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Abstract. — We report magnetotransport measurements in the quasi one dimensional (Q-1-D) organic conductor $(TMTTF)_2Br$ at pressures up to 26 kbar, down to 0.45 K in magnetic fields up to 19 T along the c· direction. It is found that a superconducting ground state is stabilized under 26 kbar at $T_c = 0.8$ K. No magnetic field induced spin density wave (FISDW) transitions are observed below 19T unlike other Q-1-D superconductors pertaining to the selenium series. The computed amplitude of the interchain coupling along transverse directions is unable to explain the missing FISDW instability.

Introduction.

Radical cation salts of the $(TMTSF)_2X$ family (where TMTSF is tetramethyltetraselenafulvalene and X = PF_6, AsF_6, ClO_4, ReO_4, ..) [1] have given rise to the first series of organic superconductors [2] and also to a wealth of new phenomena such as spin density wave (SDW) ground states and field-induced SDW phases under high magnetic fields [3]. The existence of these instabilities is the natural consequence of the open and almost flat character of the Fermi surface of quasi-one dimensional (Q-1-D) conductors [4] since the structure of $(TMTSF)_2X$ compounds is triclinic and consists of parallel segregated stacks of organic molecules and inorganic mono-valent anions. The lattice constant along the stacking direction is given by the anion-anion distance.

A few years before the discovery of the $(TMTSF)_2X$ compounds, a series of isostructural materials based on the TMTTF (tetramethyltetrathiofulvalene) had been studied extensively. The growth of the $(TMTTF)_2X$ family began with X = BF_4 [5] and went on with halogens and

( ) (Associé au CNRS).

pseudo halogen anions [6, 7]. In spite of a similar crystalline structure, sulfur (TMTTF) and selenium (TMTSF) series exhibit markedly different physical properties : selenium compounds (with centrosymmetric anions, $X = PF_6$, AsF_6, .) are all very good conductors at room temperature ($\sigma_a \geq 500\ \Omega^{-1}\ cm^{-1}$) with a conductivity increasing by two and sometimes three orders of magnitude down to low temperatures where the conductor undergoes around 12 K a sharp metal-to-insulator transition of the Overhauser type towards an antiferromagnetic SDW ground state [4]. On the other hand the conductivity of TMTTF compounds does not exceed $250\ \Omega^{-1}\ cm^{-1}$ at 300 K and even if a metallic character ($d\rho/dT > 0$) persists below room temperature, it is limited at low temperatures by a localization arising at $T\rho$ (250 and 100 K for $X = PF_6$ and Br, respectively). Below $T\rho$, the charge degrees of freedom become frozen out without the occurrence of any additional lattice or magnetic distortions while the spin susceptibility is left unaffected by the electron localization [8]. The electron localization at $T < T\rho$ has been taken as the signature of the Umklapp electron-electron repulsion g_3 within the theory of the half-filled band 1-D electron gas [9, 10]. The structure of the anions X induces a $4\ k_F$ bond modulation of the electronic charge along the conducting stack with a concomitant dimerization of the stack [11]. Since this dimerization gives rise to a small gap at $\pm 2\ k_F$, the band is effectively half-filled instead of three-quarter filled as expected from the 2:1 stoichiometry neglecting the lattice dimerization. Below $T\rho$, the development of 1-D antiferromagnetic fluctuations at a wave vector $2\ k_F$ goes along with the electron localization. These AF fluctuations may either couple to the lattice and give rise to a non-magnetic spin-Peierls ground state as for $(TMTTF)_2PF_6$ at $T_{SP} = 14$ K [12] or develop into a SDW ground state as for $(TMTTF)_2Br$ at $T_{SDW} \sim 15$ K [13].

The above mentioned sulfur compounds are only two members among the broader $(TMTTF)_2X$ series. It is the effect of high pressure which has suggested why the sulfur and selenium series belong to the same generic class of isostructural TM_2X compounds [2]. The properties of $(TMTTF)_2PF_6$ under pressure resemble those of $(TMTTF)_2Br$ at 1 bar, the latter compound put under pressure looks like $(TMTSF)_2PF_6$ which in turn becomes a superconductor under pressure very much like $(TMTSF)_2ClO_4$ at ambient pressure, figure 1 In addition, it

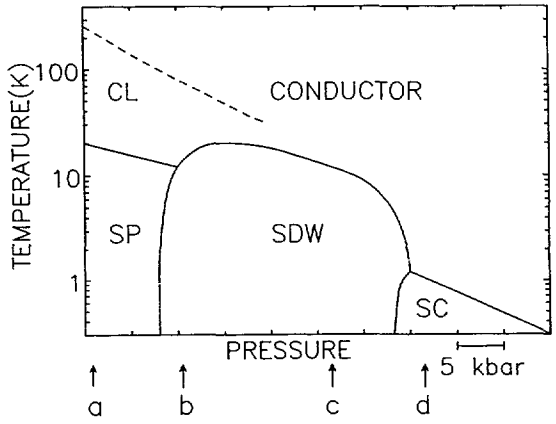

Fig. 1. — Generalized phase diagram for the $(TM)_2X$ series. The dotted line refers to the pressure dependence of T_ρ. The notation CL, SP, SDW, SC, refers to the Mott-Hubbard, spin-Peierls, spin density wave and superconducting states respectively. The lower case letters designate compounds and indicate their location at atmospheric pressure in the generalized diagram : a $(TMTTF)_2PF_6$, b $(TMTTF)_2Br$, c $(TMTSF)_2PF_6$, d $(TMTSF)_2ClO_4$.

has been shown that the dielectric ground state of $(TMTTF)_2Br$ can be suppressed under a pressure of 25 kbar [14]. The compound then retained a very large conductivity down to helium temperature but no superconducting ground state could be ascertained in spite of a minor resistance drop near 3.5 K observed in some samples without any accompanying diamagnetic shielding signal [14].

The present work reports a new study performed on $(TMTTF)_2Br$ single crystals under pressure at very low temperatures. The existence of a superconducting ground state at $T_c = 0.8$ K ($P = 26$ kbar) is firmly established by resistive and magnetoresistive measurements. $(TMTTF)_2Br$ becomes thus the first compound in the $(TMTTF)_2X$ series to reveal a superconducting ground state.

Experimental results.

Single crystals of $(TMTTF)_2Br$ were prepared by the galvanostatic oxidation of purified TMTTF [15, 16] (crystallization in ACN, 20 mg) under a nitrogen atmosphere at 20 °C. Tetrabutylammonium bromide (Fluka, 240 mg) in anhydrous THF (30 ml), (distilled from Na/Ph_2CO immediately before use), was used as supporting electrolyte. A H-shape cell was employed with platinum electrodes. A constant current (2 μA) was applied for a period of 10 days to obtain black-shiny needles (length 0.5-1 cm) formed in the anode compartment. The crystals were collected (10-12 mg) and washed twice with THF then with ether and finally dried under vacuum.

Longitudinal resistivity measurements have been performed using the classical four contact technique up to 26 kbar using a Be-Cu pressure cell and a ^3He cryostat. The pressure dependence of the longitudinal conductivity was found to be in a very good agreement with the existing literature [14], namely $\sigma_a(24\text{ kbar})/\sigma_a(1\text{ bar}) = 11$. At a pressure of 24 kbar a

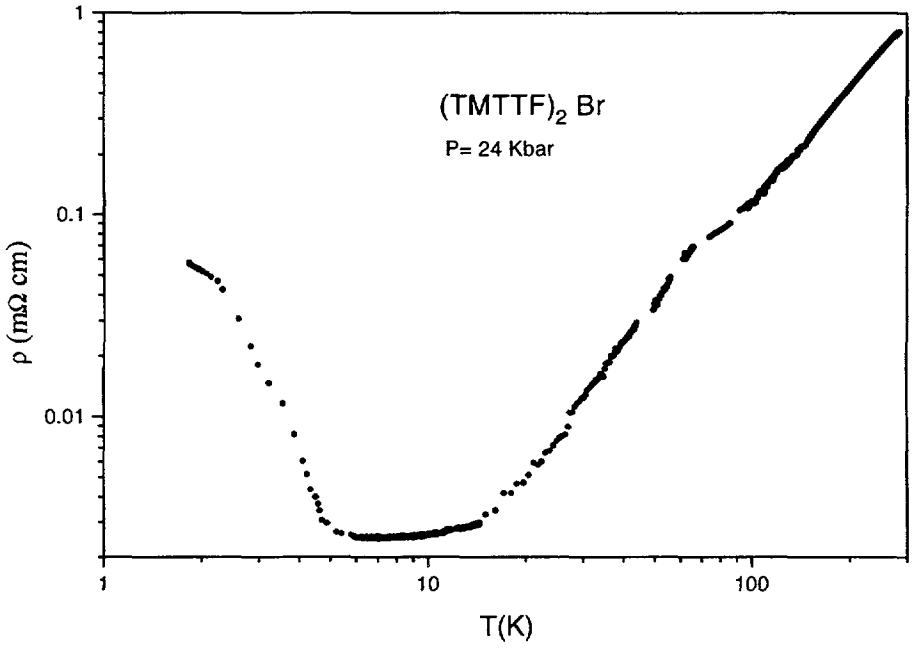

Fig. 2. — Resistivity of the $(TMTTF)_2Br$ samples versus temperature at $P = 24$ kbar. Note the SDW transition at $T_S = 5$ K.

notable increase of the conductivity is observed on cooling down to 5 K, figure 2, but an insulating ground state sets in below 5 K. After warming the sample up to room temperature the pressure was increased up to 26 kbar. The sample resistance rose by a factor about 15 during the latter procedure. However we believe this rise can be attributed mostly to a modification of the current path through the sample since the subsequent cooling under 26 kbar still revealed a metal-like behaviour with a ratio $\frac{\sigma_a(2\ \mathrm{K})}{\sigma_a(300\ \mathrm{K})} = 90$ (Fig. 3, inset).

On cooling below 2 K the resistance was still weakly temperature dependent and began to drop sharply ar 1 K. At the lowest possible temperature of 0.45 K the resistance was 17 times smaller than the 1 K value. Furthermore, the application of a magnetic field along the c^*-axis (according to the observation of the well formed natural faces of the sample) restored under 0.1 T the extrapolated value of the high temperature resistance. Moreover we did not notice any change in the $R(T)$ behaviour increasing the measuring current from 10 to 100 μA. These data show unambiguously that a bulk superconducting ground state can be stabilized in $(TMTTF)_2Br$ at $T_c = 0.8$ K with a 10 %-90 % transition width of 0.4 K. Despite very significant differences in the pressure conditions the superconductivity of $(TMTTF)_2Br$ bears much resemblance with that found in $(TMTSF)_2ClO_4$ at 1 bar or in $(TMTSF)_2PF_6$ under 9 kbar as far as values of T_c and H_{c2} are concerned [4]. One pressure run only has been performed up to 26 kbar as permanent deformations of the pressure cell prevent several uses of the same vessel.

The application of a pressure suppresses the transition temperature of the SDW ground state in $(TMTSF)_2X$ compounds above $P_c = 9$ kbar and 13 kbar for $X = PF_6$ [2] and ReO_4 [17], respectively, while the critical pressure of $X = ClO_4$ is obviously zero or negative [18]. The superconductivity of these materials is suppressed with a small field ≤ 0.1 T applied along the weak coupling c^* direction and a cascade of SDW phase characterized by a quantization of the

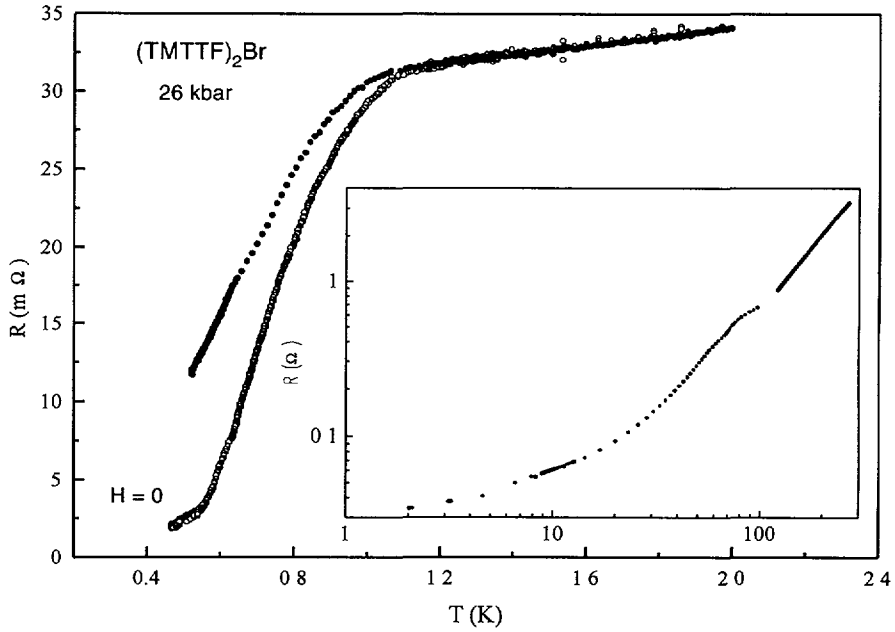

Fig. 3. — The superconducting transition at 26 kbar both for zero field and at a small transverse field. Inset : the temperature dependence of the resistance for the whole temperature range.

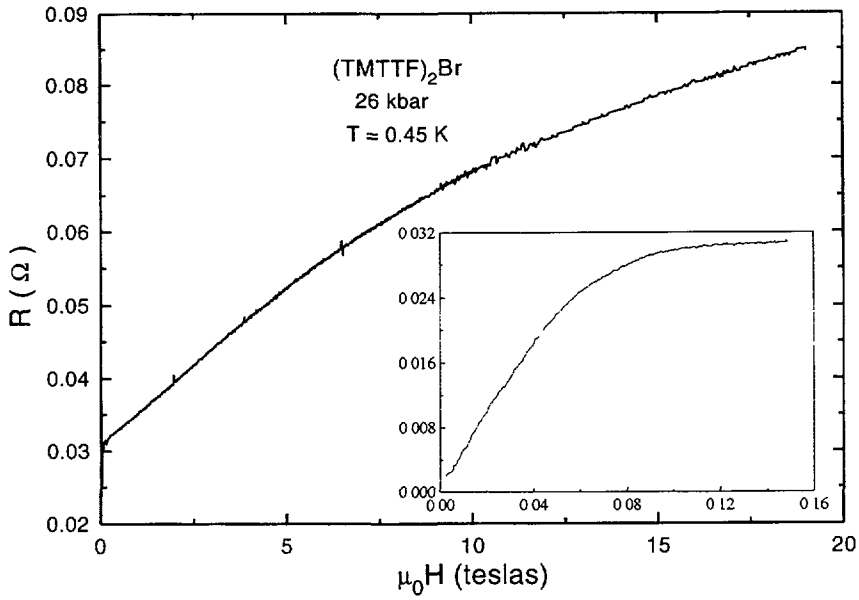

Fig. 4. — Magnetoresistance of the sample at 26 kbar and 0.45 K up to 19 T with no signature of field-induced phase transitions. Inset : low-field data presenting the superconducting transition and the upper critical field.

hall resistance is observed above 6 T [19]. This phenomenon has been interpreted within the framework of a « standard » theory in which the magnetic field drives the Q-1-D electron gas more one dimensional, restoring the SDW instability [20, 21]. So far, all $(TMTSF)_2X$ compounds showing superconductivity at $P > P_c$ have also shown a stabilization of SDW phases above a threshold field whose value is always less than 10 T, namely 4, 6, 6.5 T in $X = ClO_4$ [22] [23], PF_6 [24, 25] and ReO_4 [26], respectively. The case of $X = NO_3$ [27] is interesting as neither superconductivity nor FISDW phases have been stabilized at $P > P_c$. It has also been suggested that the existence of FISDW phases and superconductivity could be connected [28]. In this model the magnetic field may renormalize the electron-electron attraction which gives rise to superconductivity and turn it into a repulsive interaction which favors the stabilization of a density wave state. The relation between superconductivity and FISDW phases is therefore an important issue for understanding the properties of $(TM)_2X$ compounds.

The high pressure cell containing the $(TMTTF)_2Br$ sample at 26 kbar was thus moved to the Grenoble High Magnetic Field Laboratory and the magnetoresistance was measured up to 19 T at T = 0.47 K ($H \parallel c^*$) The signature of superconductivity similar to that shown in figure 3 was recovered but no anomaly of $\Delta R/R_0$ or even of its field derivative could be observed below 19 T and taken as the signature of FISDW phase transitions, see figure 4.

Band structure calculations.

In order to discuss the absence of FISDW instability in this Q-1-D conductor where superconductivity is present, band parameters responsible for the 2 and 3-D couplings should be considered. The instability towards SDW phases at zero temperature is governed by the

Table I — *Important parameters (see Fig. 5 for the labels) of the calculated dispersion relations for* $(TMTTF)_2Br$ *and* $(TMTSF)_2PF_6$ *using their room temperature and ambient pressure structures. The calculations were carried out using a basis set of either single-ζ or double-ζ Slater type orbitals. Numbers in brackets are the band parameters obtained from the two-dimensional multi-ζ calculation in reference* [30]. *All values are in* eV.

	$(TMTTF)_2Br$		$(TMTSF)_2PF_6$	
	Single-ζ	double-ζ	single-ζ	double-ζ
W_I	0.149	0.316 (0.372)	0.221	0.518 (0.595)
W_{II}	0.178	0.417	0.218	0.573
W_a	0.354	0.797 (0.859)	0.495	1.218 (1.366)
W_b	0.030	0.052 (0.044)	0.070	0.140 (0.114)
W'_b	0.082	0.257 —	0.126	0.248
W_c	0.002	0.002 —	0.002	0.003
W'_c	0.002	0.004 —	0.002	0.002

coupling along the c-direction [29]. To the best of our knowledge only 2-D calculations have so far been reported [30, 31] Therefore, three-dimensional tight-binding band structure calculations, including explicitly the anions and the methyl groups were carried out using the room temperature and ambient pressure structures of $(TMTTF)_2Br$ [17] and $(TMTSF)_2PF_6$ [32]. Thus, our calculations differ from the previous ones [30, 31] in that all donor-donor and donor-acceptor transfer integrals are calculated. An effective Hamiltonian of the Extended Hückel type [33] was used and all valence electrons were taken into account in the calculations. The basis set consisted of Slater type orbitals of both single-ζ and double-ζ quality. The exponents and contraction coefficients for the orbitals were taken from Clementi and Roetti [34]. The H_{ii} (eV) values used were : − 21.4 and − 11.4 for C 2s and 2p ; − 20.0 and − 13.3 for S 3s and 3p ; − 13.6 for H 1s ; − 22.1 and − 13.3 for Br 4s and 4p ; − 20.5 and − 13.2 for Se 4s and 4p ; − 40.0 and − 18.1 for F 2s and 2p, and − 20.2 and − 12.5 for P 3s

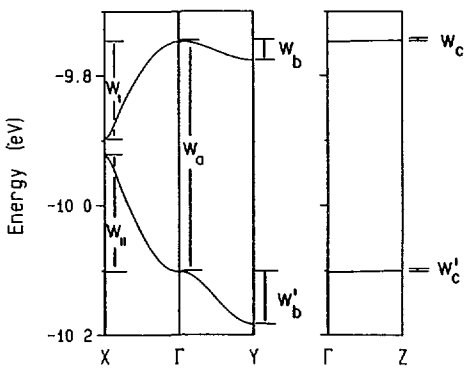

Fig. 5. — Dispersion relations calculated for $(TMTTF)_2Br$ on the basis of its room temperature and ambient pressure structure. *Γ*, *X*, *Y* and *Z* refer to the wave vectors (0, 0, 0), ($a^*/2$, 0, 0), (0, $b^*/2$, 0) and (0, 0, $c^*/2$), respectively. The calculations use a basis set of single-ζ Slater orbitals. For the calculations with double-ζ Slater orbitals see table I.

and 3p. The off-diagonal matrix elements of the Hamiltonian were calculated according to the modified Wolfsberg-Helmholz formula [35]. Results of the calculations using the ambient pressure and temperature structures [7, 32] are summarized in table I. Figure 5 gives a schematic picture of the band structure for $(TM)_2X$ compounds.

Discussion.

Using a simplified orthorhombic model for the energy dispersion, and a linearization of the dispersion law close to the Fermi energy along the conducting direction the quantity t_b^2/t_a provides a measure for the degree of unnesting of the Q-1-D Fermi surface when the transfer integral along the **c**-direction is neglected [29]. The tight binding energy dispersion relation thus becomes :

$$\varepsilon(\mathbf{k}) = v_F(|k_1| - k_F) - 2\, t_b \cos k_1\, b - 2\, t_b' \cos 2\, k_1\, b \tag{1}$$

where

$$t_b' = (1/2\sqrt{2})\, t_b^2/t_a \tag{2}$$

for the three quarter-filled band situation which prevails neglecting the structural dimerization. In the absence of the last term, namely $t_b' \ll t_b$, equation (1) represents a perfectly nested Fermi surface.

Pressure increases t_b' at a rate which can be derived from the study of the conductivity anisotropy under pressure at room temperature together with equation (2). The measured pressure dependence of selenium compounds is similar for both components of the conductivity along **a** and **b** [36, 37]. Since σ_a/σ_b is proportional to $(t_a/t_b)^2$ assuming a diffusive transverse motion of the electrons as shown by the absence of transverse plasma edge [38] we derive from equation (2), $d \ln t_b'/dP \approx d \ln t_b/dP \approx d \ln t_a/dP$. The pressure dependence of the bare band width has been derived for sulfur and selenium compounds by NMR experiments [39] and leads to $d \ln t_a/dP \approx 2.7$ and $2.2\ \%\,\text{kbar}^{-1}$, respectively.

Within the framework of the « standard » theory the ambient pressure SDW is destroyed when the deviation from perfect nesting reaches a critical value t_b' which is of the order of the SDW energy gap [29, 40], namely $t_b' \approx 0.88\, T_{SDW}^0$ whre T_{SDW}^0 is the SDW temperature of a perfectly nested Fermi surface [41]. There also exists another approach within the « standard » model to estimate the unnesting parameter t_b' since the effect of the deviations from perfect nesting is suppressed when the energy between adjacent Landau levels becomes larger than t_b' [29]. Thus the ultimate SDW phase with quantum levels becomes larger than t_b' [29]. Thus the ultimate SDW phase with quantum number $N = 0$ is reached at a field corresponding to $\omega_c/t_b' = 1.6$ (for $t_b/t_b' = 10$).

The derivation of t_b' either from the knowledge of P_c or from the determination of the $N = 1$ to $N = 0$ phase boundary in the FISDW phase diagram of $(TMTSF)_2PF_6$ using $T_{SDW}^0 = 20$ K which is the maximum value for T_{SDW} in the $(TM)_2X$ phase diagram and an effective mass unity leads to $t_b' = 17.6$ and 16 K, respectively, at $P \approx P_c$ (9 kbar). These experimental findings for t_b' are in fairly good agreement with the calculated values in table II including corrections which are requested from the high pressure (9 kbar) and from the thermal contraction leading to an overall increase of the theoretical values which may be evaluated to about 20 % following the known pressure dependence of the bare band width in the sulfur compound [36]. Furthermore, the pressure dependence of the FISDW diagram has shown that the transition fields at finite temperature increase at the same rate under pressure (30 % kbar $^{-1}$ in $(TMTSF)_2ClO_4$) [42] supporting the validity of the 2-D standard model [21] for the description of the phase diagram at finite temperature.

Table II. — *Calculated t'_b and t'_c values according to equation* (2). *We have taken* $W_{a,b,c} = 4\, t_{a,b,c}$. *All values are in* meV.

	$(TMTTF)_2Br$		$(TMTSF)_2PF_6$	
	single-ζ	double-ζ	single-ζ	double-ζ
t'_b	0.22	0.29	0.87	1.41
t'_c	3.9×10^{-4}	6.35×10^{-4}	8.6×10^{-4}	6×10^{-4}

As far as $(TMTTF)_2Br$ is concerned, a similar value of t'_b should also be reached at the pressure which is required to suppress the SDW phase at low temperatures. The values of t'_b for $(TMTTF)_2Br$ in table II are however markedly smaller than those found for $(TMTSF)_2PF_6$ even if an enhancement of about 70 % due to the higher critical pressure (26 kbar) is taken into account.

The discrepancy may be understood in terms of the very high pressure which is needed to suppress the SDW phase in this compound. Consequently, substantial modifications of the structure under 26 kbar cannot be ruled out and the results of the computation in table II may underestimate significantly the deviation from perfect nesting at high pressure. However, the small t'_b values for $(TMTTF)_2Br$ in table II including the 70 % enhancement imply that the $N = 1$ to $N = 0$ transition related to the amplitude of t'_b should still be visible at a magnetic field smaller than 19 T.

Unlike the 2-D coupling along **b** a finite interaction along the third direction does provide a finite value of the threshold field H_I for the appearance of FISDW phases at zero temperature which is given by the equation :

$$T_c^{2D}(H_I) \sim t'_c \tag{3}$$

where $T_c^{2D}(H)$ displays the critical line for a strictly 2-D conductor [29]. Table II does not show any major difference between sulfur and selenium compounds as far as t'_c is concerned. This finding is not too surprising as no contribution to the interchain coupling coming from chalcogen orbitals is expected along this direction. Given the amplitudes of the interchain couplings of table II, equation (3) would lead to threshold fields at zero temperature less than 0.5 T. Therefore, three-dimensionality is unable to explain threshold fields of the order of 3 T and 3.5 T which have been observed at very low temperatures in $(TMTSF)_2ClO_4$ [43] and $(TMTSF)_2PF_6$ [44], respectively. We have also checked that no unusually large change of t_c occurs under pressure for $(TMTTF)_2Br$. The transverse conductivity along **c** has been measured up to 22 kbar with a very good accuracy, figure 6. Above 10 kbar, σ_c increases faster than σ_a and the anisotropy drops by a factor 2 in 22 kbar. Hence t'_c is about twice larger at P_c as compared to the ambient pressure conditions but still remains too small to preclude the observation of a threshold field below 19 T.

The electron life time could be another obstacle to the stabilization of FISDW phases since H_T would thus be determined by $T_c^{2D}(H_T) = \hbar/\tau$. Our present knowledge of $\hbar/\tau$ is rather limited but a crude estimate can be derived from the experimental pressure and temperature dependence of the conductivity. Using $\hbar/\tau \leq E_F$ (1 500 K) which can be expected since the compounds exhibits a metal-like conduction under normal conditions we thus obtain the upper limit $\hbar/\tau \sim 1.5\,K$ at $T = 1$ K under 26 kbar according to the increase of three orders of

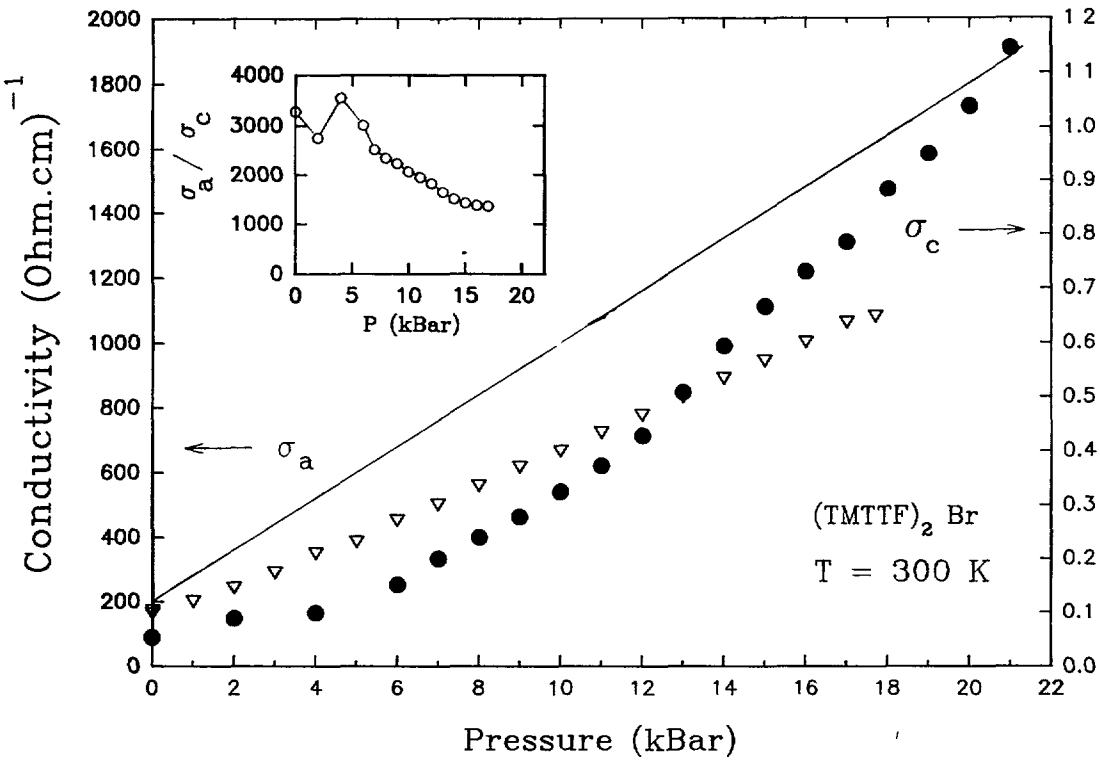

Fig. 6. — Typical pressure dependence of the conductivity along the a and c-directions, triangles and dots, respectively, (data for the a-direction [14] are also indicated, continuous line). The inset shows the pressure dependence of the anisotropy.

magnitude of $\sigma_{\|}$ which is currently observed. It is possible that such a finite energy level broadening may affect the stability of FISDW phases and push it above 19 T. The limitation by a finite life time does not seem to interfere with the stability of FISDW phases of selenium compounds since the overall agreement between different published FISDW phase diagrams is very satisfactory for these compounds. however, it has been shown that the threshold field in $(TMTSF)_2ClO_4$ can be influenced by the presence of anion disorder in raipidly cooled samples [22].

In addition, we may notice from the inspection of figure 4 that rapid magneto-oscillations which are observed in ClO_4 [46] and ReO_4 [46] selenium compounds even below H_T when electron orbits are open are not seen in $(TMTTF)_2Br$. This may also be due to a finite electron life time effect.

Conclusion.

In summary, we have performed magneto-transport measurements under pressure in a member of the $(TM)_2X$ series, $(TMTTF)_2Br$, which shows at ambient pressure a Mott-Hubbard localization below 100 K and a commensurate SDW ground state at 14 K [47]. Superconductivity is stabilized at T_c = 0.8 K under 26 kbar. $(TMTTF)_2Br$ thus becomes the first compound in the sulfur series to exhibit superconductivity and this finding confirms that the physics of both selenium and sulfur series must be treated on an equal footing [48]. However, unlike all

other superconducting members of the $(TM)_2X$ series $(TMTTF)_2Br$ has failed to reveal both the stabilization of FISDW phases and the existence of rapid magneto-oscillations below 19 T. The computed value of the interchain coupling along the **c** direction together with the pressure dependence of the transverse conductivity predict that FISDW phases should be observable just above the critical pressure as for other $(TMTSF)_2X$ compounds. At this stage it should be kept in mind that the distance between the actual pressure and the critical pressure is an important parameter for the stability of FISDW phases since the threshold field as well as all characteristic fields between FISDW sub-phases are known to exhibit a pronounced pressure dependence of about 30 % kbar^{-1} in $(TMTTF)_2ClO_4$ [42] and about 10 % kbar^{-1} in $(TMTSF)_2PF_6$ [49]. We cannot discard the possibility that the distance from the critical pressure is too large to observe the stabilisation of FISDW phases below 19 T.

Up to now a finite electron life time smearing the observation of quantum effects at low temperature cannot be ruled out but is hard to reconcile with the large value of the conductivity which is retained with no saturation at low temperatures.

Admittedly pressure experiments on $(TMTTF)_2Br$ up to 30 T and even at higher fields in the pulsed-field range should be very valuable. Other members of the $(TMTTF)_2X$ series, $X = PF_6$, ClO_4, . should also become superconducting although at a pressure exceeding 40 kbar [14].

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