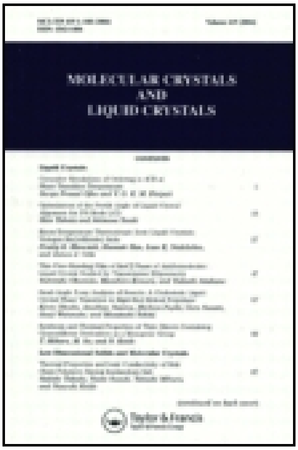

Molecular Crystals and Liquid Crystals
Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/gmcl16

X-Ray Diffuse Scattering Study of Some $(TMTSF)_2X$ and $(TMTTF)_2X$ Salts

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Version of record first published: 14 Oct 2011.

To cite this article: J. P. Pouget, R. Moret, R. Comes, K. Bechgaard, J. M. Fabre & L. Giral (1982): X-Ray Diffuse Scattering Study of Some $(TMTSF)_2X$ and $(TMTTF)_2X$ Salts, Molecular Crystals and Liquid Crystals, 79:1, 485-499

To link to this article: http://dx.doi.org/10.1080/00268948208070995

Mol. Cryst. Liq. Cryst., 1982, Vol. 79, pp. 129–143
0026-8941/82/7901-0129$06.50/0

(Proceedings of the International Conference on Low-Dimensional Conductors, Boulder, Colorado, August 1981)

X-RAY DIFFUSE SCATTERING STUDY OF SOME $(TMTSF)_2X$ AND $(TMTTF)_2X$ SALTS

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Received for publication October 26, 1981

X-ray diffuse scattering experiments on the superconducting salts $(TMTSF)_2X$ with X = PF_6, AsF_6 and ClO_4 reveal no charge density wave instability, in contrast with the poorly conducting $(TMTTF)_2PF_6$ salt. New structural phase transitions coinciding with conductivity anomalies are found in $(TMTSF)_2NO_3$, $(TMTSF)_2ReO_4$ and $(TMTTF)_2ClO_4$. It is suggested that they correspond to order-disorder transitions involving the non-centrosymmetric counter ions. Relationships between these structural instabilities and superconductivity are also discussed.

INTRODUCTION

Although theoretical studies predicted fifteen years ago[1] that a one-dimensional (1D) interacting electron gas could

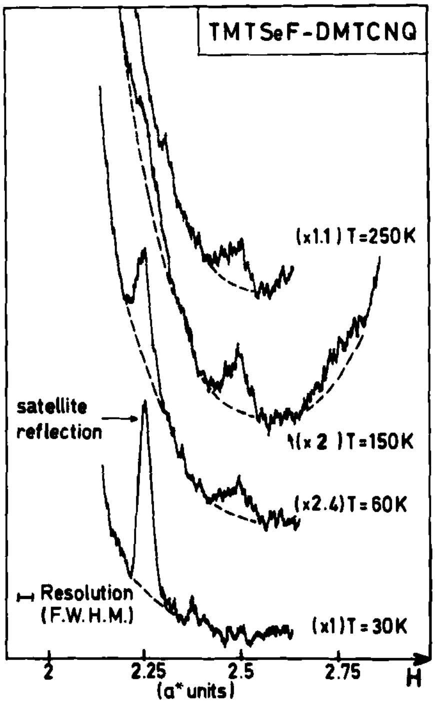

FIGURE 1

Microdensitometer readings of X-ray patterns from TMTSF-DMTCNQ showing the 0.25 a ($2k_F$) and 0.5 a* ($4k_F$) scattering, and the condensation of the $2k_F$ scattering in a satellite reflection below the Peierls transition which takes place at 42 K.*

show instabilities in the charge density wave (CDW), spin density wave (SDW) and superconducting responses, experimental studies of the two-chain organic charge transfer salts of the TCNQ family have revealed the first instability only. The 1D CDW instability was unambiguously proven, via the electron-phonon coupling, by the formation at $2k_F$ of a planar Kohn anomaly in the phonon spectrum (k_F is the Fermi wave vector of the 1D electron gas). The divergence of the 1D CDW fluctuations causes a softening of the Kohn anomaly (and an enhancement of the X-ray $2k_F$ diffuse scattering), which drives a structural distortion towards an insulating Peierls state.

This behaviour is well illustrated by the charge transfer organic salt TMTSF-DMTCNQ which presents a $2k_F$ CDW instability below 225 K[23]. As figure 1 clearly shows, the $2k_F$ X-ray scattering increases as the temperature decreases towards the Peierls transition which occurs at 42 K. At this temperature the compound undergoes a structural distortion characterized by the modulation wave vector : (1/4 a*, 1/3 b*, 0 c*). In addition figure 1 shows that

TMTSF-DMTCNQ presents a $4k_F$ scattering at room temperature but, unlike TTF-TCNQ, this scattering does not increase at low temperature and does not condense into satellite reflections .

The discovery of pressure-induced[4] superconductivity in the one-chain salt $(TMTSF)_2PF_6$ has renewed the possibilities for the experimental study of instabilities in the 1D electron gas. In particular this concerns the search for eventual structural instabilities in the family of one-chain compounds $(TMTTF)_2X$ and $(TMTSF)_2X$ and their comparison with those reminded above for the family of two-chain compounds.

In the one-chain compounds X are inorganic ions of various geometries :
- PF_6, AsF_6 (octahedron),
- ClO_4, ReO_4, BF_4 (tetrahedron)
- NO_3 (triangle).

They are located at the inversion center of the triclinic unit cell[5]. These anions are singly charged and, owing to the 1 : 2 stoichiometry of the salts, they leave 1/2 hole per donor molecule (value which, surpringly, is also found in TMTSF-DMTCNQ at atmospheric pressure), which leads to the commensurate value $2k_F = a^*/2$ in a 1D band scheme (there are 2 organic molecules per stack in the unit cell).Until recently, this was believed to be the primary effect of the anions. However, we shall show below that, in some cases[6-7] , they have a subtle influence on the electronic properties of the organic stacks.

So far single-chain compounds with donor molecules are the only ones to exhibit a superconducting behaviour. Neither the two chain compounds, nor the single-chain compounds with acceptor molecules like $Qn(TCNQ)_2$, display the same property. It has been recently established[8], from the study of $(NMP)_x(Phen)_{1-x}$ TCNQ alloys, that the crossover from a two-chain to a one chain situation reduces interchain screening effects and enhances electron-electron interactions. This change might be at the origin of new instabilities shown by one-chain compounds $(TMTTF)_2X$ and $(TMTSF)_2X$, and could explain why these salts behave so differently from the earlier studied two-chain compounds. The influence of electron-electron interactions on the onset of superconductivity in $(TMTSF)_2PF_6$ has already been considered theoretically[9-10] .

I. SEARCH FOR $2K_F$ CDW INSTABILITIES

The simplest situation occurs with $(TMTSF)_2ClO_4$ which remains metallic at atmospheric pressure in the whole temperature range, and undergoes a phase transition to the superconducting state at 1.4 K[11]. X-ray diffuse scattering experiments show that a very weak and broad planar scattering, at the $2k_F$ wave vector in chain direction, can be detected below about 150 K (fig 2). However unlike the $2k_F$ scattering of TMTSF-DMTCNQ, this scattering does not grow appreciably in intensity as the temperature is lowered. This observation is the structural illustration of the mutual exclusion of superconducting and CDW instabilities.

$(TMTSF)_2PF_6$ and $(TMTSF)_2AsF_6$ exhibit a relatively more complex phase diagram. These compounds are superconductors under pressure only, but at room pressure they undergo, at about 12 K, a metal-insulator phase transition[13]. X-ray diffuse scattering experiments again show that below about 125 K, a very weak planar $2k_F$ scattering just emerges from the thermal background (see figure 3 for $(TMTSF)_2PF_6$ and ref 12 for $(TMTSF)_2AsF_6$). This scattering becomes undetectable below about 35 K, which could be explained either by the vanishing of the $2k_F$ anomaly itself or by the effect of the decreasing thermal population factor on a temperature independent phonon anomaly. The non-divergence of the CDW instability might be due either to the suppression of 1D fluctuations by interchain coupling[14] or to a crossover to another type of fluctuation[9]. In any case, this result clearly proves that the 12 K metal-insulator transition is not a Peierls transition. With these data, it was suggested one year ago[3], that it might be a Slater transition[15] to an insulating magnetic state. Since then, a lot of experiments[16] have shed light on its magnetic nature, but its magnetic periodicity has not yet been determined by diffraction experiments.

An interesting comparison can be made with the sulfur analogue $(TMTTF)_2PF_6$ which does not exhibit superconductivity. X-ray measurements (fig 4) show that a quasi-planar $2k_F$ anomaly develops below about 60 K[3]. As the temperature decreases the $2k_F$ scattering sharpens, increases in intensity and, below about 10-15 K, gives rise to very weak satellite reflections (the transition tem-

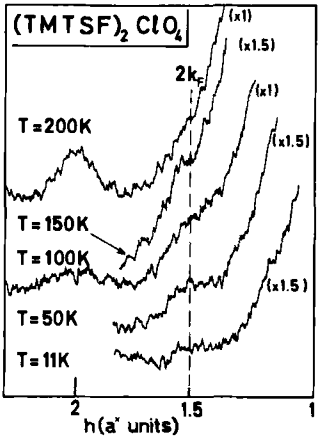

FIGURE 2 : Microdensitometer readings of X-ray patterns from $(TMTSF)_2ClO_4$ *showing the very weak 0.5* $a^*(2k_F)$ *scattering below about 150 K.*

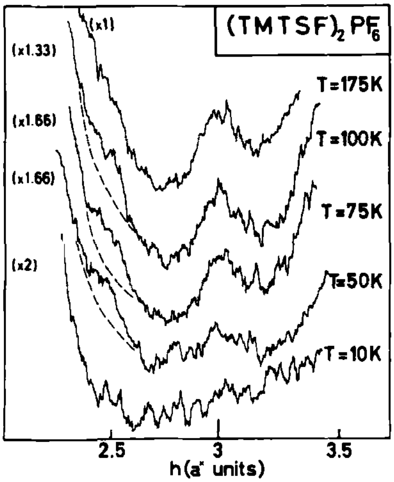

FIGURE 3 : Microdensitometer readings of X-ray patterns from $(TMTSF)_2PF_6$ *showing the weak 0.5* a^* $(2k_F)$ *scattering between about 125 K and 35 K.*

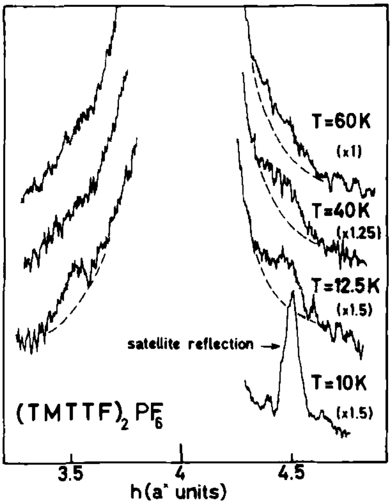

FIGURE 4 : Microdensitometer readings of X-ray patterns from $(TMTTF)_2PF_6$ *showing the 0.5* a^* $(2k_F)$ *scattering below 60 K and its condensation into a satellite reflection at about 10 K.*

perature is very sensitive to X-ray irradiation). This phase transition is accompanied by a decrease of the magnetic susceptibility below 15 K[17]. These results suggest that, unlike the other salts, $(TMTTF)_2PF_6$ presents the features of a Peierls transition. However, one can notice that the phase transition occurs at very low temperature (15 K) and that it is announced within a narrow temperature range (∿ 45 K) by quasi-1D fluctuations of relatively weak amplitude. In this case, the Peierls transition might be less pure than in the earlier studied TCNQ compounds, due to the somewhat localized character of the conduction electrons[17].

II LOW TEMPERATURE SUPERSTRUCTURE FORMATION

1D X-ray diffuse scattering and satellite reflection intensities considered in figures 1 to 4 are in fact much weaker (between about 7 and 4 orders of magnitude) than the intensity of a common Bragg reflection of the average lattice. This corresponds to weak precursor effects and minor structural distortions, respectively. We shall present below much larger structural modifications occuring in the $(TMTTF)_2X$ and $(TMTSF)_2X$ salts, where X is explicitely a non-centrosymmetric counter ion.

These effects are well illustrated on figure 5 which presents X-ray patterns from $(TMTTF)_2ClO_4$ taken at various temperatures. At room temperature (fig 5a), only main Bragg reflections of the triclinic P$\bar{1}$ lattice are observed. At low temperature, figure 5c reveals strong superstructure reflections half way between ⊥ayers of main Bragg reflections perpendicular to the a stacking direction, indicating a structural change. On figure 5b, 30 K above the phase transition, broad and isotropic diffuse spots are observed in place of the low temperature superstructure reflections. Similar observations have been made for $(TMTSF)_2ReO_4$ and $(TMTSF)_2NO_3$[7].However no such superstructure reflection could be detected above 10 K either in the atmospheric pressure superconducting salt $(TMTSF)_2ClO_4$ or in salts with octahedral (centrosymmetric) counter ions like PF_6 or AsF_6.

The wave vectors and superstructure cells are given in table I for three compounds investigated, along with the transition temperature as deduced from the vanishing of the superstructure reflection intensity. Figure 6 shows

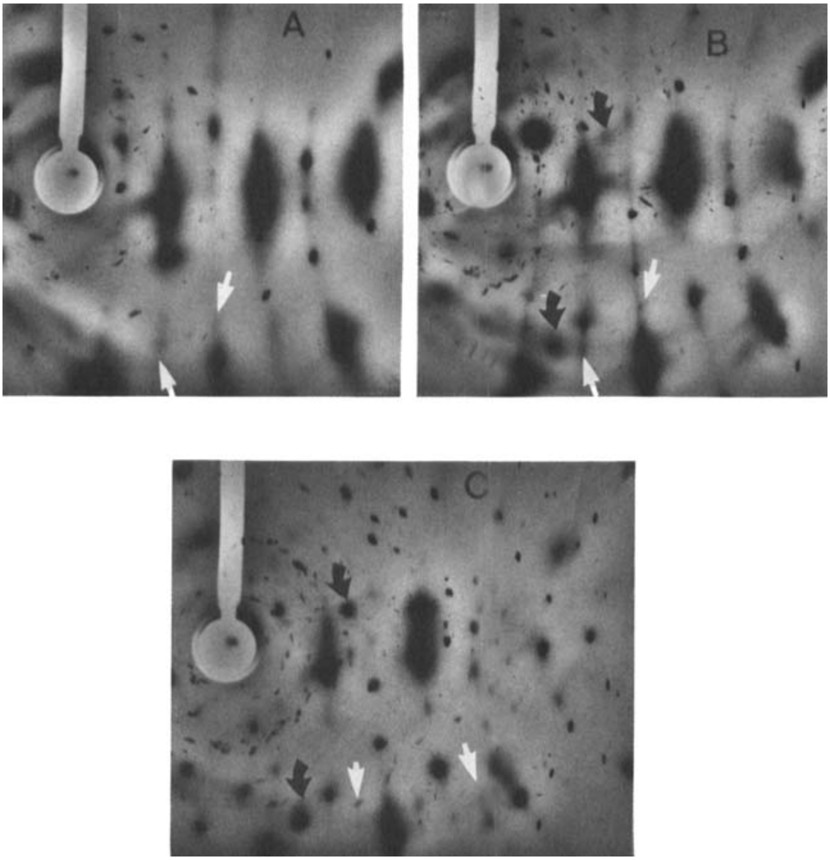

FIGURE 5 : X-ray patterns from (TMTTF)$_2$ClO$_4$ at (a) 295 K, (b) 100 K and (c) 50 K. Black arrows point to the low temperature superstructure reflections in (c) and to the high temperature isotropic precursor scattering in (b). White arrows point to the a ($4k_F$) scattering in (a) and (b) and to the 0.5 a*($2k_F$) scattering in (c). (In these patterns the a direction is horizontal).*

Table I

	$\vec{q}_{super.}$	Superstructure	T_c (K)
$(TMTSF)_2NO_3$	$(\frac{1}{2}, 0, 0)$	$2\vec{a} \times \vec{b} \times \vec{c}$	41
$(TMTSF)_2ReO_4$	$(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$	$(\vec{a}+\vec{b}) \times (\vec{b}+\vec{c}) \times (\vec{c}+\vec{a})$	176
$(TMTTF)_2ClO_4$	$(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$	$(\vec{a}+\vec{b}) \times (\vec{b}+\vec{c}) \times (\vec{c}+\vec{a})$	70
$(TMTSF)_2ClO_4$	No superstructure reflection observed above 10 K		

that the temperature dependence of the superstructure reflections is similar in $(TMTTF)_2ClO_4$, $(TMTSF)_2ReO_4$ and $(TMTSF)_2NO_3$ which suggests similar structural modifications in the three salts. Two other common structural features are worth noting :

a) the superstructure reflections have a relatively high intensity (about 1/10 of a main Bragg reflection), which is further of the same order of magnitude for the three salts ;

b) the precursor scattering consists of broad and isotropic diffuse spots in the whole temperature range where it is observed (see figure 7 for a clear illustration in the case of $(TMTSF)_2ReO_4$) and is never transformed into a diffuse sheet.

This means that the phase transition at T_c is not a Peierls transition and that the associated fluctuations do not reflect the electronic anisotropy of these materials. Figure 7 in the case of $(TMTSF)_2ReO_4$ and figure 6 for $(TMTSF)_2NO_3$ show that these fluctuations are enhanced when T_c is approached from above, which means that the primary order parameter of the transition has a structural origin.

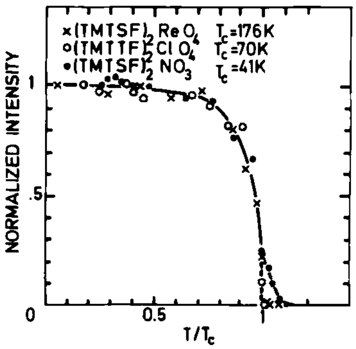

FIGURE 6 :Temperature dependence of the superlattice reflection intensity as a function of reduced temperature (T/T_c) in $(TMTSF)_2ReO_4$, $(TMTTF)_2ClO_4$ and $(TMTSF)_2NO_3$. Note the critical scattering above T_c for the latter compound.

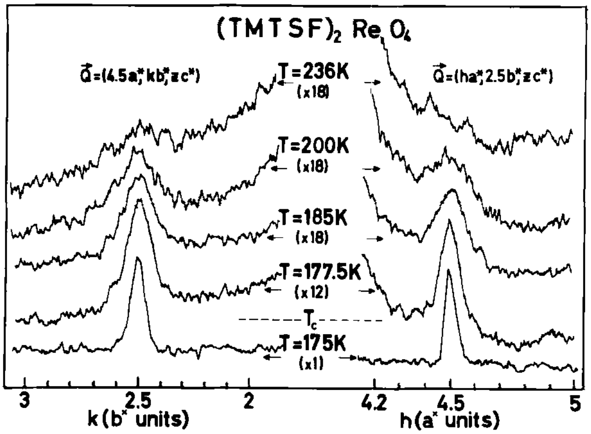

FIGURE 7 : Microdensitometer readings of X-ray patterns from $(TMTSF)_2ReO_4$ showing the broadening of superlattice reflections above T_c in the form of isotropic diffuse spots along a^* and b^*.

Structural determinations, performed at room temperature, give the $P\bar{1}$ triclinic space group for all $(TMTSF)_2X$ (5b)[18] and $(TMTTF)_2X$ (5a)[19] salts and locate the counter ions on the inversion center. This implies a structural disorder for the non-centrosymmetric counter ions considered in this section, with at least two orientations related by inversion. Therefore it is tempting to relate the strong structural modification observed at T_C to an order-disorder transition involving the counter ions. Since the oxygen atoms are mainly involved in such a transition it would explain why superstructure reflections of comparable intensities are observed in the three compounds independently of the organic molecule (S or Se). However, this suggestion must be confirmed by a full structure determination below T_C and does not exclude simultaneous structural distortions of the organic stacks.

In the case of $(TMTSF)_2ReO_4$ and $(TMTTF)_2ClO_4$, the superstructure corresponds to an alternation of the 2 tetrahedron orientations in the a, b and c directions. However such a type of order does not minimize the direct electrostatic energy, which might mean that the superstructure is in fact stabilized by a gain of electronic energy coming from the opening of an energy gap (see below)[20]. A somewhat different situation arises with $(TMTSF)_2NO_3$ where the ordering occurs in the $\vec{a}$ direction only (table I). Unfortunately the orientation of the triangular NO_3^- ion is still unknown and the change of electrostatic coupling energy for different orientations cannot be estimated. Anyway, a full treatment of the phase transition must take into account the structural and electronic degrees of freedom and their coupling, because the $a^*/2$ component of the superstructure reflections corresponds here to the value of $2k_F$. The phase transition is therefore associated with a modification of the Fermi surface and consequently affects the electronic properties.

In contrast to the apparent unity of the structural data (figure 6), these compounds present very different electric behaviours at T_C. In $(TMTSF)_2ReO_4$, a first order metal-insulator transition, with a small drop in the electrical conductivity, occurs at 182 K[21]. Accompanying the structural transition, a gap opens at the Fermi level. In $(TMTTF)_2ClO_4$, which is a poor conductor at low temperature, the phase transition is also characterized by a drop of the magnetic susceptibility[22], a specific heat anomaly and a 25 % increase of the electrical conductivity[17]. The hysteresis and jump of the electrical conductivity reveal the first order character of the transition, while the entropy jumps by $\Delta S \simeq R \log 2$ at T_C, a value expected for the removal of two-fold degeneracy of the tetrahedron orientations[17]. In $(TMTSF)_2NO_3$, the transition is detected by anomalies in the temperature dependence of the electrical conductivity and the thermoelectric power, although no discontinuity is observed around 41 K[13]. Its second character, compared to the first order one of $(TMTTF)_2ClO_4$ and $(TMTSF)_2ReO_4$, is in agreement with the stronger divergence of structural precursor effects in $(TMTSF)_2NO_3$ (see figure 6). In $(TMTTF)_2ClO_4$ and $(TMTSF)_2NO_3$, the increase of electrical conductivity observed at or below T_C might be explained by an increase of the carrier mobility (the scattering mechanism is reduced by the counter ion ordering). In the case of the highly conducting $(TMTSF)_2NO_3$, the increase in mobility must overcome the loss of carriers due to superstructure-induced Fermi surface perturbations. The non observation of a metal-insulator transition indicates that the Fermi surface of $(TMTSF)_2NO_3$ is not planar and that until T_C at least, coherent electronic transfer takes place between stacks. Such an explanation is consistent with the development of a transverse plasma edge at low temperature and atmospheric pressure[23] and closed orbit, under pressure[24] in the related $(TMTSF)_2PF_6$. However partial gaps opened at the Fermi surface, either by the potential coming from the NO_3^- anion order in chain direction or (and) by the eventual distortion of the organic lattice with the same periodicity, must be very weak (weaker than the interstack hopping integrals).

Besides the isotropic main features described above, two kinds of temperature-dependent, quasi-planar X-ray diffuse scattering features have been observed in $(TMTTF)_2ClO_4$ and $(TMTSF)_2ReO_4$. They are shown in figure 5 for $(TMTTF)_2ClO_4$:

a) below T_C, very faint quasi-1D scattering in the form of sheets perpendicular to the stacking direction and with a wave vector of $a^*/2$ ($2k_F$), becomes clearly visible. This is also shown in the case of $(TMTSF)_2ReO_4$ on the microdensitometer readings presented figure 8. This scattering cannot be detected at low

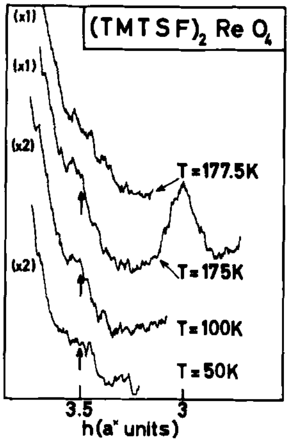

FIGURE 8 : Microdensitometer readings of X-ray patterns from (TMTSF)$_2$ReO$_4$ showing the very weak 0.5 a (2k$_F$) scattering emerging from the thermal scattering below T$_c$.*

temperature (T ∿ 20 K) ;

b) at room temperature, figure 5a shows another quasi 1D scattering running through the Bragg reflections of the main lattice, thus corresponding to the a* (4k$_F$) periodicity. Unlike a thermal scattering, the diffuse sheets sharpen with decreasing temperature (figure 5b), but still vanish at low temperature (figure 5c) where they seem to merge into the main Bragg reflections.

Both anomalies need further studies.

CONCLUSION

Structural studies of the superconducting compounds such as (TMTSF)$_2$PF$_6$, (TMTSF)$_2$AsF$_6$ and (TMTSF)$_2$ClO$_4$ fail to reveal the CDW instability usually observed in one dimensional conductors. This can be generally interpreted as reflecting the mutual exclusion of the instabilities leading respectively to the superconducting state and to the Peierls insulator. But the observation, in a limited temperature range of very weak 2k$_F$ anomalies, implies that the 3 salts investigated are not too far from a CDW instability. The sulfur analogue however (TMTTF)$_2$PF$_6$ presents this instability.

Now if we consider salts with non-centrosymmetric counter ions, it is remarkable to observe that the only compound which does not present the counter ion ordering effect, $(TMTSF)_2ClO_4$, undergoes the superconducting phase transition at atmospheric pressure. However its sulfur analogue, $(TMTTF)_2ClO_4$, presents this counter ion ordering. An another interesting compound is $(TMTSF)_2NO_3$ for which the counter ion ordering maintains the metallic state. Conducting measurements show that this phase transition is nearly pressure independent and always corresponds to a metal-(semi) metal transition[25]. Moreover under pressure (when the 12 K metal insulator phase transition is suppressed), $(TMTSF)_2NO_3$ never becomes superconducting[25]. With these compounds one observes a close correlation between the existence of superconductivity and the absence of counter ion ordering[16]: the ordering with the $2k_F$ periodicity in chain direction either leads to an insulating state or opens partial gaps which, by depressing the density of state at the Fermi level, leads to unfavourable conditions for superconductivity with high critical temperatures [26]. In this respect, $(TMTSF)_2ReO_4$ is the only compound of the family displaying both superconductivity (above 11 Kbar) and the counter ion ordering transition. One should note that this transition which occurs around 180 K at atmospheric pressure, at the same time as the metal insulator transition, might disappear under pressure as does the metal insulator transition[27]. Usually an order-disorder transition occurs if the gain in interaction energy overcomes the tunneling energy between the double-trough potential which favours the disordered state[28]. It is thus possible that pressure will increase so much the tunneling energy of ReO_4 that the ordered state might be suppressed and the metallic phase stabilized[29]. This would bring $(TMTSF)_2ReO_4$ back into the assumed rule of mutual exclusion of superconductivity and counter ion ordering.

ACKNOWLEDGEMENTS

We wish to acknowledge very fruitful exchanges with our colleagues in Orsay working in the field of one dimensional conductors. We are also very grateful to S. Barisic, C. Coulon, V.J. Emery and J.B. Torrance for several illuminating discussions.

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