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Neutron Diffraction Evidence of Structural Phase Transitions at 150 K and 90 K in Trimethylammonium-Iodide-7,7,8,8, Tetracyano p-Quinodimethane (TMA⁺, TCNQ^{2/3-}, (I⁻)_{3/4})

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Neutron Diffraction Evidence of Structural Phase Transitions at 150 K and 90 K in Trimethylammonium-Iodide-7,7,8,8, Tetracyano *p*-Quinodimethane (TMA^+ , $\text{TCNQ}^{2/3-}$, $(\text{I}_3^-)_{1/3}$)

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Neutron photographs have been obtained down to 7 K and additional neutron data have been recorded on a 4-circle diffractometer for single crystals of the title compound. They show that below 150–160 K weak satellites appear at $\frac{1}{3}b^*$ of the main TMA–TCNQ lattice reflections. This is consistent with a charge transfer of $0.66e$ and suggest that the transition is of a “metal-insulator” type rather than a “semiconductor-semiconductor” type. Below 80–90 K a second transition occurs which is associated with a splitting of reflections along $\sim c^*$. The main lattice is then triclinic and the satellites are still present.

INTRODUCTION

The reaction product $\text{TMA}^+ \text{TCNQ}^{2/3-} (\text{I}_3^-)^{1/3}$ of Trimethylammonium iodide and Tetracyanoquinodimethane (TCNQ) was the first example¹ of a ternary quasi one-dimensional molecular conductor with one cation and two different

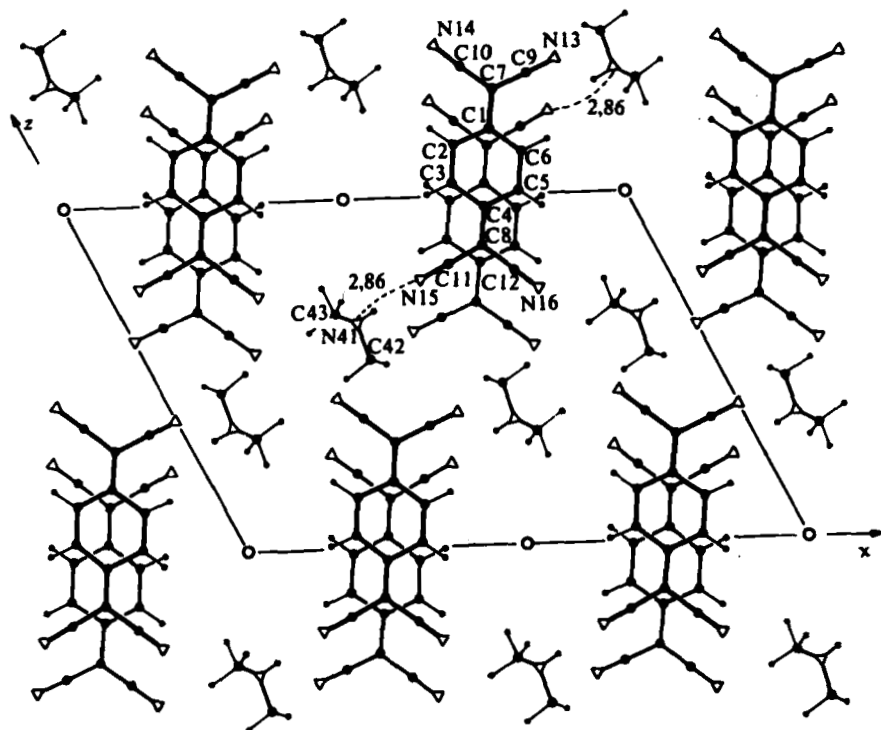


FIGURE 1 Projection of the structure of TMA.TCNQ.I along b (from Ref. 16). O: iodine; Δ : nitrogen; \bullet : carbon; \cdot : hydrogen.

segregated acceptor chains (Figure 1) presumably only one of which, TCNQ, is conducting. The X-ray diffraction pattern may be explained by considering two interpenetrating lattices: the main (A) lattice being due to TMA and TCNQ while the second (B) lattice is of the iodine atoms. Delhaes *et al.* (1976)² and Filhol (1979)³ have described samples with the (B) lattice "disordered" (diffuse planes h , $(n/3)k$, l modulated along a^*) while Coppens *et al.* (1980)⁴ have observed crystals with an "ordered" (B) lattice. In fact even in this latter case a certain amount of disorder still remains, as Bragg spots are superimposed on diffuse lines on X-ray photographs⁵ with long time exposures. For "disordered" samples of TMA.TCNQ.I the electrical conductivity (σ) along the chain axis, always decreases with temperature, while "ordered" samples show a broad maximum of conductivity at $T_M = 240$ K.⁶

Both kinds of samples exhibit an abrupt change in temperature-dependent conductivity at temperatures in the range 150–160 K. In addition anomalies at about 89 K have been observed in the Young modulus and microwave dielectric constant.⁷ The 89 K anomaly has been attributed to the freeze out of methyl-groups while the 150–160 K anomaly and the σ maximum at $T_M = 240$ K are

still subjects of controversy. In fact Epstein *et al.* (1977/1978)^{8,9} have proposed a model for conduction which postulates a semiconducting state for TMA.TCNQ.I at all temperatures despite "metal-like" conductivity above T_M . In that case the 150–160 K anomaly would be attributed to a "semiconductor-semiconductor" transition. In contrast, Coulon *et al.* (1981)¹⁰ have recently attributed the physical properties of the high temperature state of this kind of ternary iodine-containing 1D conductors to strong electron-electron interactions. In this description the 150–160 K anomaly is associated with a "metal-insulator" transition (Peierls distortion). In order to shed light on the structural nature of the possible transitions at 150–160 K and 80–90 K we have performed a neutron diffraction study of TMA.TCNQ.I crystals down to 7 K. The first results of this work are described here and the results of an additional X-ray and electron diffraction¹¹ study at low temperature will be published later.

EXPERIMENTAL

Samples

Three kinds of samples were used. Compound (I) was hydrogenated; compound (II) synthesized from TMA (d_{10}) gas was fully deuterated; compound (III) synthesized from TMA (d_9)H⁺·Cl[−], was deuterated except for the quaternary hydrogen of the cation. Single crystals of needle shape were grown from acetonitrile; the solution being slowly cooled from $\sim 40^\circ\text{C}$. Samples were characterized by means of X-ray diffraction. They all show diffraction patterns of the "ordered" type (Bragg spots) with nevertheless a slightly larger amount of disorder (diffuse lines) than for the "ordered" samples of Coppens *et al.* (1980). The d.c. electrical conductivity (σ) of crystals of compounds (I) and (II) have been measured by the classical four-probe technique. For the few samples investigated, the conductivity at high temperature shows a saturation rather than a real maximum (Figure 2) and the transition at ~ 150 K is clearly visible on the plot of $D(T) = -\partial(\ln \sigma)/\partial(1/T)$.

Neutron photographs

The instrument used was the normal-beam Weissenberg camera "D12A"¹² located at the end of a thermal neutron guide tube at the High Flux Reactor (HFR) of the Institute Laue-Langevin at Grenoble. The incident beam, monochromatized by a flat graphite monochromator, had a flux of about $2 \cdot 10^6$ n/cm²/sec at the sample position and a wavelength of 1.489 Å. "D12A" was equipped with a reservoir-type helium cryostat with a thin walled aluminium tail. Oscillating radial Soller slits¹³ placed between the film and the external shield of the cryostat were used to eliminate the lines produced by the alumin-

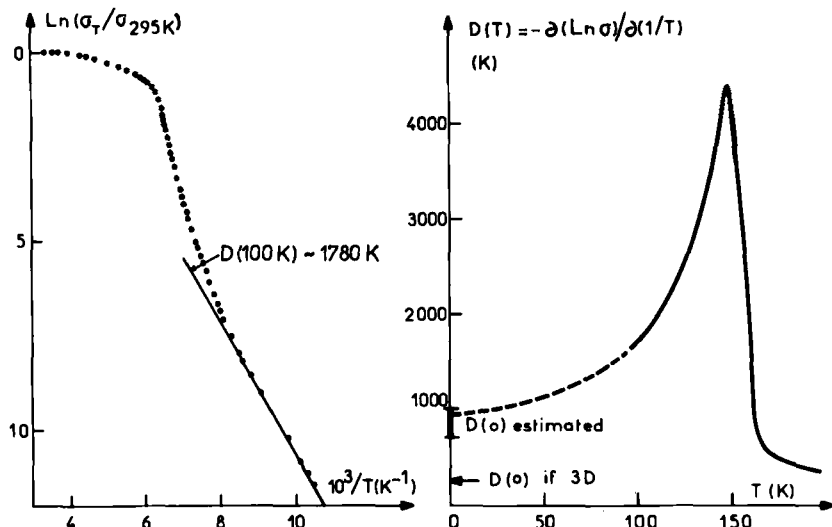


FIGURE 2 Temperature dependence of the longitudinal d.c. conductivity (σ) of a sample of TMA.TCNQ.I. The sample was a single crystal of compound (I) measured by means of the classical four-probe technique (platinum-paste contacts). The observed value of σ 300K is $\sim 30 \Omega^{-1} \text{cm}^{-1}$. The derivative $D(T) = -\partial(\ln \sigma)/\partial(1/T)$ indicates that the temperature T_1 of the first transition is ~ 150 K.

ium cryostat walls. The “fixed crystal—fixed film” technique was used to obtain neutron photographs (Figure 3) from crystals ($\sim 0.1 \text{ mm}^3$) oriented with b^* parallel to the camera axis and either a^* or c^* parallel to the beam axis. The measurements were performed at 300 K, 165 K, 115 K, 80 K and 7 K for the compound (II) and at 165 K, 145 K, 100 K and 60 K for the compound (III). The mean exposure time was about 60 hours.

4-Circle neutron diffractometer

Measurements were then performed on the 4-circle neutron diffractometer “D8” located at the end of a thermal neutron beam tube at the HFR. The neutron beam had flux of about $5.4 \cdot 10^7 \text{ n/cm}^2/\text{sec}$ at the sample position and a wavelength of $1.2635(2) \text{ \AA}$ (Cu(200) monochromator in transmission). The cryogenic device was a closed-cycle one-stage cryorefrigerator.¹⁴ The sample was an hydrogenated single crystal (I) with volume of about 0.5 mm^3 . Small diameter beam limiters were set for both the incident and diffracted beam to obtain a low background ($< 1 \text{ count/sec}$). The localization of peaks was performed by means of scans along reciprocal rows while intensities were recorded with $\theta/2\theta$ scans.

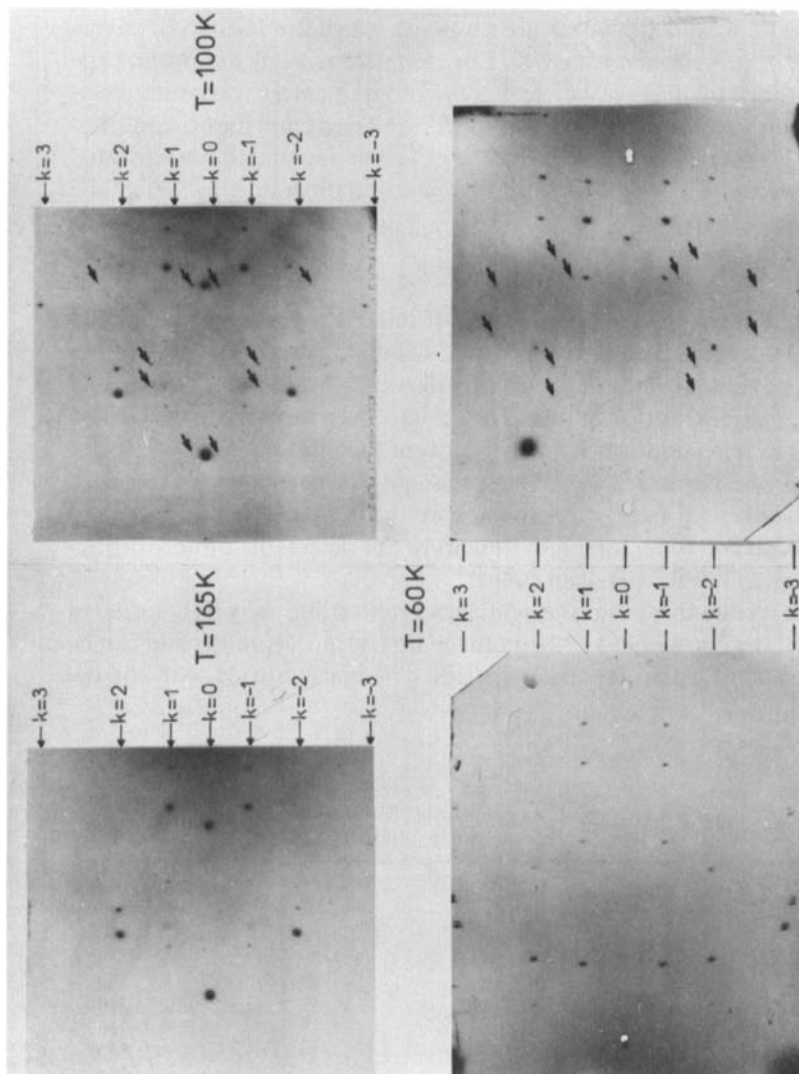


FIGURE 3 Neutron photographs of TMA.TCNQ.I at 165 K, 100 K and 60 K. They were obtained by the "fixed-crystal, fixed-film" technique from a single crystal of sample II oriented with a^* parallel to the beam axis. The satellites are indicated by arrows. For 165 K and 100 K, only the most significant parts of the films are shown. For 60 K, the right and left parts of the pattern are given. On this latter, split reflexions are mainly visible on the left side because the splitting of a reflexion $h k \xi$ is observed only if the corresponding $[hk\xi]$ reciprocal row is tangent to the Ewald circle.

RESULTS

Neutron photographs (Figure 3) taken at temperatures above 150 K show only the Bragg spots of the main (A) lattice while the pattern of the (B) iodine lattice is not visible because iodine atoms are weak neutron scatterers.

Below 150 K, weak extra spots are observed which are located on layers $nk \pm \frac{1}{3}$ and are present down to 7 K. For technical reasons and beam time shortage we failed to obtain a Weissenberg photograph of a superlattice reciprocal plane and thus the superlattice spots were indexed from the existing photographs by means of an Ewald construction. This shows that they are located on a (A') lattice with $a' = a$, $b' = 3b$ and $c' = c$ and the following systematic extinctions are observed:

$$h' = 2n \text{ if } k' = 2nk \pm \frac{1}{3} \text{ and } h' = 2n + 1 \text{ if } k' = (2n + 1)k \pm \frac{1}{3}.$$

At 4-circle diffractometer examination of limited zones of the reciprocal lattice at 100 K were performed by means of scans along reciprocal rows. The Miller indices (Table I) of the observed superlattice reflections (s.r.) agree fully with the systematic extinctions above. The s.r. have been localized in the vicinity of strong main reflections (m.r.) with a ratio of the intensities $I(\text{s.r.})/I(\text{m.r.})$ which is, in the best case (s.r.: 11, $\frac{4}{3}$, 5) less than $\frac{8}{100}$. The particular s.r. recorded photographically with an ~ 3 day exposure were only associated with small to medium m.r. structure factors, and thus were not detectable on the diffractometer using ≤ 100 sec per scan point.

These first results show that the neutron s.r. reflections may be considered as being satellites located at $\frac{1}{3}b^*$ from the main (A) lattice points and can be neither attributed to the (B) lattice (period $\frac{3}{2}b$) nor confused with the (B)

TABLE I

Miller indices (hkl) and relative peak intensities (I_r) of the superlattice reflections observed at 100 K on a neutron 4-circle diffractometer.

h	k	l	I_r	h	k	l	I_r
10	$-1/3$	2	360	-9	$4/3$	-6	450
-4	$-1/3$	-4	260	10	$5/3$	2	350
10	$1/3$	2	350	-6	$7/3$	-2	380
8	$1/3$	5	300	-6	$7/3$	-3	320
-6	$1/3$	-2	510	-2	$7/3$	-4	340
-2	$1/3$	-4	270	-6	$7/3$	-4	520
-4	$1/3$	-4	280	5	$8/3$	1	320
-6	$1/3$	-4	400	11	$8/3$	3	430
11	$2/3$	5	620	11	$8/3$	4	310
11	$4/3$	5	1000	9	$8/3$	5	300
13	$4/3$	5	340	11	$8/3$	5	720
				-7	$8/3$	-4	300

lattice reflections. In fact these latter—observed only by means of X-ray and electron diffraction¹¹—which are also present on the $(2n + 1)k \pm \frac{1}{2}$ layers—are absent on the $2nk \pm \frac{1}{2}$ layers and have constant intensities in the temperature range 100 K–300 K.³

The photographs taken below 80–90 K show a change in the aspect of the main and superlattice spots and some of them appear to be split. An Ewald construction indicates that the splitting of the reciprocal points occurs in a direction which is c^* or close to it.

The intensity change of two superlattice reflections (Figure 4) and the change in intensity and full width at half height of four mainlattice reflections (Figure 5) were recorded for some increasing temperatures in the range 70–180 K. These results confirm the existence of both the $T_i^I = 150$ –160 K and the $T_i^{II} = 80$ –90 K phase transitions.

DISCUSSION

The 150–160 K phase transition is continuous (2nd order) or only weakly first order, as shown by Figure 4, and is characterized by the appearance of superlattice reflections. The periodicity of these satellites along the column axis, namely $b' = 3b$, is consistent with a charge transfer of $0.66 e$ per TCNQ molecule as suggested by the stoichiometry of TMA.TCNQ.I.⁶ This supports the

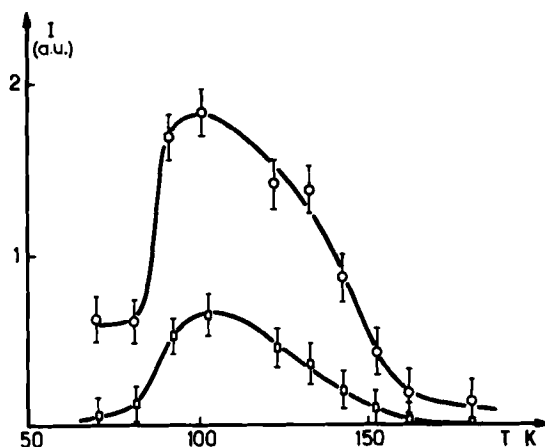


FIGURE 4 Intensities of two superlattice reflections as a function of temperature. O: $11, \frac{4}{3}, 5$; \square : $6, \frac{7}{3}, 4$. The lines are only guides for the eye. The drop in intensity below 90 K may be due to the fact that the detector was not set properly to take into account the additional $\frac{1}{3}a^*$ component of the q -vector of the satellite reflexions. This extra component has been discovered only recently, by means of electron diffraction.¹¹

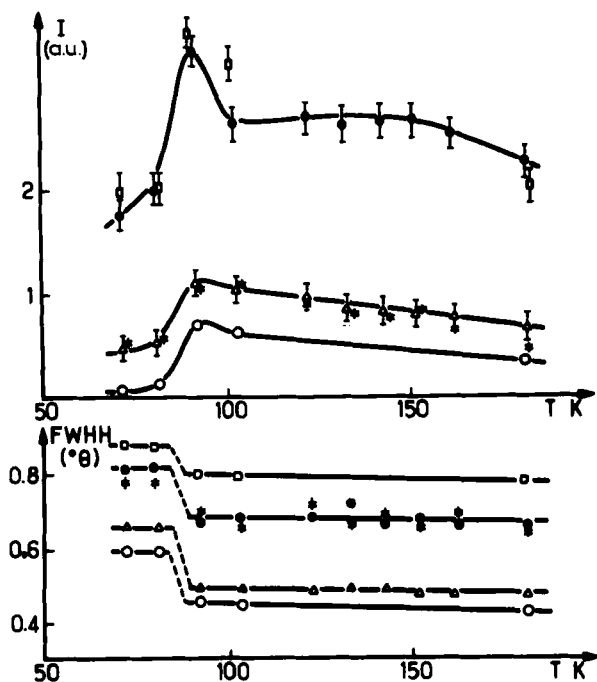


FIGURE 5 Intensity (I) and full-width at half-height (FWHH) of five main lattice reflections as a function of temperature ●: 11, 1, 5; □: 11, 3, 5; △: 6, 2, 4; ○: 4, 0, 4; *: 16, 0, 0. " I " is given with arbitrary units (a.u.) and "FWHH" is given in Bragg angle θ (degrees). The lines are only guides for the eye.

work of Coulon *et al.* (1981)¹⁰ who described this phase transition as a Peierls distortion of the lattice associated with a condensation of the charge density waves.

In fact, such an instability has already been observed for some organic metals and has been the subject of detailed studies in the case of TTF.TCNQ (tetrathiafulvalenium 7,7,8,8 tetracyano *p*-quinodimethane) (for a review see Ref. 15). It is thus well established that, at the metal insulator phase transition (T_{MI}), weak extra spots appear which have a wave vector twice the Fermi wave vector k_F .

A final proof for such an instability in TMA.TCNQ.I would be the occurrence of 1D precursors above T_I^1 but these latter may be extremely weak if the compound is so three-dimensional that the 1D fluctuations only occurs over a very small temperature range.

The dimensionality of TMA, TCNQ.I has therefore been estimated from the d.c. conductivity data. The derivative $D(T) = -\partial(\ln \sigma)/\partial(1/T)$ extrapolated to $T = 0$ K (Figure 2) gives a semiconducting gap $D(0)$ of 700 K–1000 K. Thus the magnitude of $\Delta = D(0)/kT_I^1$ which is 1.77 in the 3D case¹⁶ and in-

creases steadily with the 1D character of the compound—is in the range 5–7 for TMA.TCNQ.I. This latter value may be compared to $\Delta \sim 4$ observed for TTF.TCNQ.¹⁷ In other words, in spite of the uncertainty of the extrapolation from 95 K to 0 K, this result—as well as the results of Coulon *et al.* (1981)¹⁰—are in favor of a strong electronic 1D character for TMA.TCNQ.I and thus 1D precursors should be observable.

In the case of organic conductors, the X-ray observation of 1D precursors normally requires cameras with extremely low background, while, up to now, TMA.TCNQ.I has only been studied with standard X-ray cameras. However, it should be noticed that a very weak diffuse line located at $\frac{2}{3}b^*$ —i.e. on the layer having the strongest satellites below T_1^1 —has been observed at ~ 160 K. The nature of this extra diffuse line (disorder or 1D precursor?) is not yet unambiguously established and further measurements including a study of the temperature dependence of the intensity are planned.

Assuming the transition will be proven to be truly of the Peierls type in TMA.TCNQ.I, we may take the discussion further. In fact the electronic properties of TMA.TCNQ.I (one-conducting chain, $\frac{1}{3}$ filled electronic band) are easier to understand than those of compounds with double conducting chains such as TTF.TCNQ.

The position of the spots in the a^*, c^* planes, namely $a'^* = a^*, c'^* = c^*$ gives the relative phases (Figure 6) for the charge density waves borne by the various TCNQ chains (the origin of the phases is chosen on the central chain). In other words, along the axis x, y, z of the cell (Figure 6), the new components of the softmode q_o are:

$$q_o(x) = \frac{\pi}{dx} \quad q_o(y) = 2k_F \quad q_o(z) = \frac{2\pi}{dz}$$

with dx and dz the distance between two neighboring chains in the x and z directions respectively. If we also consider the direction “ δ ” of the transverse interaction, between charge density waves, labelled “3” on Figure 6, then:

$$q_\delta(\delta) = \frac{\pi}{d(\delta)}$$

a result which is consistent with the calculations of Coulon *et al.* (1981)¹⁰ in which the transverse interactions between charge density waves are assumed to have a coulombic origin. The high value of $T_{MI} \sim 150$ K observed for TMA.TCNQ.I is then associated with the strong interchain coupling “3”.

These latter authors also point out the role of the I_3 chains which act as an external potential whose effect on T_{MI} is a function of the iodine sublattice. This may be further discussed in the case of TMA.TCNQ.I where the three dimensional ordering of the iodine sublattice has been investigated.^{3,4}

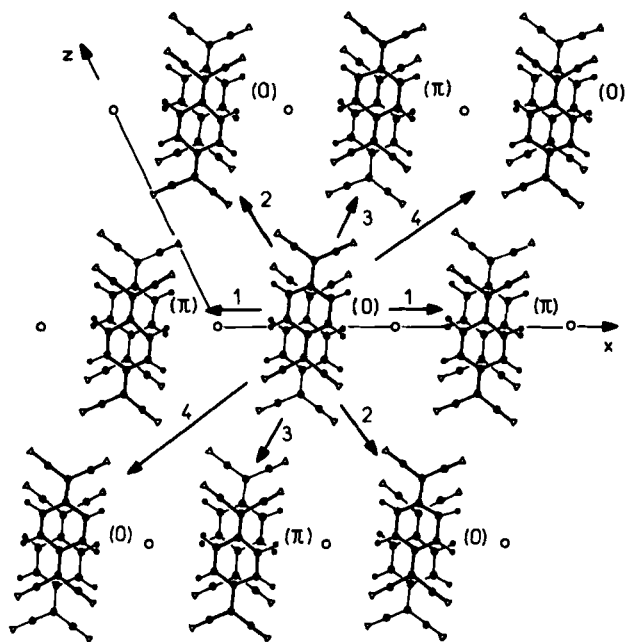


FIGURE 6 Relative phases of the charge density waves in TMA.TCNQ.I. The arrow indicate the various interactions between the charge density waves. It has been shown⁹ that the interaction numbered "3" is the dominating one.

In the case of a strong transverse disorder in the iodine sublattice, the interaction between the charge density waves and the I_3 chains averages to a very small value and is thus negligible. In the case of ordered samples, as a first approximation, the iodine external potential may be described by a charge modulation:

$$\delta\rho_I = \rho_I \cos q_I r$$

with

$$q_I(x) = \frac{2}{3} \frac{\pi}{dx} \quad q_I(y) = 2k_F \quad q_I(\delta) = \frac{2\pi}{d\delta}$$

Although its y component is the same as $q_o(y)$, the two others are different, thus preventing any linear coupling with the charge density waves. This coupling is then described by high order terms only and is again not a relevant parameter for the description of the metal-insulator phase transition in the case of TMA.TCNQ.I. The situation may be different with other kinds of I_3 ordering and a systematic comparison of a series of iodine-containing organic conductors is presently being carried out.

The second phase transition ($T_{\text{I}}^{\text{II}} = 80\text{--}90\text{ K}$) is best characterized by a splitting (Δc^*) of the main reflections, a splitting which occurs in the c^* directions—or at least close to it—with its magnitude a function of b^* (Figure 7). The corresponding distortion is a change in lattice parameters (mainly α^* to $\sim 89.4^\circ$ instead of 90° above 90 K), the new cell being triclinic. Below T_{I}^{II} the crystal packing may be then described as follows: the TCNQ columns and the iodine chains are still parallel to b but the TCNQ planes are no longer perpendicular to this axis.

The neutron photographs obtained at 60 K and 7 K show that the satellites are still present below 80 K . At 7 K they are relatively intense and split along c^* . Therefore, the drop in the intensity of the satellites: $11, \frac{4}{3}, 5$ and $\bar{6}, \frac{7}{3}, 4$ (Figure 4) which was observed at the transition may be due to a non-monotonic thermal dependence of the order parameter or to a relative change in structure factors. An other explanation may be that the s.r. were not correctly

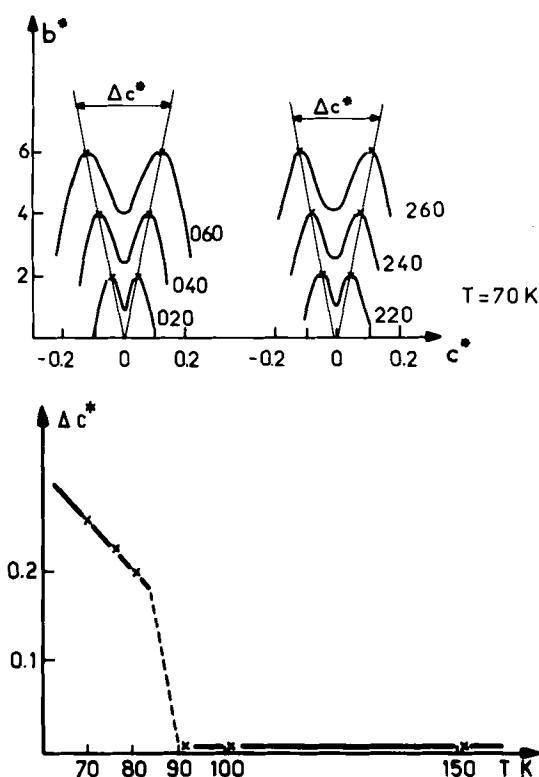


FIGURE 7 Splitting (Δc^*) of some reflections as a function of b^* and as a function of temperature. 7a: splitting Δc^* at 70 K as a function of b^* ; 7b: Δc^* of reflection 060 as a function of temperature.

centered on the detector because below T_t^{II} their wave vector have a small unforseen a^* component as recently observed by electron diffraction at 70 K.¹¹

Our results (Figure 5) are in agreement with those of Brill *et al.* (1980)⁷ who showed that this transition, associated with the freeze out of the TMA rotation, is 1st order. A surprising feature of the low temperature phase is that the twinning of the lattice (A) is such that the reciprocal points are split along c^* while at room temperature the iodine (B) lattice often appears to be twinned with the reflections split along a^* . This suggests that the crystal structure of TMA.TCNQ.I should be described in an alternative way.¹¹

To conclude we may recall that Filhol and Gaultier (1980)¹⁸ determined the neutron structure of TMA.TCNQ.I at 80 K without finding the T_t^{II} transition. In fact in the course of the experiment, broad or split line profiles were observed just after a sudden drop in temperature and the unsplit line profiles could not be recovered by heating back to 80 K (hysteresis). These facts were wrongly attributed to a crystal fracture. In other words, this "80 K structure" is the middle phase thus suggesting that the stated temperature of 80 K ($< T_t^{II} \sim 89 \text{ K}^7$) is underestimated† and/or that the temperature T_t^{II} is slightly sample (amount of disorder ?) dependent.

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References

1. A. Cougrand, S. Flandrois, P. Delhaes, P. Dupuis, D. Chasseau, J. Gaultier and J. L. Miane, *Mol. Cryst. Liq. Cryst.*, **32**, 165 (1976).
2. P. Delhaes, A. Cougrand, S. Flandrois, D. Chasseau, J. Gaultier, C. Hauw and P. Dupuis, *Proceeding of Conference on Organic Conductors and Semiconductors*, Siófok, Hungary, p. 493–498 (1976).
3. A. Filhol, M. Rovira, C. Hauw, J. Gaultier, D. Chasseau and P. Dupuis, *Acta cryst.*, **B35**, 1652–1660 (1980).
4. P. Coppens, P. Leung, K. E. Murphy, P. R. v. Tilborg, A. J. Epstein and J. S. Miller, *Mol. Cryst. Liq. Cryst.*, **61**, 1–6 (1980).
5. A. J. Epstein, J. P. Pouget, C. Coulon, unpublished results of an experiment performed at Orsay (France) (1981).

† A later recalibration in the vicinity of 80 K of the temperature sensor of the cryostat used by these authors gave temperatures 3 to 4 K higher.

6. M. A. Abkowitz, A. J. Epstein, C. H. Griffiths, J. S. Miller and M. L. Slade, *J. Am. Chem. Soc.*, **99**, 5304–5308 (1977).
7. J. W. Brill, A. J. Epstein and J. S. Miller, *Phys. Rev. B*, **20**, 681–685 (1979).
8. A. J. Epstein, E. M. Conwell, D. J. Sandman and J. S. Miller, *Solid State Commun.*, **23**, 355–357 (1977).
9. A. J. Epstein, E. M. Conwell and J. S. Miller, *Ann. N.Y. Acad. Sci.*, **313**, 183 (1978).
10. C. Coulon, S. Flandrois, P. Delhaes, C. Hauw and P. Dupuis, *Phys. Rev. B*, **23**, 2850–2859 (1981).
11. R. Ayroles, A. Filhol, B. Gallois, J. Gaultier, J. Granier and C. Hauw (1981) to be published. R. Ayroles, C. Roucau, J. Granier: Poster Session C-P22; J. Gaultier, A. Filhol, B. Gallois, D. Chasseau, C. Hauw: Poster Session C-P36—“Congrès de la Société Française de Physique” 26/6–4/7 1981, Clermont-Ferrand (France).
12. D. Hohlwein and A. F. Wright, *J. Appl. Cryst.*, **14**, 82–84 (1981).
13. A. F. Wright, M. Berneron and S. P. Heathman, *Nuclear Instruments and Methods* **180**, 655–658 (1981).
14. J. R. Allibon, A. Filhol, M. S. Lehmann, S. A. Mason and P. Simms, *J. Apply. Cryst.*, **14**, 326–328 (1981).
15. R. Comès, “Chemistry and Physics of One-Dimensional Metals” p. 315–339, Editor H. J. Keller, Plenum Press New York and London (1977).
16. J. A. Toombs, *Phys. Reports*, **C40**, 181–240 (1978).
17. Y. Tomkiewicz, A. R. Taranko and J. B. Torrance, *Phys. Rev. B*, **15**, 1017–1023 (1977).
18. A. Filhol and J. Gaultier, *Acta cryst.*, **B36**, 592–596 (1980).