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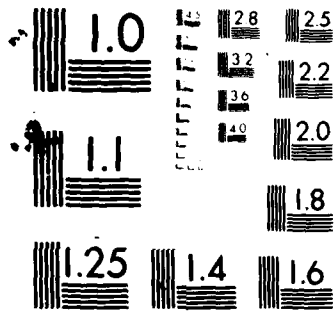
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RAMAN STUDY OF THE MECHANISM OF ELECTRICAL SWITCHING IN CU TCN6--ETC(U)
JAN 82 E I KAMITSOS, C H TZINIS, W M RISEN N00014-75-C-0883
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TECHNICAL REPORT NO. TR82-01

Raman Study of the Mechanism of Electrical Switching in Cu TCNQ Films

by

E. I. Kamitsos, C.H. Tzinis and W. M. Risen, Jr.

Prepared for Publication

in

Solid State Communications

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January 5, 1982

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4. TITLE (and Subtitle) Raman Study of the Mechanism of Electrical Switching in Cu TCNQ Films		5. TYPE OF REPORT & PERIOD COVERED Technical
		6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s) E. I. Kamitsos, C. H. Tzinis, W. M. Risen, Jr.		8. CONTRACT OR GRANT NUMBER(s) N00014-75-C-0883 NR-051-539
9. PERFORMING ORGANIZATION NAME AND ADDRESS Department of Chemistry Brown University Providence, Rhode Island 02912		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS ONR-N00014-75-C-0883 NR-051-539
11. CONTROLLING OFFICE NAME AND ADDRESS Office of Naval Research United States Navy		12. REPORT DATE January 5, 1981
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)		13. NUMBER OF PAGES 15
		15. SECURITY CLASS. (of this report)
16. DISTRIBUTION STATEMENT (of this Report) Distribution Unlimited; Approved for Public Release		
17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)		
18. SUPPLEMENTARY NOTES		
19. KEY WORDS (Continue on reverse side if necessary and identify by block number) Raman spectra, Infrared spectra, Electrical switching, Cu TCNQ, film, mechanism, TCNQ, charge transfer complexes, organic semiconductor, one-dimensional conductor. Cu-TCNQ-		
20. ABSTRACT (Continue on reverse side if necessary and identify by block number) The mechanism of electrical switching in Cu/Cu TCNQ films has been studied <u>in situ</u> by Raman spectroscopy. The initial film is Cu ⁺ TCNQ ⁻ , while neutral TCNQ was found to be present in the system after electrical switching, produced by a redox reaction caused by the field. The amount of TCNQ after switching was found to increase with the time of application of the electric field, while the amount of Cu TCNQ was reduced.		

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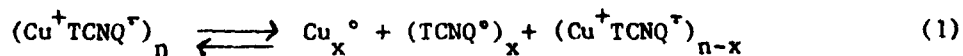
Raman Study of the Mechanism of Electrical Switching in Cu TCNQ Films

E. I. Kamitsos, C. H. Tzinis and W. M. Risen, Jr.

Introduction

The synthesis of new strong electron-acceptors such as tetracyanoethylene (TCNE)(1) and 7,7,8,8-tetracyanoquinodimethane (TCNQ)(2) has stimulated interest in organic semiconductors, and the charge-transfer complexes based on such acceptors have been widely investigated. Although such materials have not yet found extensive application, Potember, Poehler, Cowan and coworkers (3-5) recently reported achievement of reversible and rapid bistable electrical switching from a high to a low impedance state in films of copper-TCNQ (Cu-TCNQ). Switching times of approximately 10 nsec and memory effects in the low impedance state characterize the switching phenomena of this interesting system.

In order to interpret the electrical switching and memory effects in the Cu-TCNQ films, Potember, et al (3,4) postulated that a phase containing a complex salt and involving neutral TCNQ is formed as a result of a field-induced redox reaction. The film phase formed initially by the reaction of a copper substrate and TCNQ solution was proposed to be Cu TCNQ, and it was suggested that the effect of an applied field is to shift the following equilibrium to the right to form the low impedance phase:



Recently an attempt was made to confirm this postulate by measuring the infrared reflectance spectra of the film before and after the field was applied to the Cu-TCNQ film. It was reported that the spectrum obtained after the application of

the field showed a new peak in the high frequency region, at 2340 cm^{-1} . It was assigned to TCNQ° , which was presumed to have been formed under the action of the electric field. The observation of TCNQ° would be in accord with the postulate, but the position of this feature (about 100 cm^{-1} higher than that in the absorption spectrum of TCNQ), the shapes of the features, the S/N ratio of the spectra, and the report that the related systems measured all had features between 2320 and 2344 cm^{-1} suggested that further investigation was warranted.

Understanding the chemical changes accompanying the electrical switching is important to understanding the phenomena exhibited by this fascinating Cu-TCNQ system Potember, Poehler, Cowan and coworkers reported. Therefore, we have investigated the nature of the system before and after switching by Raman and infrared spectroscopic and chemical methods. Raman spectra are sensitive to the charge on TCNQ in charge-transfer salts (6) and they are free from spectral features due to electron-phonon coupling which are present in the corresponding infrared spectra (6,7). Thus, they have been used to identify the species in situ and to determine the relative amounts of each present before and after switching.

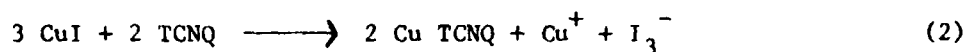
Experimental

The TCNQ used in these experiments was purchased from Aldrich Chemical Company, and was recrystallized twice from dry acetonitrile and then sublimed onto a teflon surface under vacuum. Some Cu-TCNQ films were prepared as described by Potember, et al (3). The copper foils were cleaned mechanically, and then chemically in $1.5\text{ M H}_2\text{SO}_4$. The Cu-TCNQ films were formed by dipping the clean dry Cu foil into a TCNQ solution in dry acetonitrile. It is known that TCNQ in CH_3CN reacts at 25°C with metallic Cu or Ag to form the metal salts of the anion-radical TCNQ^{\ominus} (8). The Cu-Cu TCNQ film structure was then removed from

the solution, washed with CH_3CN , and dried under vacuum to remove residual solvent. A transparent top electrode, either a thin Cr film or a SnO film, was vapor deposited on the Cu/Cu TCNQ structure. Electrical connection to the Cu electrode was made with a Cu wire before cleaning the Cu foil, while connection to the top electrode was made using either silver paint or a pressure probe.

Pure Cu TCNQ salt was prepared by mixing warm acetonitrile solutions of CuI and TCNQ in the mole ratio 3:2. The blue Cu TCNQ salt precipitated and was separated by filtration from the by-products, which remained in solution.

The 3:2::CuI:TCNQ relation was used so that I_3^- was formed by the reaction



and the oxidation of Cu TCNQ by I_2 was prevented.

Other Cu TCNQ films were formed by first vapor depositing a Cu film on clean Cu foil under high vacuum and then vapor depositing a thin film of TCNQ on it. After measuring the Raman spectra of the films as made, several were heated at 110°C in air and studied further.

The infrared spectra (Cu TCNQ salt as KBr pellet in transmission and Cu-TCNQ film in reflectance) were measured on a Perkin-Elmer 580 spectrometer. The Raman spectra were obtained on a Jarrell-Ash 25-300 spectrometer with 90° scattering geometry using the 476.5 nm line of a Spectra Physics 165 Ar-ion laser. The spectral accuracy and resolution of all measurements was 2 cm^{-1} or better.

Results and Discussion

Infrared Spectra. The infrared transmission spectrum of a KBr pellet of Cu TCNQ salt is shown in Figure 1a. It exhibits bands similar to those of such 1:1 metal TCNQ salts as Li TCNQ, Rb TCNQ (9), and Na TCNQ (10). The C-N stretching region is characterized by three bands, maxima at 2173 and 2200 cm^{-1} and a shoulder at ca 2210 cm^{-1} . The spectrum also contains the four bands reported by Bozio, et al (9) at 1578, 1508, 826 and 478 cm^{-1} .

Due to the diffusive reflectance of the Cu TCNQ film, prepared as described by Potember, et al, its reflectance spectrum showed clearly only the strong bands at 478 and 826 cm^{-1} in addition to the band structure due to uncompensated atmospheric CO_2 absorption between 2310 and 2360 cm^{-1} . When the Cu TCNQ film was either pressed between two quartz plates or rolled lightly with a small cylinder, it was possible to obtain the reflectance spectrum. The salient regions of this spectrum are shown in Figure 1b. Of particular interest is the presence of two bands in the C-N stretching region at 2216 and 2170 cm^{-1} .

There is good correspondence between the transmission spectrum of Cu TCNQ and the reflectance spectrum of the film. Although care must be exercised to allow for dispersion, in relating them, it is seen that some of the film bands are shifted only slightly (maximum 8 cm^{-1}) and the strongest bands (478 and 826 cm^{-1}) are not shifted at all. The 2216 and 2170 cm^{-1} film reflectance bands correspond to the 2200 and 2173 cm^{-1} C-N stretching bands in the Cu TCNQ transmission spectrum. Based on this, we conclude that the film as prepared (unswitched) does contain Cu TCNQ, but that its C-N stretching modes do not appear above 2300 cm^{-1} .

Potember, et al (5) reported one reflection maximum at 2320 cm^{-1} for the unswitched Cu TCNQ film and one at 2323 cm^{-1} for the Cu TCNQ salt prepared by the metathetical reaction. Since only the 1600-2800 cm^{-1} region is reported it isn't possible to compare their spectra with others in other regions. However, the bands reported at ca 2320 cm^{-1} are similar to those of CO_2 absorption left uncompensated in our spectra in Figure 1. Since a similar feature appears in their Li TCNQ spectrum, while the reported transmission spectrum of that salt shows four bands in the 2200 cm^{-1} region (strongest at 2211 cm^{-1}), and since it has been shown (7) that for K TCNQ the reflectance and transmission spectra do not differ remarkably, we conclude that the reported observation of Cu TCNQ on the film (5) was erroneous.

While the infrared spectra now show Cu TCNQ on the film, it is important to note that Potember, Poehler, Cowan, Brant, Carter and Bloch (5) showed by

photoelectron spectroscopy that the TCNQ in the unswitched Cu TCNQ film has the same oxidation state as it does in Cu TCNQ salt. Thus, the proposed composition of the unswitched film (left hand side of Eqn (1)) is confirmed by infrared and photoelectron spectroscopy.

Raman Spectra. Since TCNQ is a centrosymmetric species and has strong sharp Raman active modes which are quite sensitive to oxidation state, it was concluded that Raman spectroscopy is the preferred way to observe changes caused by switching. Since TCNQ itself is a proposed product (Eqn 1) the Raman spectrum of pure TCNQ powder in a capillary tube was measured with the 476.5 nm line at 5 mW power. This is shown in Figure 2a, and is similar to published spectra of TCNQ measured at 457.9 nm (11) or 488.0 nm (12).

The Raman spectrum of a Cu TCNQ film, prepared by the TCNQ solution method and measured using 15 mW of power at 476.5 nm, is shown in Figure 2b. The spectrum of Cu TCNQ salt, measured in the form of a spinning pellet, is shown in Figure 2c. The spectra in Figures 2b and 2c are essentially the same, which is in accord with our infrared results and the reported photoelectron results (5).

To ensure that the Cu TCNQ film was not affected by depositing the semi-transparent Cr electrode on it, the Raman spectrum of the unswitched film was measured by directing the laser source through this electrode. This spectrum, which is shown in Figure 3a, is nearly identical to the spectrum of an unswitched film to which no electrode had been applied (Figure 2b). The only difference is a small shift in the base line.

After measuring their spectra in the unswitched state, electrical switching was performed on several of the films by applying 5 V across the Cr-Cu TCNQ (film)-Cu devices which had film thicknesses of ca 3 μ . This corresponds to a field strength

of 1.7×10^6 V/m. The spectrum of a typical switched film, recorded through the Cr electrode immediately after switching to the low impedance state was achieved, is shown in Figure 3b. Two new bands appeared after switching, at 2223 and 1451 cm^{-1} , and the intensities of the bands at 1375, 980, and 734 cm^{-1} were reduced relative to those at 1603 and 1203 cm^{-1} . Since the bands at 1451 and 2223 cm^{-1} are characteristic of TCNQ (Figure 2a), these spectra demonstrate the presence of neutral TCNQ in the system after electrical switching. This clearly supports the postulate given as Equation (1) above.

The Raman spectra of Cu TCNQ and TCNQ are such that the position of the equilibrium given in Equation (1) can be estimated from the relative intensities of their distinct bands. Eight of the ten totally symmetric (Ag) Raman fundamentals of TCNQ and TCNQ⁻ (9,10,13-15) were observed in this work in the 300-2300 cm^{-1} region and are listed in Table 1. The ν_3 and ν_5 modes are not much affected by changes in the electronic structure of the TCNQ moiety, while the others, especially ν_4 , ν_6 , ν_7 , and ν_2 are quite sensitive to these changes. Intensity ratios for key bands of Cu-TCNQ (unswitched) and TCNQ are given in Table 2 along with the ratios of intensities with respect to the switched Cu TCNQ film.

Employing these ratios it is possible to calculate the mole fractions of TCNQ and Cu TCNQ after switching. One such calculation uses the assumption that the intensity of the 1598 cm^{-1} band of TCNQ present after switching adds linearly to that of the 1603 cm^{-1} band of the remaining Cu TCNQ to result in the 1600±2 cm^{-1} band. The same assumption holds for the 1203 and 1204 cm^{-1} bands. Letting p and q be the mole fractions of Cu TCNQ and TCNQ respectively after switching, so that $p + q = 1$, and the above assumptions, then

$$p = \frac{I_{1375}/I_{1603}(\text{switched})}{I_{1375}/I_{1603}(\text{unswitched})}$$

and

$$q = \frac{I_{1451}/I_{1603}(\text{switched})}{I_{1451}/I_{1598}(\text{unswitched})}$$

Using other intensity ratios analogously, four values of p are obtained, which give $\bar{p} = 0.82$, and two values of q are found, which give $\bar{q} = 0.14$. Thus, \bar{p} and $\bar{q} = 0.96$, compared to 1.0. Since there is a 2% error in each ratio this propagates to about 4% error in $\bar{p} + \bar{q}$, so the result is within experimental error. Using a number of samples, we have found that immediately after switching TCNQ is present in the range of 10 to 15 mole percent.

It was reported that the Cu/Cu TCNQ films eventually returned to the initial high impedance state after the removal of the applied field (4), it also was noted that the time required for this "back switching" (or retention of memory) appeared to depend on film thickness and duration of field application, or, perhaps, the energy dissipated in the sample. Thus, it seemed useful to determine if the relative amount of TCNQ produced also increased with time, even after switching has occurred. By applying the electric field on samples for various times, samples with variously increased amounts of TCNQ were obtained. Analysis of the spectra obtained after application of a ca 1.7×10^6 V/m field for 24 hours showed that nearly 40 mole percent of the TCNQ moieties were in the TCNQ[°] form.

The longer the field was applied the greater was the conversion of TCNQ⁺ to TCNQ[°]. It also is true that the more TCNQ[°] produced the longer is the time required for the back reaction to the high impedance state, but this does not necessarily mean that the reduction of TCNQ or even its mobility in either state is the rate limiting process. Indeed, the role of $(Cu)_n$ may be quite important.

To help understand this system in its Cu[°] TCNQ[°] state, films were made by vapor depositing TCNQ on vapor deposited Cu films on a Cu substrate. Only bands due to TCNQ were observed when the Raman spectra of such films were measured, so no reaction takes place immediately. After these films had been left in the air at 25°C for two weeks no evidence of reaction was found. When such films were heated in air at 110°C for one hour, their Raman spectra showed

that no TCNQ° remained but that the characteristic Raman bands of $\text{Cu}^+\text{TCNQ}^{\mp}$ were present. This suggests that $\text{Cu}^+\text{TCNQ}^{\mp}$ is the thermodynamically stable state, although the volatilization of TCNQ° must be considered, and further supports the postulate that the system returns to the initial state of Equation 1 when the stress is removed. Moreover, the result of this experiment supports the idea that the mechanism of the switching phenomenon is not due to thermal effects (4).

Conclusion

The mechanism of electrical switching in Cu/Cu TCNQ films has been studied in situ by Raman spectroscopy. The initial film is essentially $\text{Cu}^+\text{TCNQ}^{\mp}$. Neutral TCNQ was found to be present in the system after switching, while the amount of Cu TCNQ was reduced. The solid state equilibrium (Equation (1)) appears to describe the switching well, with approximately 10 mole percent conversion needed to reach the low impedance state. The results indicate that this equilibrium is shifted to the left (toward $\text{Cu}^+\text{TCNQ}^{\mp}$) thermally. The precise role of the electric field in switching and the role of $(\text{Cu}^{\circ})_n$ in the relaxation process were not determined, but it was shown that the amount of TCNQ produced by the switching increases with the time of application of the electric field.

Acknowledgments

This work was supported by the Office of Naval Research and by the Materials Research Laboratory of Brown University through the National Science Foundation. We are grateful to Mr. Ted Kirst of the MRL Optical Facility and Mr. Keith Warren of the MRL Microelectronics facility for their kind assistance.

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Table 1

Totally Symmetric Raman Fundamentals of TCNQ and Cu TCNQ in the 300-2300 cm^{-1} Region

Mode	TCNQ, cm^{-1}	Cu TCNQ, cm^{-1}	$\Delta\nu(\text{Cu TCNQ}-\text{TCNQ}), \text{cm}^{-1}$
ν_2	2223	2205	-18
ν_3	1598	1603	+ 5
ν_4	1451	1375	-76
ν_5	1204	1203	- 1
ν_6	948	980	+32
ν_7	710	734	+24
ν_8	600	616	+16
ν_9	333	341	+ 8

Table 2

Intensity Ratios and Mole Fractions of Cu TCNQ and TCNQ after Electrical Switching

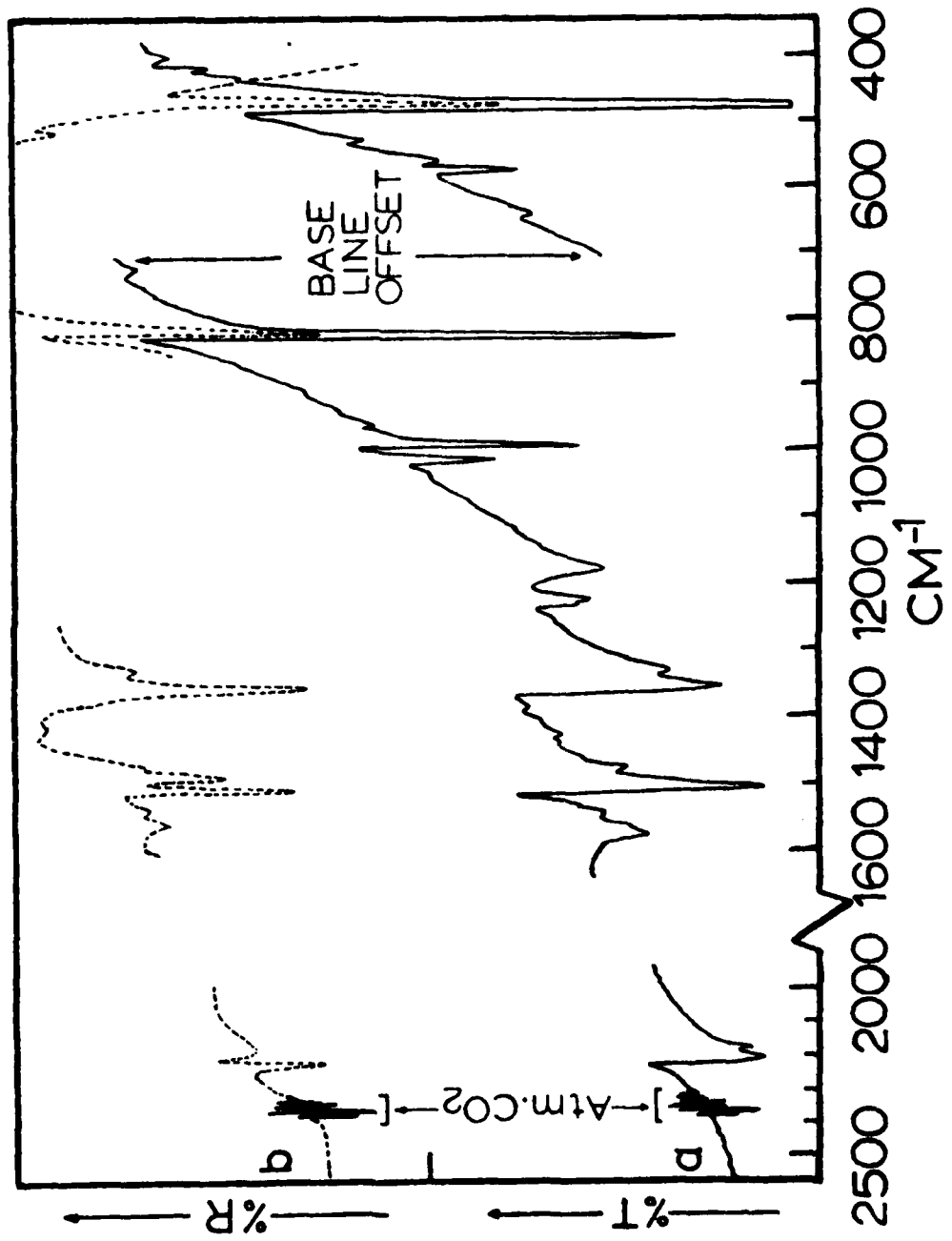
Intensity Ratios	Before Switching		After Switching	p	q
	Cu TCNQ	TCNQ			
I_{1375}/I_{1603}	0.41	-	0.33	0.81	-
I_{1375}/I_{1203}	0.43	-	0.36	0.84	-
I_{734}/I_{1603}	0.89	-	0.71	0.80	-
I_{734}/I_{1203}	0.95	-	0.77	0.81	-
I_{1451}/I_{1598}	-	2.78	0.43	-	0.15
I_{1451}/I_{1204}	-	3.77	0.47	-	0.12

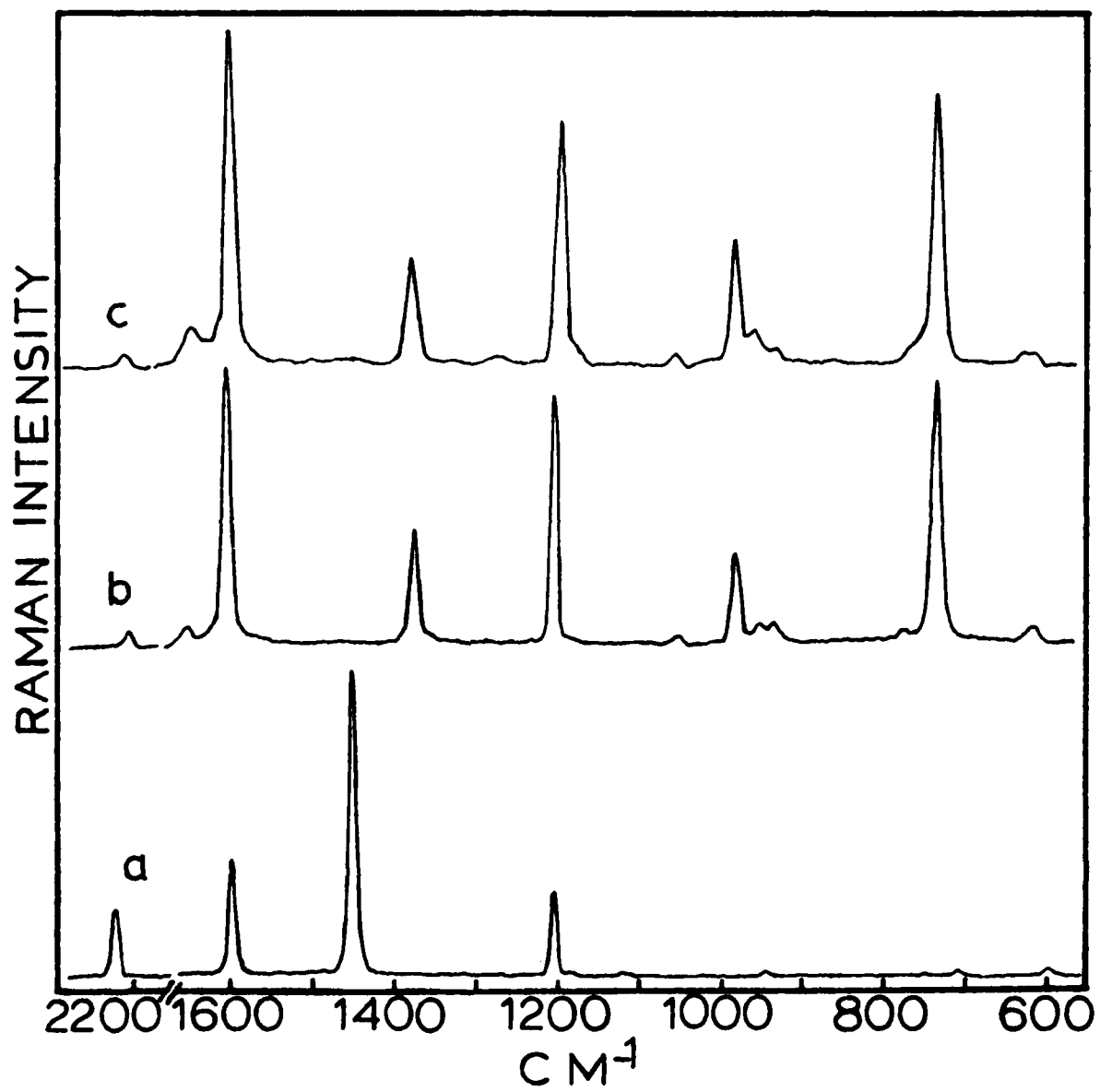
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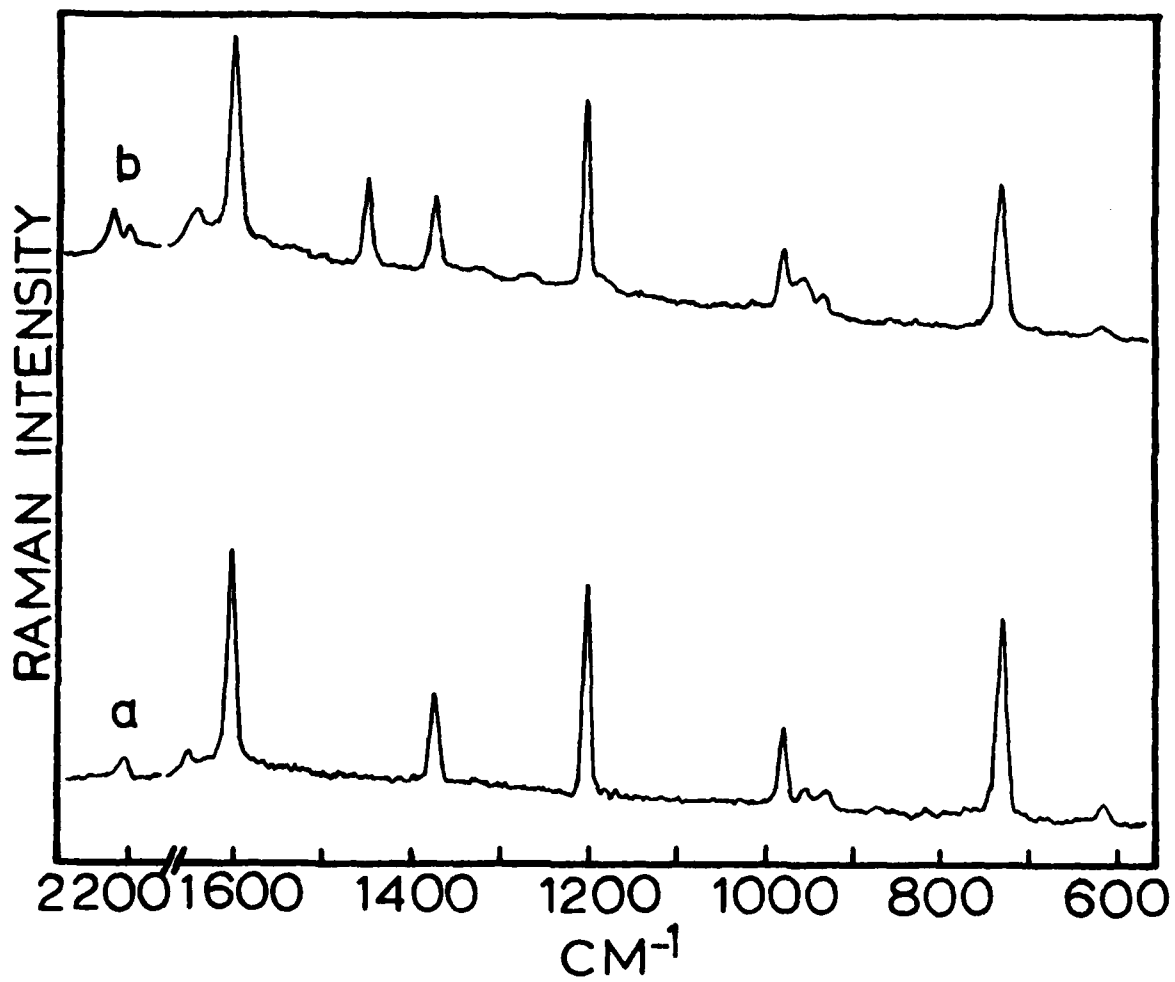
Figure 1. (a) Infrared Transmission Spectrum (KBr pellet) of Cu TCNQ Salt.
(b) Representative Regions of the Infrared Reflectance Spectrum
of Cu TCNQ Film.

Figure 2. Raman Spectra of TCNQ (a), Cu TCNQ Film as Prepared (b) and Cu TCNQ
Salt (c).

Figure 3. Raman Spectra of Cu TCNQ Film through Cr Electrode (a) and Cu TCNQ
Film after Electrical Switching (b).







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