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Anion ordering and lattice expansion in $(\text{TMTSF})_2\text{ClO}_4$

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Résumé. — Nous présentons des mesures de dilatation haute résolution dans le composé organique $(\text{TMTSF})_2\text{ClO}_4$ selon l'axe a dans les états quenché et relaxé. La contribution principale est celle des phonons. Quand T décroît à partir de 24 K, un terme négatif apparaît progressivement et nous montrons sans ambiguïté qu'il est relié au phénomène de mise en ordre des anions. L'intensité de cet effet est exactement proportionnelle à celle des pics de surstructure mesurée aux rayons X. Nous présentons enfin une analyse phénoménologique de ce résultat, basée sur un modèle d'Ising 3D anisotrope, et nous discutons les paramètres de couplage microscopiques.

Abstract. — We present high resolution thermal expansion measurements at low temperature in $(\text{TMTSF})_2\text{ClO}_4$ along the a -axis in both quenched and relaxed states. The main contribution arises from phonons. Below 24 K, an additional negative contribution develops progressively; we show that it is unambiguously due to the anion ordering phenomenon, with a well-defined relation of proportionality between this extra thermal expansion and the superstructure peak intensity. This result is analysed within a phenomenological approach, based on an anisotropic 3D Ising model and microscopic coupling constants are discussed.

1. Introduction.

$(\text{TMTSF})_2\text{ClO}_4$ was the first organic compound in which superconductivity at ambient pressure was observed at 1.3 K by resistivity measurements [1] and Meissner effect [2]. The stabilization of the superconducting state depends strongly on the cooling rate. Specific heat [3], EPR [4] and resistivity [4] measurements show that a slow cooling (typically 0.2 K/min, relaxed state) leads to superconductivity while a fast cooling (typically 2 K/s, quenched state) leads to a SDW insulator state [5]. Moreover these two different behaviours depend on the occurrence of the anion ordering transition which takes place at about 24 K [6, 7]. When the sample is cooled down from 40 K very slowly, the non-centrosymmetric anions order; the observed superstructure is characterized by a wave vector $q = (0, 1/2, 0)$. On the other hand, rapid cooling produced generally not an exactly disordered state at low temperature, but an intermediate state where the degree of order can be measured by the superstructure peak intensity [8]. It has been shown that in such intermediate states the superconducting and SDW instabilities of the electron gas are competing with each other [4].

In view of understanding the microscopic coupling

between the anion ordering and these interesting electronic gas properties, it is important to know how the lattice is affected by the order. Previous X-ray measurements [9] did not give precise information in the temperature region of anion ordering, nevertheless they showed that thermal expansion roughly follows a quasilinear behaviour between 50 K and 300 K, with an average thermal expansion coefficient along the a -axis $\alpha_a = 7.6 \times 10^{-5} \text{ K}^{-1}$. It is interesting to notice that this value is very close to that measured in $(\text{TMTSF})_2\text{PF}_6$ [10], i.e. $\alpha_a = 7.4 \times 10^{-5} \text{ K}^{-1}$, suggesting that the main contribution to the thermal expansion can be considered roughly independent of the anion in the $(\text{TMTSF})_2\text{X}$ series.

We have performed previously a high resolution capacitive thermal expansion study in $(\text{TMTSF})_2\text{PF}_6$ [11] and showed that thermal expansion arises from phonons in the whole range of temperature. Combining these two features we can estimate the phonon contribution to the thermal expansion of $(\text{TMTSF})_2\text{ClO}_4$ in the further analysis of our data presented here. We first give the experimental details, followed by the experimental results. In the second half of the paper we present a phenomenological model to interpret the data and finally we discuss the deduced coupling constants.

2. Experimental.

We measure the thermal expansion using a standard capacitance technique whose sensitivity reaches a few 10^{-8} for Δ/l . Details of the method can be found elsewhere [12]. Single crystals of $(\text{TMTSF})_2\text{ClO}_4$ have been prepared by electrochemical method. Three samples, 4 mm long, were mounted in the thermal expansion cell with the measurement direction along the a -axis. We measured the thermal expansion of two sets of samples between 1.2 K and 100 K and results were reproducible.

The relaxed state was obtained using the following process : after the initial cooling down to 1.2 K, the samples were heated up to 40 K, and then cooled again with a progressive decrease of the heating to give a controlled rate of -0.2 K/min between 40 K and 1.5 K. Due to the fact that the capacitance measurement requires vacuum in the cell, it is much more difficult to obtain a quenched state. Indeed after the first heating up to 40 K, it is necessary to simultaneously cut the heater and introduce a small amount of exchange gas in the cell to achieve a fast cooling rate of -2 K/s for the thermometer but perhaps not exactly for the samples. So the state we indicate as quenched is much more probably an intermediate one, as we will see later.

3. Results.

The thermal expansion in the whole range of temperature for the relaxed state is displayed in figure 1. The data have been obtained during a slow warming of the relaxed state obtained by the thermal treatment described above. There is already clear evidence that the anion ordering process, which takes place below 24 K, induces a contribution to the thermal expansion as indicated by the anomaly at 24 K. Furthermore figure 2 clearly shows the difference between quenched and relaxed state below the anion ordering temperature. In order to extract this anion ordering contribution from the data, we assume that above 24 K the thermal expansion is only due to phonons because quenched and relaxed states exhibit no difference at all in this region. Moreover, a power law analysis of the results in this region shows a lot of similarities with analogous data from the $(\text{TMTSF})_2\text{PF}_6$ salt [11]. Below 24 K estimate of the phonon contribution is more difficult : nevertheless, below 16 K we know that the anion ordering configuration, may be incomplete, is frozen [8] so that any thermal expansion must be due to the background : indeed, a T^4 law is well obeyed up to 12 K [12], as in $(\text{TMTSF})_2\text{PF}_6$ [11], and again we attribute it to the phonon contribution. Finally, to obtain this background over the whole range of temperature, it is easy to connect the two measured parts below 12 K and above 24 K by comparison with $(\text{TMTSF})_2\text{PF}_6$ [11]. We can now focus on the anion ordering contribution which is simply obtained from the raw data by subtract-

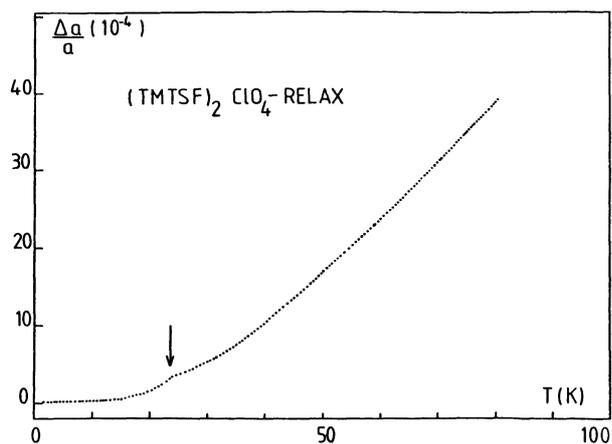


Fig. 1. — Thermal expansion of $(\text{TMTSF})_2\text{ClO}_4$ in the relaxed state. The arrow indicates the anion-ordering temperature.

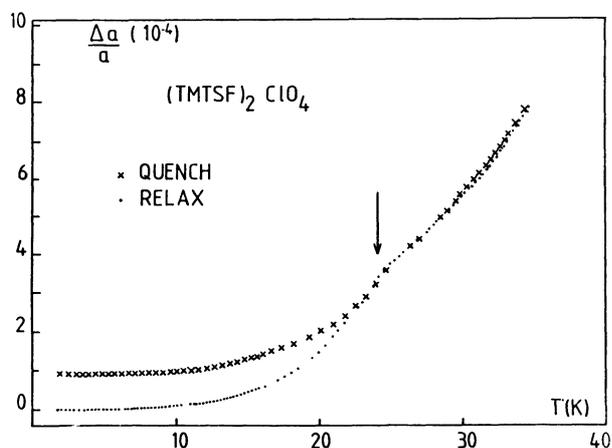


Fig. 2. — Low temperature thermal expansion of $(\text{TMTSF})_2\text{ClO}_4$ in relaxed and quenched states. The arrow indicate the anion-ordering temperature.

ing the estimated phonon background. This negative contribution is displayed in figure 3. We can immediately see that rapid cooling produces an intermediate state because one expects that a totally disordered state would not have such a thermal expansion term. Remembering that the superstructure X-ray peak intensity provides a measurement of the degree of order, we quote such data from reference [8] in figure 4. The similarity of figures 3 and 4 are striking and quantitatively we obtain from the two experiments the well-defined relation :

$$\left(\frac{\Delta a}{a}\right)_{a.o.} = -A \langle S_q \rangle^2, \quad (1)$$

where $\left(\frac{\Delta a}{a}\right)_{a.o.}$ is the anion ordering contribution to the relative thermal expansion along the a -axis and $\langle S_q \rangle^2$ is the square of the order parameter, propor-

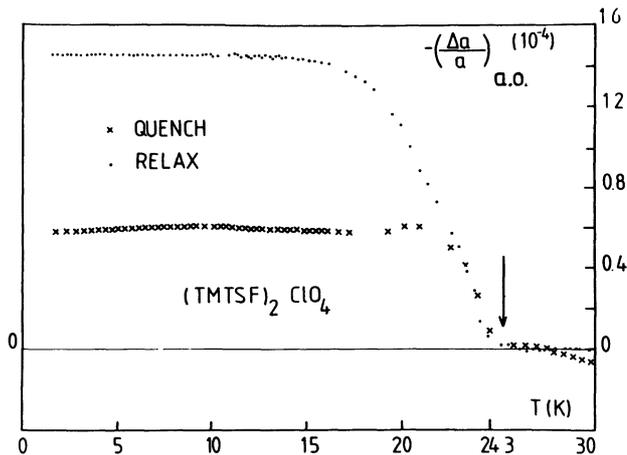


Fig. 3. — Anion ordering contribution to the thermal expansion of $(\text{TMTSF})_2\text{ClO}_4$ in relaxed and quenched states.

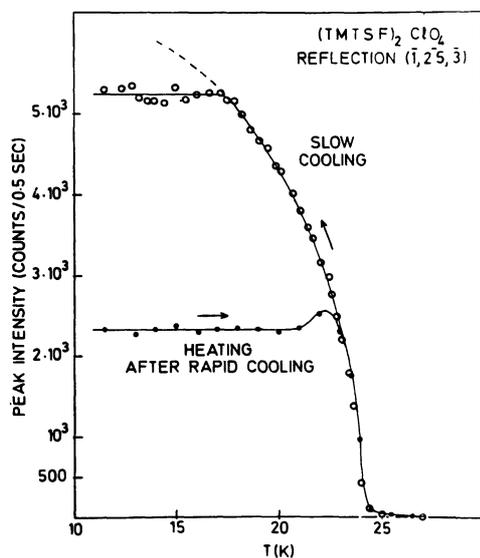


Fig. 4. — Superstructure peak intensity versus temperature ; from reference [8].

tional to the intensity of the superstructure peak at $q = (0, 1/2, 0)$. Note that the ratio between quenched and relaxed states, which were achieved in a very similar manner in the thermal expansion and X-ray peak intensity experiments, is also approximately the same, so the relation (1) is obeyed even in the intermediate state. Before coming to the theoretical considerations on the coupling between the lattice and the anion ordering phenomenon, let us remark that quenched and relaxed states differ at low temperature by about 10^{-4} for $\Delta a/a$. Assuming a compressibility of about 0.5 %/kbar as measured at room temperature in $(\text{TMTSF})_2\text{PF}_6$ [13], the equivalent pressure is about 200 bars. If we follow Kagoshima *et al.* [7], who claimed that superconductivity is related to the lattice

parameter value, the application of 200 bars or more to a quenched state at low temperature would induce the same superconductivity as is observed in the relaxed state. However conductivity experiments under pressure [14] show that the SDW instability of the electron gas is only suppressed for pressures above 3 kbars, and is then replaced by a metallic but not superconducting state. In conclusion, it appears that it is the anion ordering itself, and not its action on the lattice, which is important for the stabilization of superconductivity in $(\text{TMTSF})_2\text{ClO}_4$.

4. Phenomenological approach of the phase transition.

In order to understand, under T_c , the temperature behaviour of the extra-lattice contraction ε due to the anion order, we first perform a Landau-Ginzburg approach of the phase transition, which gives some insight into the coupling between the anion order parameter and ε . Then, in the whole temperature range, we treat the system as an assembly of Ising spins in interaction, that we solve by a one site and a cluster mean field approximation. (In this treatment, and in the absence of further information, we implicitly assume that the lattice deformations, due to the anion ordering process, along the three axes are decoupled). We present these two approaches successively and then discuss the microscopic nature of the couplings.

4.1 LANDAU-GINZBURG APPROACH. — Denoting $\langle S_q \rangle$ the anion order parameter, the free energy, in the absence of distortion ε is classically developed in the neighbourhood of T_c :

$$F_0 = \frac{a}{2} (T - T_c) \langle S_q \rangle^2 + \frac{b}{4} \langle S_q \rangle^4. \quad (2)$$

When a lattice distortion takes place at the transition, the free energy contains in addition an elastic term of the form $\frac{1}{2} C \varepsilon^2$ (where C is an effective elastic constant) and a term of coupling between $\langle S_q \rangle$ and ε which reads $K \varepsilon \langle S_q \rangle^2$ to lowest order, due to the symmetry of the high temperature phase :

$$F = \frac{a}{2} (T - T_c) \langle S_q \rangle^2 + \frac{b}{4} \langle S_q \rangle^4 + \frac{1}{2} C \varepsilon^2 + K \varepsilon \langle S_q \rangle^2. \quad (3)$$

The minimization of F leads to the simple relationship between ε and $\langle S_q \rangle$:

$$\varepsilon = -\frac{K}{C} \langle S_q \rangle^2. \quad (4)$$

A negative coupling constant K is associated to an expansion of the lattice while a contraction requires $K > 0$. Equation (4) seems in good agreement with the

experimental result ; however this approach is not valid in the temperature range where the order parameter has developed.

4.2 ISING MODEL ON A DEFORMABLE LATTICE.

4.2.1 The model. — The two orientations of the tetrahedral anions observed by X-ray measurements [15] in this family of Bechgaard salts can be represented by Ising $S = \pm 1$ spins ; such a representation was already used by Bruinsma and Emery [16] to describe the competition between the two orders characterized by the wave vectors $q_1 = (1/2, 1/2, 1/2)$ and $q_2 = (0, 1/2, 0)$. We postpone a more detailed description of this model at the end of the last section. Here we simply consider an assembly of N spins $S = \pm 1$ interacting with their nearest neighbours in three independent directions **a**, **b**, **c** :

$$\mathcal{H} = -J_1 \sum_{\langle ij \rangle_{\mathbf{a}}} S_i S_j - J_2 \sum_{\langle ij \rangle_{\mathbf{b}}} S_i S_j - J_3 \sum_{\langle ij \rangle_{\mathbf{c}}} S_i S_j. \quad (5)$$

The three exchange constants $J_1 J_2 J_3$ depend upon the lattice parameters and this provides a mechanism of coupling between the order parameter, namely $\langle S \rangle$ and the deformation ε . When the entropy of the phonon field is neglected, ε obeys a simple relationship deduced from the minimization of the free energy of the system with respect to the lattice parameter a :

$$C \varepsilon = -\frac{1}{N} \frac{\partial \langle \mathcal{H} \rangle}{\partial a}, \quad (6)$$

where $\langle \mathcal{H} \rangle$ is the mean value of the Hamiltonian \mathcal{H} evaluated on the ground state of the system ; as a consequence :

$$C \varepsilon = \frac{dJ_1}{da} \langle S_1 S_2 \rangle_{\mathbf{a}} + \frac{dJ_2}{da} \langle S_1 S_2 \rangle_{\mathbf{b}} + \frac{dJ_3}{da} \langle S_1 S_2 \rangle_{\mathbf{c}}. \quad (7)$$

ε is thus completely determined by the behaviour of the first neighbour spin-spin correlation functions $\langle S_1 S_2 \rangle$ along the three directions, together with the temperature dependence of the exchange coefficients : the latter has two contributions, one which comes from the usual T dependence of the parameters a , b , c (i.e. the background term), and the other which comes from the variation of ε . In a first step, we will neglect the first contribution, and come back to it later.

4.2.2 Standard mean field approximation. — In the usual mean field approximation, any correlation function $\langle S_1 S_2 \rangle$ is replaced by the square of the order parameter, and there is no short range correlations between fluctuations. It is thus immediately found that

ε is proportional to $\langle S \rangle^2$ at all temperatures, and for the q_2 order it reads :

$$\varepsilon = \frac{1}{C} \frac{dJ_{\text{eff}}}{da} \langle S \rangle^2, \quad (8)$$

with $J_{\text{eff}} = J_1 - J_2 + J_3$. The temperature dependence of $\langle S \rangle$ is obtained straightforwardly from the minimization of the spin part of the free energy, classically written :

$$F = NJ_{\text{eff}} \langle S \rangle^2 - NkT \text{Log} \left\{ 2 \cosh \frac{2J_{\text{eff}} \langle S \rangle}{kT} \right\}, \quad (9)$$

so that :

$$\langle S \rangle = \tanh \left[\frac{2J_{\text{eff}} \langle S \rangle}{kT} \right]. \quad (10)$$

We have solved numerically the coupled equations (8) and (10) for different values of the dimensionless parameter $\eta = \frac{1}{CJ_0} \left(\frac{dJ_{\text{eff}}}{da} \right)^2$, limiting the development of J_{eff} to the two first terms :

$$J_{\text{eff}} = J_0 + \frac{dJ_{\text{eff}}}{da} \cdot \varepsilon. \quad (11)$$

Results are displayed in figure 5 ; the change of the nature of the transition from second to first order occurs at $\eta = 1/3$. η is the only parameter of the problem once $\langle S \rangle$ is plotted as a function of T/T_c . We have searched the best agreement with experimental results only for T in the range $[0.6 T_c, T_c]$ because we are not sure that, within our experimental procedure a complete relaxed state is achieved at low temperature ; in this case, the order parameter would

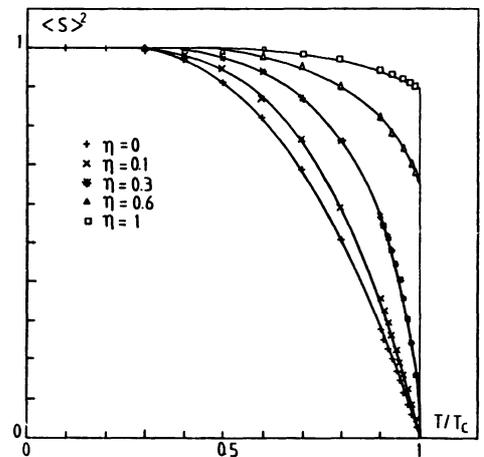


Fig. 5. — Thermal variation of the ordering parameter, for different coupling constants η in the one site mean-field approximation.

saturate at a value less than one ; and indeed Ribault *et al.* [17] observed that the Hall effect remains very sensitive to the cooling rate, even for rates much smaller than the standard value of -0.2 K/min. The best fit of figure 6 was obtained for $\eta = 0.1$. But in doing so we have neglected the usual thermal variation of J_0 which acts in the same direction as ε , i.e. helping $\langle S \rangle$ to saturate more quickly. The following qualitative estimates are obtained by taking $\eta \sim 0.05$.

First, the value of the transition temperature $T_c = 2J_0 = 24$ K gives an effective exchange coupling $J_0 = 12$ K. From the ratio between the measured anion ordering thermal expansion and the order parameter, we deduce $\frac{1}{C} \left| \frac{dJ_{\text{eff}}}{da} \right| = 1.43 \times 10^{-3} \text{ \AA}$ (Eq. (8)). The two quantities C and $\left| \frac{dJ_{\text{eff}}}{da} \right|$ can then be evaluated separately using the above estimation of $\eta = 0.05$. We obtain $\left| \frac{dJ_{\text{eff}}}{da} \right| = 860 \text{ K.\AA}^{-1}$ and $C = 6 \times 10^5 \text{ K.\AA}^{-2}$ but it is clear from the definition of η that a ten times smaller value of η would reduce in the same proportion both $\left| \frac{dJ_{\text{eff}}}{da} \right|$ and C while their ratio would remain well defined and independent of η . In addition, the total change of J_{eff} between T_c and 0 K is approximately $\Delta J_{\text{eff}} \sim 0.9$ K. Unfortunately, the above estimate of $\left(\frac{dJ_{\text{eff}}}{da} \right)$ and C seem to be much larger than the expected ones. Indeed, C corresponds to an elastic constant per unit volume of about $10^{13} \text{ erg cm}^{-3}$ which is two orders of magnitude larger than in most solids, and particularly TTF-TCNQ [18] ; on the other hand, $\left| \frac{dJ_{\text{eff}}}{da} \right|$ leads to a prediction of $\frac{dT_c}{dp} \sim 70 \text{ K.kbar}^{-1}$ (note that this estimation is really a gross one because

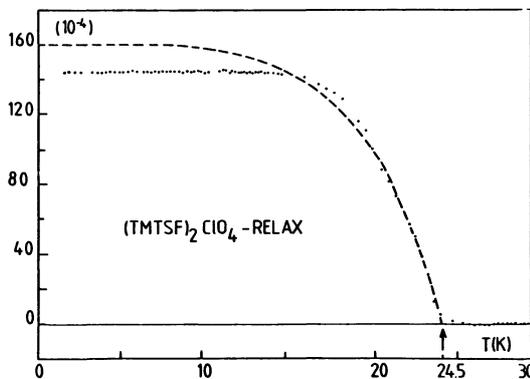


Fig. 6. — Comparison between the experimental anion ordering contribution to the thermal expansion in the relaxed state (...) and the square of the ordering parameter in the one site mean-field approximation ($\eta = 0.1$) (---).

of the large anisotropy of the system, but we are only concerned here with the orders of magnitude) which is 5 to 10 times greater than the measured one [19]. These two points suggest that a realistic coupling constant η should be smaller than the one deduced from the mean-field treatment. Part of this discrepancy can be eliminated by performing a treatment within a Bethe approximation.

4.2.3 Bethe approximation. — It is possible to refine the previous treatment and take into account correctly short range correlations inside a cluster. We have chosen the cluster built with a central spin and its six nearest neighbours ; the details of the calculation are standard and can be found elsewhere [20]. The three coupling constants J_1, J_2, J_3 play independent roles and, in order to show how the preceding results are modified, we have chosen $J_1 = -J_2 = J_3 = J$. The same dimensionless parameter η occurs and the change from second to first order occurs also close to 0.3.

At a given η , ε grows more rapidly to saturation than in the simple mean field approximation, and it has a non zero value above T_c due to the existence of short range correlations as shown in figure 7. As a conse-

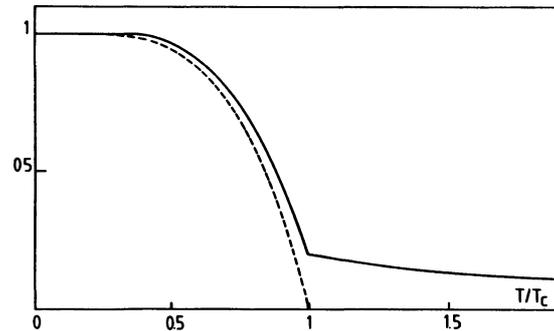


Fig. 7. — Comparison between the square of the ordering parameter $\langle S \rangle^2$ evaluated in the one site mean-field approximation ($\eta = 0.1$) (---) and the correlation function $\langle S_0 S_1 \rangle$ evaluated in the Bethe approximation ($\eta = 0$) (solid line).

quence, comparison with experimental results is difficult, because the way we have extracted the anion ordering contribution to the thermal expansion implicitly neglects the fluctuations above T_c and it is also difficult to discriminate between the behaviour of $\langle S \rangle^2$ and $\langle S_1 S_2 \rangle$ once the fluctuations have been subtracted. However the best value of η is appreciably lower than in the one site mean field approximation : $0 \leq \eta \leq 0.01$ reducing the deduced values of $\frac{dJ_{\text{eff}}}{da}$ and C to reasonable ones.

5. Discussion.

We have been able to get orders of magnitude of the parameters entering our phenomenological model : the transition temperature gives $J_{\text{eff}} \sim 12$ K, the sign of the lattice distortion ε indicates that $dJ_{\text{eff}}/da < 0$ while an estimate of the dimensionless lattice anion coupling $\eta \leq 0.01$ seems to be reasonable to yield correct values of elastic constants and of the pressure variation of the transition temperature. Several points are worth discussing :

i) within our approximations the value of η that fits the data always lies in the range where the transition is second order. Nevertheless we know that the mean field approximations break down close to T_c , and with more elaborate arguments than ours, Bergmann and Halperin [21] have proved that, whatever the value of the spin lattice coupling η , the transition of a system of Ising spins on a deformable lattice, is always first order : there is a jump of the order parameter at the transition, but this jump becomes infinitely small as η goes to zero. It is clear that in thermal expansion measurements, as well as in X-ray measurements, which are more related to the square of the order parameter, a small jump could not show up clearly. Research of the presence of a latent heat at the transition by specific heat measurements would help concluding on this point.

ii) The presence of an extracontraction at the transition indicates that the effective exchange constant decreases when the lattice expands : $dJ_{\text{eff}}/da < 0$. In this model, J_1, J_2 and J_3 represent half the difference in energy between configurations with parallel or antiparallel spins along a given direction. If the first neighbour interaction is of a direct Coulomb type one may expect $\left| dJ_1/da \right| \gg \left| \frac{dJ_2}{da} \right|, \left| \frac{dJ_3}{da} \right|$ and with $J_1 > 0$ (necessary to obtain $q_a = 0$) one indeed gets $dJ_1/da < 0$. On the other hand, $dJ_1/da < 0$ could also be compatible with a Coulomb interaction between anions mediated by the metallic polarizability $\chi(q)$ of the electronic gas ; since $\chi(q)$ varies as the inverse of the hopping integral $\beta \sim e^{-pa}$ and is the more rapidly varying quantity with a , one would get $\frac{dJ_1}{da} \sim -p \left| J_1 \right|$; but in this case J_1 would be negative and no ferromagnetic order along a could exist.

iii) One has to note that the above result would be changed if some of the interactions between anions were frustrated. Such a model was developed by

Bruinsma and Emery [16] : they introduced three antiferromagnetic interactions J'_1, J'_2 and J'_3 in the diagonal plane of the triclinic lattice, and this leads to a frustrated ground state as described in the topologically equivalent 2D model of Ising spins on a triangular lattice. It is interesting to study the predictions of this model as concerns lattice expansion at the transition. By similar arguments as previously, one would find that the J'_1 and J'_3 interactions are the most dependent upon a , so that the distortion in this case would be :

$$\varepsilon = \frac{\langle S \rangle^2}{C} \left(\frac{dJ'_1}{da} - 2 \frac{dJ'_3}{da} \right).$$

J'_1 and J'_3 are assumed negative in the model due to a mediation of the interactions by the electron gas ; assuming again that $\chi(q)$ is the most rapidly varying quantity with a , one gets $C\varepsilon \sim p \langle S^2 \rangle (J'_1 - 2J'_3)$. A contraction $\varepsilon < 0$ would thus be associated with the inequality $J'_1 < 2J'_3$. But J'_3 was assumed in the model to be the largest of the three interactions in absolute value so that spins in the diagonal directions of the lattice cell would always be antiparallel $J'_3 \ll J'_1$. From our point of view, it is difficult to reconcile the two inequalities $J'_3 \ll J'_1$ and $J'_1 < 2J'_3$ while our model, which postulates a positive interaction J'_1 along the a direction does not present such kind of difficulties. Our model is also fitted to a description of isotropic fluctuations above T_c but it does not pretend to predict which characteristic wave vector will be stabilized at the transition.

6. Conclusion.

We have measured the thermal expansion of $(\text{TMTSF})_2\text{ClO}_4$ between 1.5 and 100 K. Below 24.3 K which is the anion ordering temperature, a negative contribution develops progressively ; this can be associated unambiguously with the anion ordering phenomenon. Identifying the background thermal expansion with a phonon term, we have extracted the anion ordering contribution which turns out to be exactly proportional to the superstructure peak intensity. Finally we give a phenomenological treatment of the anion ordering in terms of Ising spins weakly coupled to the lattice. From this discussion the main experimental features can be simply understood.

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