

$-x$, $-\frac{1}{2} + y$, $1 - z$). A less clear-cut, apparently bifurcated, hydrogen-bonding system is seen in the contacts O(4a)—H(4a)··O(4) and O(4a)—H(4a)··O(3), with O··O 3.10, 3.16 Å, H··O 2.36, 2.42 Å respectively, receptor atoms at $-x$, $-\frac{1}{2} + y$, $-z$.

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Di(3,3',4,4'-tetramethyl-2,2',5,5'-tetraselenafulvalenium) Difluorophosphate,* (C₁₀H₁₂Se₄)₂PO₂F₂, at 293 and 125 K

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Abstract. (TMTSF)₂PO₂F₂: $M_r = 997.07$, triclinic, $P\bar{1}$, $Z = 1$, Mo $K\alpha$, $\lambda = 0.71073$ Å, $\mu = 112.8$ cm⁻¹, $F(000) = 465$. At 293 K: $a = 7.271$ (1), $b = 7.704$ (1), $c = 13.361$ (2) Å, $\alpha = 84.28$ (1), $\beta = 86.70$ (1), $\gamma = 70.50$ (1)°, $V = 701.8$ (2) Å³, $D_x = 2.359$ g cm⁻³, $R(F) = 0.046$, $R_w = 0.029\%$, $S = 1.46$ for 1962 reflections. At 125 K: $a = 7.128$ (2), $b = 7.694$ (2), $c = 13.201$ (5) Å, $\alpha = 85.35$ (3), $\beta = 87.23$ (3), $\gamma = 69.76$ (2)°, $V = 676.9$ (4) Å³, $D_x = 2.446$ g cm⁻³, $R(F) = 0.038$, $R_w = 0.029\%$, $S = 1.68$ for 2005 reflections. (TMTSF)₂PO₂F₂ exhibits a metal–insulator ($M-I$) phase transition at 137 K. The structures both above and below the transition temperature are isomorphous with other 2:1 TMTSF:X ($X =$ monovalent anion) salts. In the room-temperature structure the PO₂F₂⁻ ion is positioned with its phosphorus atom at the origin ($\bar{1}$ at 0,0,0). At 125 K (*i.e.* below the transition temperature) the phosphorus atom is shifted away from the origin by 0.38 Å with a total P–P separation of 0.76 Å. There is no indication of doubling of any of the three crystallographic axes, therefore the anion is disordered at both temperatures. This behavior has not been previously observed for a TMTSF salt

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containing any anion. Although the PO₂F₂⁻ anion possesses a dipole moment, this has little or no effect on the electrical properties. Rather, these properties are dominated almost entirely by the random potentials induced by the anion disorder that persists even below the 137 K phase transition. Therefore, contrary to other (TMTSF)₂X systems such as $X = PF_6^-$ and AsF_6^- , it is unlikely that (TMTSF)₂PO₂F₂ will exhibit superconductivity even under an applied pressure.

Introduction. Structures of electrically conducting and superconducting salts of stoichiometry (TMTSF)₂⁺MX₄⁻, where TMTSF is C₁₀H₁₂Se₄ and MX₄⁻ is an anion of tetrahedral geometry, have been determined for $MX_4^- = BF_4^-, ClO_4^-, BrO_4^-, ReO_4^-$ and FSO₃⁻ (for a summary see Thorup, Rindorf, Soling, Johannsen, Mortensen & Bechgaard, 1983). Of these only FSO₃⁻ is an asymmetric dipolar ion. It has a $M-I$ phase transition at 86 K, but that temperature lies below the lowest temperature (125 K) at which the crystal structure has been studied (Williams, Beno, Appelman, Capriotti, Wudl, Aharon-Shalom & Nalewajek, 1982). The difluorophosphate, PO₂F₂⁻, anion almost certainly has a higher dipole moment than FSO₃⁻, and the (TMTSF)₂PO₂F₂ salt has a $M-I$ transition at 137 K (Cox, Boyssel, Moses, Wudl, Chen, Ochsenein, Heeger, Walsh & Rupp, 1984), which lies above the temperature of 125 K that can be reached conveniently for X-ray crystallographic studies. The difluorophosphate salt therefore provides an opportunity of determining the structure above and below the phase transition. The results of the study are described

* Alternative nomenclature: di(4,4',5,5'-tetramethyl-2,2'-bi-1,3-diselenolyliiden)ium difluorophosphate.

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Table 1. Data collection and crystal parameters

Crystal size (mm)	0.042 × 0.083 × 0.40	
Scan range (°)	2.6	
Scan rate (° min ⁻¹)	1.95–11.72	
Total background/scan time	0.5	
Range of 2θ (°)	3–55	
Check reflections; decay	132, 114, $\bar{2}\bar{3}1$; none	
Frequency of checks	After every 93 reflections	
Function minimized in	$\sum w(F_o ^2 - F_c^2)$, $w = 1/\sigma(F_o^2)$	
least-squares refinement		
T of data collection (K)	293 (1)	125 (1)
Number of reflections measured	3528	3399
Number of unique reflections	1962	2005
with $I > 3\sigma(I)$		
Range of h, k, l	0–9, –8–9, –15–15	0–9, –9–9, –17–17
Final $R(F)$ (%)	4.6	3.8
Final R_w (%)	2.9	2.9
Final goodness-of-fit (S)	1.46	1.68
R_{int}	0.020	0.021
Transmission coefficient,	0.773/0.638	0.765/0.625
max./min.		

below and they provide new insight into the relationship between anion disorder and the electrical properties of (TMTSF)₂X systems.

Experimental. Crystals grown by electrocrystallization at 279 K, using 4 mM TMTSF and 0.9 M N(*n*-Bu)₄PO₂F₂ in 1,1,2-trichloroethane at 1.0 μA cm⁻² current density. The literature procedure of Williams (1984) was followed and the crystals were grown at slightly higher temperature than that reported by Cox *et al.* (1984). Crystals twinned in various degrees. Syntex P₂ automated diffractometer, graphite monochromator, cold-stream system that allowed cooling of crystal to 125 K. The twinning operation is a mirror plane perpendicular to crystallographic *a* axis so the two crystal fragments had a common *Ok**l* zone of reflections. Data collected (θ –2θ scans) from larger fragment (85% of total crystal) at 293 and 125 K. Crystal cooled to 168 K, *i.e.* about 30° above transition temperature, for determination of cell dimensions. [$a = 7.175$ (2), $b = 7.701$ (2), $c = 13.282$ (3) Å, $\alpha = 84.70$ (2), $\beta = 87.37$ (2), $\gamma = 69.80$ (2)°, $V = 685.7$ (3) Å³, $D_x = 2.414$ g cm⁻³.] Cell volume precisely linear with temperature. Cell dimensions, in all three cases, determined from least-squares refinement of setting angles of 24 reflections with $16 < 2\theta < 25^\circ$. Detailed information about data collection and final agreement factors of the refinement are given in Table 1. Intensities corrected for Lorentz factor, polarization and absorption. Latter correction used measured crystal dimensions and eight faces that bounded the crystal. Intensities of equivalent reflections averaged, those for which $F_o > 3\sigma(F_o)$ retained for structural calculations, all $F_o(Ok$ *l*) removed (164 and 154 reflections, respectively, for 293 and 125 K) because of being affected by twinning. Atomic and anomalous scattering factors from *International Tables for X-ray Crystallography* (1974). All computations using local modification of *UCLA Crystallographic Program Package* (Strouse, 1978).

Refinement of room-temperature structure. For a starting model the parameters of four Se and ten C atoms from the (TMTSF)₂SiF₅ structure (Eriks, Beno, Bechgaard & Williams, 1984) were taken, since the (TMTSF)₂X structures are all identical in the positions of the TMTSF molecules. The model refined smoothly, first with isotropic, then with anisotropic thermal parameters, and all the other atoms including the hydrogen atoms in the structure could be located. The problem that usually arises, especially in the room-temperature structures, is in obtaining unambiguous positions for the atoms of the anion. The ion is located around the center of inversion at the origin and is therefore disordered since the tetrahedral PO₂F₂⁻ ion does not possess $\bar{1}$ symmetry. In addition, it was found to be impossible to recognize the difference between O and F atoms. Several models were tried, all containing four half-oxygen atoms, or two half-oxygen atoms plus two half-fluorine atoms, surrounding the phosphorus atom: (1) P at 0,0,0; (2) P moved 0.3 Å away from 0,0,0 in the direction shown by the low-temperature model; and (3) P moved half the distance of (2). The first model was the only one that gave satisfactory results using the room-temperature data. It results in the agreement factors given in Table 1 and distances in Table 3, and showed no large correlation factors; the highest Δ/σ for non-hydrogen atoms was 0.16, and the highest residual peak in the difference Fourier map was 0.69 e Å⁻³. All three models, however, led to positions of the atoms of the TMTSF molecule that were identical within one standard deviation. After the final refinement cycle the values of $F(Ok$ *l*) were calculated and compared with their observed values. The scale factor $s = F_o/F_c$ was 1.086, and $R = \sum ||F_o| - |sF_c|| / \sum |F_o| = 0.037$ for the 164 *Ok**l* reflections that had not been used in the refinement.

Refinement of low-temperature structure. The beginning of the refinement was similar to that described above for 293 K, with the phosphorus atom placed at 0,0,0. A high persisting peak in the difference maps, close to the origin (0.6 Å), was an early indication that the phosphorus atom might not be at 0,0,0 in this case. Therefore, the phosphorus atom was moved half the distance indicated by the difference map, and it moved further away from the origin in subsequent cycles in agreement with the difference-map coordinates. Occupation factors for all anion atoms were fixed at 0.50. In the final cycles, all non-hydrogen atoms were refined with anisotropic thermal parameters, hydrogen atoms with fixed isotropic parameters ($B = 1.5$ Å²). The choice between oxygen and fluorine atoms was based solely on the calculated P–O(F) distances, the longer ones being taken as P–F. This assignment is in agreement with accepted P–O and P–F distances, (*International Tables for X-ray Crystallography*, 1968), although it may be somewhat dubious because of the high thermal motion. The final least-squares refinement showed no

correlation factors higher than 0.8; max. Δ/σ was 0.08 for C, 0.01 for O(F), 0.20 for H, and the highest peak in the difference map was $0.80 \text{ e } \text{\AA}^{-3}$, located close to the anion. Calculation of $F_c(OkI)$ after the refinement, as above, gave $s = 1.082$ and $R = 0.028$ for the 154 OkI reflections.

Since the PO_2F_2^- ion was definitely found to be off-center at 125 K, the possibility existed that the phase transition at 137 K had generated an ordered phase. Data were therefore collected at 125 K based on a $2a, 2b, 2c$ cell. 1470 reflection intensities were determined in the range $3 < 2\theta < 20^\circ$. 184 of these had all-even indices. Of the remaining 1286 reflections there were six with significant intensity [$5\sigma(I) < I < 12\sigma(I)$]. All six had all-odd indices (see *Discussion*).

Final positional parameters for the non-hydrogen atoms of both structures are listed in Table 2. The most important bond distances and angles are given in Table 3.*

Discussion. The important result of this study is that between 293 and 125 K the PO_2F_2^- ion moves from a position that is centered about the origin to one that is displaced from the origin by 0.375 \AA yielding a well-resolved site separation (P–P) of 0.75 \AA (Fig. 1). $(\text{TMTSF})_2\text{PO}_2\text{F}_2$ is reported to have a strong $M-I$ transition at 137 K (Cox, Boysel, Moses, Wudl, Chen, Ochsenein, Heeger, Walsh & Rupp, 1984). From our results it appears that during that transition the anion moves to its off-center position. The only other structure that has been reported containing a dipolar anion is that of the fluorosulfonate, FSO_3^- (Williams, Beno, Appelman, Capriotti, Wudl, Aharon-Shalom & Nalewajek, 1982). In that case, the $M-I$ transition lies at 86 K, and there is no indication that the anion has shifted to an off-center position at 125 K (the lowest temperature at which the crystal structure has been studied). The anion that does display a similar but much smaller displacement is the nonpolar perrenate ion in $(\text{TMTSF})_2\text{ReO}_4$, which has a $M-I$ transition at 180 K (Moret, Pouget, Comès & Bechgaard, 1982), and in which the Re atom moves to a position only 0.12 \AA from the origin below the transition temperature. In the ReO_4^- case, however, the structure below the transition is ordered with a supercell of dimensions $2a, 2b, 2c$, and it displays appreciable displacements of the TMTSF cations as well, as evidenced by a large number of rather intense superlattice reflections. In the present structure of the PO_2F_2^- salt the evidence for ordering

* Lists of structure factors, anisotropic thermal parameters, hydrogen-atom parameters, bond distances and angles in the TMTSF molecule and a figure showing two inversion-related PO_2F_2^- ions have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39738 (25 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

below the transition temperature is almost completely absent (six rather weak reflections observed), so the average disorder of the anion definitely remains below the transition point.

Table 2. Atomic parameters for $(\text{TMTSF})_2\text{PO}_2\text{F}_2$

	x	y	z	$U_{\text{eq}}(\text{\AA}^2 \times 10^4)^{\dagger}$
$T = 125 \text{ K}$				
Se(1)	0.6989 (1)	0.6563 (1)	0.3840 (1)	144 (2)
Se(2)	0.8416 (1)	0.2498 (1)	0.4898 (1)	131 (2)
Se(3)	0.7721 (1)	0.4282 (1)	0.7223 (1)	131 (2)
Se(4)	0.6184 (1)	0.8355 (1)	0.6159 (1)	137 (2)
C(1)	0.7488 (8)	0.5044 (8)	0.5065 (4)	121 (20)
C(2)	0.7772 (9)	0.4539 (9)	0.2994 (5)	168 (21)
C(3)	0.8377 (8)	0.2796 (9)	0.3448 (5)	157 (21)
C(4)	0.7647 (11)	0.5042 (11)	0.1889 (6)	216 (26)
C(5)	0.9087 (11)	0.1022 (10)	0.2944 (6)	187 (25)
C(6)	0.7165 (8)	0.5789 (9)	0.6000 (5)	136 (20)
C(7)	0.6845 (9)	0.6317 (9)	0.8043 (5)	188 (22)
C(8)	0.6208 (10)	0.8070 (9)	0.7594 (5)	192 (22)
C(9)	0.6967 (13)	0.5842 (11)	0.9177 (6)	309 (29)
C(10)	0.5443 (13)	0.9806 (11)	0.8146 (7)	316 (30)
P(1)	0.0095 (7)	-0.0360 (6)	0.0219 (3)	237 (17)
O(1)	0.0235 (19)	-0.1662 (17)	0.1123 (9)	533 (50)
O(2)	-0.0538 (38)	0.1572 (19)	0.0320 (12)	1008 (107)
F(1)	-0.0985 (16)	-0.0876 (22)	-0.0623 (8)	754 (58)
F(2)	0.2138 (13)	-0.1040 (19)	-0.0352 (8)	658 (49)
$T = 293 \text{ K}$				
Se(1)	0.7029 (1)	0.6579 (1)	0.3854 (1)	370 (3)
Se(2)	0.8353 (1)	0.2512 (1)	0.4892 (1)	364 (3)
Se(3)	0.7704 (1)	0.4207 (1)	0.7196 (1)	355 (3)
Se(4)	0.6277 (1)	0.8280 (1)	0.6148 (1)	358 (3)
C(1)	0.7469 (8)	0.5061 (8)	0.5070 (5)	325 (25)
C(2)	0.7772 (8)	0.4581 (9)	0.3018 (5)	316 (24)
C(3)	0.8333 (8)	0.2865 (10)	0.3459 (5)	339 (25)
C(4)	0.7678 (14)	0.5148 (13)	0.1916 (6)	509 (36)
C(5)	0.8970 (12)	0.1103 (11)	0.2941 (7)	489 (34)
C(6)	0.7185 (8)	0.5740 (9)	0.5974 (5)	311 (23)
C(7)	0.6907 (9)	0.6209 (10)	0.8011 (5)	367 (25)
C(8)	0.6272 (9)	0.7944 (10)	0.7572 (5)	358 (24)
C(9)	0.6991 (14)	0.5737 (14)	0.9124 (6)	581 (38)
C(10)	0.5570 (15)	0.9672 (13)	0.8095 (7)	575 (37)
P(1)	0.0000	0.0000	0.0000	1045 (280)
O(1)	0.0267 (46)	-0.1193 (28)	0.1045 (15)	2059 (112)
O(2)	-0.0294 (22)	-0.1556 (20)	-0.0479 (12)	1055 (81)
F(1)	0.1329 (32)	0.0340 (53)	0.0088 (37)	4408 (260)
F(2)	-0.1909 (21)	0.1251 (22)	0.0372 (13)	1482 (89)

$$\dagger U_{\text{eq}} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

Table 3. Bond distances (\AA) and angles ($^\circ$)

	293 K	125 K	293 K	125 K
P–O(1)	1.58 (2)	1.478 (12)	O(1)–P–O(2)	90.1 (10) 120.5 (8)
–O(2)	1.49 (1)	1.412 (15)	–F(1)	95.0 (26) 109.3 (8)
–F(1)	1.10 (2)	1.532 (11)	–F(2)	90.0 (11) 106.8 (7)
–F(2)	1.49 (1)	1.547 (10)	O(2)–P–F(1)	131.2 (18) 113.0 (9)
P–P*	0	0.750 (6)	–F(2)	109.3 (8) 110.8 (9)
			F(1)–P–F(2)	119.1 (8) 92.8 (6)
	293 K	125 K	293 K	125 K
Se(1)–Se(2 ⁱ)	4.033 (1)	3.974 (2)	P C(9 ⁱⁱ)	— 4.039 (9)
Se(1)–Se(3 ^j)	4.036 (1)	3.972 (2)	P C(10 ^j)	— 4.088 (10)
Se(1)–Se(3 ^h)	3.875 (1)	3.814 (2)	P C(5 ⁱⁱⁱ)	4.078 (9) 3.799 (9)
Se(1)–Se(2 ⁱⁱ)	4.126 (1)	4.048 (1)	O(1)–H(9B)	— 2.28 (9)
			–H(5A)	— 2.64 (9)
			–H(4A)	— 2.68 (9)
Se(2)–Se(4 ^h)	3.938 (1)	3.883 (2)	O(2)–H(9B)	— 2.43 (9)
Se(2)–Se(4 ⁱ)	3.964 (1)	3.889 (2)	–H(4C)	— 2.43 (9)
Se(1)–Se(4 ^h)	3.889 (1)	3.780 (1)	–H(5A)	— 2.47 (9)
Se(4)–Se(4 ^h)	3.957 (2)	3.861 (2)	–H(9C)	2.44 (9) —
Se(2)–Se(2 ^{iv})	3.806 (2)	3.720 (2)	F(1)–H(9A)	— 2.35 (8)
Se(2)–Se(4 ^v)	4.199 (1)	4.226 (1)	–H(5A)	— 2.42 (9)
Se(3)–O(1 ^v)	3.187 (18)	2.919 (12)	–H(10C)	— 2.54 (8)
			F(2)–H(5A)	— 2.63 (9)

Symmetry operations: (i) $1-x, 1-y, 1-z$; (ii) $2-x, 1-y, 1-z$; (iii) $1-x, 2-y, 1-z$; (iv) $2-x, -y, 1-z$; (v) $x, -1+y, z$; (vi) $1-x, -y, 1-z$; (vii) $-1+x, y, z$.

The first nine Se–Se distances are in the sequence d_1-d_9 , Se(3)–O(1) is d_{10} . The last Se–Se distance is d_{11} .

The direction of displacement of the $PO_2F_2^-$ is of interest. The phosphorus atom moves almost precisely in the line origin–P–O(1)–Se(3), so the entire ion is much closer to Se(3) at 125 K than at room temperature. This has several consequences. The first is that the Se(3)–O(1) distance is extremely short below the transition: 2.919 Å, compared with the sum of van der Waals radii of 1.90 (Se) + 1.52 (O) = 3.42 Å (Bondi, 1964). This is the shortest Se(3)–X separation yet seen in this class of compounds and it refutes a previous suggestion (Thorup, Rindorf, Soling, Johannsen, Mortensen & Bechgaard, 1983) that short Se–(F,O) contacts are correlated with superconductivity, since the $PO_2F_2^-$ salt does not become superconductive even under applied pressure (Cox *et al.*, 1984). Secondly, the displacement of the anion gives rise to a rather large number of short (<2.6 Å) contacts between oxygen (or fluorine) and methyl hydrogen atoms, as can be seen in Table 3 and Fig. 2. The low-temperature structure has seven of these hydrogen bonds, while the room-temperature structure has only one. The significance of hydrogen-bond formation and anion ordering in $(TMTSF)_2X$ salts has been discussed elsewhere (Beno, Blackman, Leung & Williams, 1983). It should be noted that the direction of displacement of the $PO_2F_2^-$ ion is quite different from that of the ReO_4^- ion discussed above.

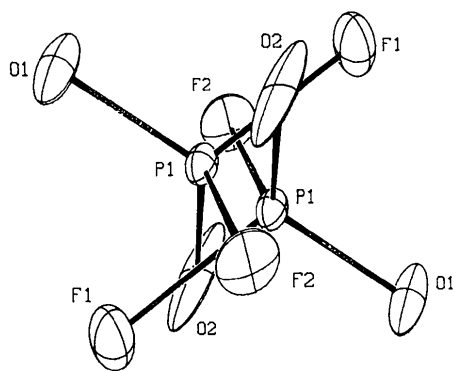


Fig. 1. $PO_2F_2^-$ ion, showing the P–P' site separation, derived from the 125 K data, below the *M*–*I* phase transition at 137 K.

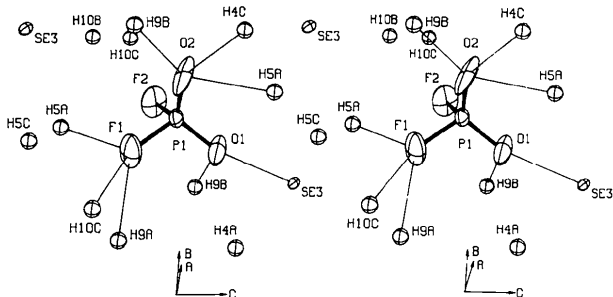


Fig. 2. Stereoview of the $PO_2F_2^-$ ion in its cavity surrounded by methyl-group hydrogen atoms and a selenium atom. Short O(F)···H contacts are indicated by thin lines.

Hydrogen-atom positions were refined at both temperatures. At 293 K the average C–H distance was 0.97 Å, the range 0.70–1.11 Å, and the standard deviation of a C–H bond length 0.09 Å. At 125 K the values were 0.97 Å, 0.74–1.19 Å and 0.09 Å, respectively.

The geometry of the anion was found with accuracy better than usual for these compounds. At both temperatures, after the refinement, only one residual peak close to the anion remained in the difference map: at 293 K a peak of $0.53 e \text{ \AA}^{-3}$ at 1.43 Å from the origin, and at 125 K a peak of $0.85 e \text{ \AA}^{-3}$ at 1.21 Å from the phosphorus atom. The anion geometry is tetrahedral, and the distances and angles found at 125 K agree well with those reported for KPO_2F_2 (Harrison, Thompson & Trotter, 1966) and for $RbPO_2F_2$ (Granier, Durand, Cot & Galigné, 1975). Clearly, however, the atomic positions of the anion could not be determined with great precision, as evidenced by the very high anisotropic motion of these atoms and the unlikely value of 1.10 Å for one P–F distance at 293 K. This difficulty has been encountered in all structure determinations of $(TMTSF)_2X$ compounds. It is an obvious consequence of the disorder of the anion in almost all of these crystals.

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