)	186 (3)
O(4)	-1950 (24)	1134 (22)	322 (16)	109 (3)

Subsequent structure-analysis calculations were performed with the XRAY system (Stewart, Kruger, Ammon, Dickinson & Hall, 1972). ORTEP (Johnson, 1965) was used to produce the illustrations.

Description of the structure and discussion

Geometry of the TMTSF molecule

In Fig. 1 is shown the numbering of the atoms. Bond lengths and angles are given in Tables 4 and 5. The

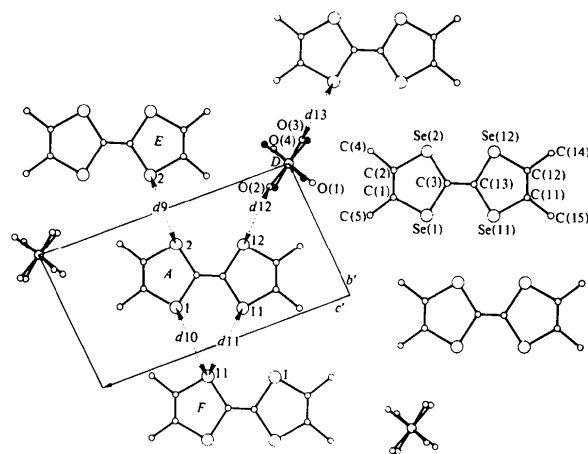


Fig. 1. TMTSF molecule with atom numbering corresponding to Tables 1–5. The view is along a showing numbering corresponding to Table 6 for shorter interstack distances (Se–Se and Se–O). b' and c' are projections of b and c respectively. The symmetry operations are: $A(x, y, z)$, D ('white' atoms: $x, 1 + y, z$), $E(-x, 2 - y, 1 - z)$ and $F(1 - x, -y, 1 - z)$.

Table 4. Bond lengths (Å) in the TMTSF molecule

	(TMTSF) ₂ ReO ₄ 295 K	(TMTSF) ₂ ReO ₄ 120 K	(TMTSF) ₂ ClO ₄ 295 K
C(3)—C(13)	1.334 (14)	1.29 (4)	1.399 (13)
C(3)—Se(2)	1.867 (9)	1.85 (2)	1.878 (7)
C(3)—Se(1)	1.896 (9)	1.89 (3)	1.865 (8)
C(13)—Se(11)	1.864 (9)	1.88 (3)	1.873 (7)
C(13)—Se(12)	1.907 (9)	1.96 (3)	1.856 (8)
C(2)—Se(2)	1.908 (9)	1.91 (3)	1.877 (9)
C(1)—Se(1)	1.896 (10)	1.91 (3)	1.904 (8)
C(11)—Se(11)	1.903 (11)	1.90 (3)	1.878 (9)
C(12)—Se(12)	1.897 (11)	1.87 (3)	1.890 (8)
C(1)—C(2)	1.322 (14)	1.31 (3)	1.354 (11)
C(11)—C(12)	1.323 (14)	1.31 (3)	1.361 (11)
C(2)—C(4)	1.512 (15)	1.48 (4)	1.487 (12)
C(1)—C(5)	1.490 (14)	1.42 (4)	1.468 (12)
C(11)—C(15)	1.513 (16)	1.61 (4)	1.509 (12)
C(12)—C(14)	1.487 (14)	1.46 (4)	1.494 (12)

Table 5. Bond angles (°) in the TMTSF molecule

	(TMTSF) ₂ ReO ₄ 295 K	(TMTSF) ₂ ReO ₄ 120 K	(TMTSF) ₂ ClO ₄ 295 K
Se(2)—C(3)—C(13)	125.5 (7)	126 (2)	121.9 (6)
Se(1)—C(3)—C(13)	121.1 (7)	119 (2)	123.6 (5)
Se(11)—C(13)—C(3)	125.2 (7)	128 (2)	121.1 (6)
Se(12)—C(13)—C(3)	121.1 (7)	121 (2)	123.2 (5)
Se(1)—C(3)—Se(2)	113.4 (5)	114 (2)	114.6 (5)
Se(11)—C(13)—Se(12)	113.7 (5)	111 (2)	115.8 (5)
C(2)—Se(2)—C(3)	94.5 (4)	94 (1)	94.0 (4)
C(1)—Se(1)—C(3)	94.8 (4)	94 (1)	94.7 (3)
C(11)—Se(11)—C(13)	94.4 (4)	95 (1)	93.2 (4)
C(12)—Se(12)—C(13)	94.2 (4)	96 (1)	94.0 (3)
C(1)—C(2)—Se(2)	119.2 (8)	120 (2)	119.9 (6)
C(2)—C(1)—Se(1)	118.1 (7)	118 (2)	116.8 (6)
C(12)—C(11)—Se(11)	119.3 (8)	122 (2)	119.4 (6)
C(11)—C(12)—Se(12)	118.4 (8)	117 (2)	117.6 (6)
C(4)—C(2)—Se(2)	112.2 (7)	112 (2)	113.9 (6)
C(5)—C(1)—Se(1)	114.7 (7)	113 (2)	114.8 (6)
C(15)—C(11)—Se(11)	114.3 (7)	113 (2)	115.6 (6)
C(14)—C(12)—Se(12)	115.9 (8)	119 (2)	116.6 (6)
C(1)—C(2)—C(4)	128.6 (9)	128 (3)	126.2 (8)
C(2)—C(1)—C(5)	127.1 (10)	130 (2)	128.4 (8)
C(12)—C(11)—C(15)	126.4 (10)	126 (3)	125.0 (8)
C(11)—C(12)—C(14)	125.7 (10)	124 (3)	125.7 (8)

values are all in good agreement with those found in the charge-transfer salts (1:1 compounds) with DMTCNQ, 2,5-dimethyl-7,7,8,8-tetracyano-*p*-quinoximethane (Andersen, Bechgaard, Jacobsen, Rindorf, Soling & Thorup, 1978), and in (TMTSF)₂PF₆ (Thorup, Rindorf, Soling & Bechgaard, 1981). There is no statistically significant difference in bond lengths or bond angles between TMTSF at 295 K and at 120 K in (TMTSF)₂ReO₄. The formal double bonds in (TMTSF)₂ClO₄ corresponding to C(3)—C(13), C(1)—C(2), and C(11)—C(12) are slightly longer than those in (TMTSF)₂ReO₄. The TMTSF molecules deviate from the idealized *mmm* symmetry and planarity as described for the TMTSF molecule in (TMTSF)₂PF₆ (Thorup *et al.*, 1981).

Geometry of ReO₄⁻ and ClO₄⁻ anions

Bond lengths and angles are given in Table 6. The shape of the anions is a distorted tetrahedron. The

Table 6. Bond lengths (Å) and angles (°) in the ReO₄⁻ and ClO₄⁻ anions

	ReO ₄ ⁻ , 295 K	ReO ₄ ⁻ , 120 K	ClO ₄ ⁻ , 295 K
O(1)—Re/Cl	1.70 (2)	1.78 (5)	1.42 (2)
O(2)—Re/Cl	1.66 (2)	1.66 (3)	1.44 (2)
O(3)—Re/Cl	1.58 (2)	1.66 (5)	1.31 (3)
O(4)—Re/Cl	1.65 (2)	1.76 (3)	1.46 (2)
O(1)—Re/Cl—O(2)	102 (1)	104 (2)	106 (1)
O(1)—Re/Cl—O(3)	108 (1)	108 (2)	113 (1)
O(1)—Re/Cl—O(4)	105 (1)	109 (2)	102 (1)
O(2)—Re/Cl—O(3)	124 (1)	117 (2)	121 (2)
O(2)—Re/Cl—O(4)	112 (1)	110 (2)	106 (1)
O(3)—Re/Cl—O(4)	105 (1)	108 (2)	107 (1)
Se(12)—O(2)—Re/Cl	150 (1)	152 (2)	153 (2)
Se(12)—O(3)—Re/Cl	125 (1)	115 (2)	123 (1)

non-centrosymmetric anions are positioned at sites of inversion symmetry. It follows that the anions have two different, but symmetrically equivalent, orientations. Experimentally it is found that anions occupy the two orientations randomly at room temperature, *i.e.* they are statistically or dynamically disordered.

Packing

The TMTSF molecules are stacked along the short *a* axis in the same type of 'zigzag' stacking and with the same overlap as in (TMTSF)₂PF₆ (Thorup *et al.*, 1981) and with the stacks arranged in sheets parallel to the *ab* plane. In Fig. 2 is shown a side view of the stacks. The two crystallographically independent distances between neighboring molecular planes are equal in all three structures within experimental error (Table 7, *d*1 and *d*2). The shorter Se—Se distances within a stack (*d*3 to *d*8) are very nearly equal in (TMTSF)₂ReO₄ at 295 K and in (TMTSF)₂ClO₄, while they are shortened between 1.2 and 1.8% in (TMTSF)₂ReO₄ at 120 K. Fig. 1 shows a projection of the structure in the TMTSF molecular plane. Two of the shorter inter-chain Se—Se distances (*d*9 and *d*10) are significantly longer in (TMTSF)₂ReO₄ at 295 K than in (TMTSF)₂ClO₄ [3.827 (2) and 3.902 (2) against

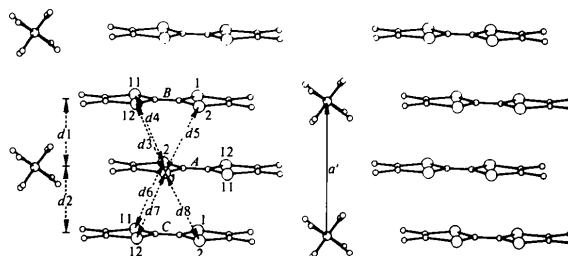


Fig. 2. Side view of stacks (tilted 10°) showing numbering corresponding to Table 6 for shorter distances (Se—Se) within a stack. *a'* is the projection of *a*. The symmetry operations are: *A*(*x*, *y*, *z*), *B*(1 - *x*, 1 - *y*, 1 - *z*) and *C*(-*x*, 1 - *y*, 1 - *z*).

Table 7. Intermolecular distances (Å)

For explanation of the symbols *d*1 to *d*13 see Figs. 1 and 2.

	(TMTSF) ₂ ReO ₄ 295 K	(TMTSF) ₂ ReO ₄ 120 K	(TMTSF) ₂ ClO ₄ 295 K
<i>d</i> 1	3.64 (2)	3.56 (4)	3.64 (2)
<i>d</i> 2	3.64 (2)	3.59 (4)	3.63 (2)
<i>d</i> 3	4.043 (2)	3.971 (5)	4.034 (2)
<i>d</i> 4	3.979 (2)	3.909 (5)	3.964 (2)
<i>d</i> 5	4.036 (2)	3.988 (5)	4.031 (2)
<i>d</i> 6	3.924 (2)	3.867 (5)	3.934 (2)
<i>d</i> 7	3.873 (2)	3.822 (4)	3.871 (2)
<i>d</i> 8	4.138 (2)	4.068 (5)	4.121 (2)
<i>d</i> 9	3.827 (2)	3.699 (4)	3.778 (1)
<i>d</i> 10	3.902 (2)	3.794 (4)	3.865 (1)
<i>d</i> 11	3.933 (2)	3.845 (5)	3.955 (1)
<i>d</i> 12	3.16 (2)	3.01 (3)	3.34 (2)
<i>d</i> 13	3.58 (2)	3.59 (4)	3.82 (3)

3.778 (1) and 3.865 (1) Å]. The third distance (*d*11) is slightly longer in (TMTSF)₂ClO₄ than in (TMTSF)₂ReO₄. In (TMTSF)₂ReO₄ at 120 K the distances *d*9, *d*10 and *d*11 are shortened between 2.2 and 3.4%.

The perrhenate and the perchlorate anions are located in a cavity produced by the TMTSF molecules. They also form sheets parallel to the *ab* plane, but with no intrasheet contacts. The shortest contact distance between the TMTSF molecule and ReO₄⁻ at 295 K is 3.16 (2) Å (*d*12), which is considerably shorter than the contact distance of 3.40 Å derived from Pauling's (1960) van der Waals radii for Se and O or the 3.42 Å derived from Bondi's (1964) values. The shortest distance between TMTSF and ClO₄⁻ is 3.34 (2) Å, only slightly shorter than the contact distance.

The structure of (TMTSF)₂ReO₄ at 120 K was determined with the same disorder in the ReO₄⁻ anion as at 295 K. The number of observations only allowed anisotropic thermal parameters for Re and Se atoms. Examination of the structure of (TMTSF)₂ReO₄ at a temperature below the transition temperature with the Weissenberg technique at 120 K revealed a doubling of the repeat distance along all three axes. The additional reflections at 120 K were all very much weaker than

the reflections measured for the average structure calculations. An ordering of the ReO₄⁻ group in such a way that the orientation alternates from one ion to the neighboring ion in all three directions can explain the additional reflections, as suggested by Pouget, Moret, Comès, Bechgaard, Fabre & Giral (1982). The fact that the 120 K structure refined well in space group *P*1 with quite normal anisotropic thermal parameters for Re and Se suggests that the anion ordering just mentioned is the only structural change involved in the metal-insulator transition at 182 K.

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References

- ANDERSEN, J. R., BECHGAARD, K., JACOBSEN, C. S., RINDORF, G., SOLING, H. & THORUP, N. (1978). *Acta Cryst.* B34, 1901–1905.
- BECHGAARD, K., CANEIRO, K., RASMUSSEN, F. B., OLSEN, M., RINDORF, G., JACOBSEN, C. S., PEDERSEN, H. J. & SCOTT, J. C. (1981). *J. Am. Chem. Soc.* 103, 2440–2442.
- BECHGAARD, K., JACOBSEN, C. S., MORTENSEN, K., PEDERSEN, H. J. & THORUP, N. (1980). *Solid State Commun.* 33, 1119–1125.
- BONDI, A. (1964). *J. Phys. Chem.* 68, 441–451.
- International Tables for X-ray Crystallography* (1974). Vol. IV, pp. 99, 149. Birmingham: Kynoch Press.
- JACOBSEN, C. S., PEDERSEN, H. J., MORTENSEN, K., RINDORF, G., THORUP, N., TORRANCE, J. B. & BECHGAARD, K. (1982). *J. Phys. C*. Submitted.
- JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.
- PAULING, L. (1960). *The Nature of the Chemical Bond*, 3rd ed., p. 260. Ithaca: Cornell Univ. Press.
- POUGET, J. P., MORET, R., COMÈS, R., BECHGAARD, K., FABRE, J. M. & GIRAL, L. (1982). *Mol. Cryst. Liq. Cryst.* 53. To be published.
- SHELDRICK, G. M. (1976). *SHELX 76*. A program for crystal structure determination. Univ. of Cambridge, England.
- STEWART, J. M., KRUGER, G. J., AMMON, H. L., DICKINSON, C. & HALL, S. R. (1972). The XRAY system – version of June 1972. Tech. Rep. TR-192. Computer Science Center, Univ. of Maryland, College Park, Maryland.
- THORUP, N., RINDORF, G., SOLING, H. & BECHGAARD, K. (1981). *Acta Cryst.* B37, 1236–1240.