

Amplitude and nature of the charge-density-wave displacements in $K_2Pt(CN)_4Br_{0.3} \cdot 3.2D_2O$ (KCP) at low temperatures

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In this paper we introduce the concept of flexible and rigid response of a molecular unit to a charge density wave (CDW) in the electron gas. We show that the CDW satellite reflections observed in a neutron-diffraction study at 7 K on a single crystal of $K_2Pt(CN)_4Br_{0.3} \cdot 3.2D_2O$ (KCP) can be adequately explained by a rigid sinusoidal displacement of the $Pt(CN)_4$ complexes in response to the CDW instability in the d_{z^2} band formed from the Pt atoms. The amplitude of the displacement wave is determined to be $(0.0047 \pm 0.0005)\bar{c}$, where \bar{c} is the lattice parameter, 5.692 Å at 7 K, parallel to the $Pt(CN)_4$ chains.

I. INTRODUCTION

The original discovery by Comes *et al.*¹ of the diffuse planes of scattering perpendicular to the tetragonal \bar{c} axis in $K_2Pt(CN)_4Br_{0.3} \cdot 3.2H_2O$ (KCP) in a room-temperature x-ray-diffraction experiment was a major breakthrough in our understanding of the one-dimensional nature of this salt. Since that time, a number of x-ray- and neutron-diffraction papers have appeared which have served both to clarify certain aspects of the problem and to obscure or confuse others.²⁻⁶

It is now clear that KCP contains sinusoidal atomic displacement waves which, at low temperatures, give rise to a reasonably sharp three-dimensional array of satellite Bragg reflections. The view has been that the atomic displacement waves result from a Peierls' distortion⁷ in the one-dimensional band derived from the overlapping d_{z^2} orbitals of the Pt atoms. The recent discovery of charge density waves (CDW) in certain layered metal dichalcogenides,⁸ vanadium sulfide,⁹ magnetite,¹⁰ and niobium dioxide,¹¹ suggests that we should broaden our view regarding KCP and consider the sinusoidal atomic displacement waves, and their phase "fluctuations" at higher temperatures, as resulting from a CDW instability in the electron gas as originally discussed by Overhauser.¹²

We recognized some time ago from our low-temperature neutron-diffraction measurements of certain odd- l Bragg reflections that the average centrosymmetric crystal structure deduced by Krogmann and Hausen¹³ must be wrong in certain details. Subsequent neutron and x-ray-diffraction studies by Williams *et al.*,⁴ Deiseroth and Schulz,⁵ and Peters and Eagen⁶ have shown the structure to be noncentrosymmetric. Figure 1 shows the basic elements of the crystal structure and includes the "disordered defect" water site discussed in Ref. 6. Since the observed satellite peaks result from a modulation of the underlying average structure by a CDW,

it would seem that a reasonably precise knowledge of the average position of each atom in the tetragonal unit cell would be necessary in order to explain the observed satellite intensities. However, the response of the K, Br, and H_2O in each unit cell to the CDW seems to be of secondary importance.

The purpose of this paper is to introduce the essentially new concept of flexible and rigid response of a molecular unit to a CDW and to present low-temperature neutron-diffraction data on the CDW satellite peaks in a deuterated KCP single crystal which are adequately explained in terms of a rigid response of the nearly planar $Pt(CN)_4$ complexes to the CDW. A complementary study on how the satellite peaks spread out onto diffuse planes as the CDWs dephase with increasing temperature is reported elsewhere.¹⁴

II. NEUTRON SCATTERING FROM A CRYSTAL CONTAINING CDW'S

In this section we will develop formulas for the neutron-scattering cross section for a crystal containing charge-density waves. Of course, the general ideas apply equally well to x-ray and electron diffraction. The results will be specialized to KCP in Sec. III.

Following Overhauser,¹² if there is a wave of the form

$$\rho(\vec{r}) = \rho_0(1 + \beta \cos \vec{Q} \cdot \vec{r}) \quad (1)$$

in the electron gas which modulates the charge density, the atoms in the crystal will respond by shifting their equilibrium positions. Each atom may respond differently in both phase and amplitude to the CDW. To first order, the response must be linear and therefore purely sinusoidal. The problem in a diffraction experiment is to determine the amplitude and phase of the sinusoidal response of each atom in the underlying average unit cell.

There are two basic assumptions one can make regarding the phase of the atomic response. The first is to assume that the phase of the displace-

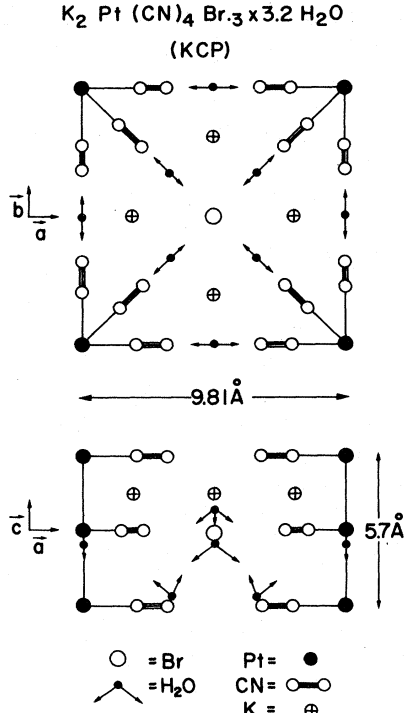


FIG. 1. Average crystal structure of KCP as projected on the planes $(x, y, 0)$ and $(x, 0, z)$. A disordered-defect water molecule, which occupies the center of the cell when the Br ion is absent, is shown in the bottom projection.

ment of each atom is determined by its average position. This type of response we term "flexible." In KCP, where the d_{z^2} band of electrons (in which the CDW instability is thought to exist) is more or less localized on the strands of the Pt atoms parallel to the \vec{c} axis, we might expect the phase of the displacement of the CN ligands associated with a given Pt atom to be determined by the average position of the Pt atom. This type of response we term "molecular" or "rigid." In general, the displacements in a complex unit cell may be a combination of both modes where molecules rigidly displace and where the displacements among molecular units are flexible.

A. Flexible response

Following the convention common to crystallography, we write the average position of an atom as $\vec{R}_{\vec{L}}(i, j)$. The index \vec{L} denotes the unit cell, the index i denotes a type of atom, and j runs over the equivalent symmetry positions appropriate for the i th atom. We will first examine the special case where the atomic positions are given by

$$\vec{r}_{\vec{L}}(i, j) = \vec{R}_{\vec{L}}(i, j) + \vec{A}(i) \sin \vec{Q} \cdot \vec{R}_{\vec{L}}(i, j), \quad (2)$$

where the amplitudes $\vec{A}(i)$ are parallel to the \vec{c} axis. Foreseeing the specialization to KCP, we define

$$\vec{Q} = (\pi/a)(\hat{x} + \hat{y}) + Q_z \hat{z}, \quad (3)$$

so that the displacement on adjacent chains, separated by a distance a , will be 180° out of phase. Q_z is assumed to be incommensurate with the tetragonal reciprocal lattice.

The interaction of thermal neutrons with a crystal containing a CDW is

$$V(\vec{r}) = \sum_{\vec{L}, i, j} b_i \delta(\vec{r} - \vec{r}_{\vec{L}}(i, j)), \quad (4)$$

where b_i is the coherent nuclear scattering length of the i th atom multiplied by a Debye-Waller factor.

In order to calculate the scattering cross section, we need the Fourier transform of $V(\vec{r})$, namely

$$\vec{V}(\vec{q}) = \sum_{\vec{L}, i, j} b_i \exp(i\vec{q} \cdot \{\vec{R}_{\vec{L}}(i, j) + \vec{A}(i) \sin[\vec{Q} \cdot \vec{R}_{\vec{L}}(i, j)]\}). \quad (5)$$

By use of the Jacobi-Anger expansion,

$$e^{ix \sin \theta} = \sum_{n=-\infty}^{+\infty} J_n(x) e^{in\theta}, \quad (6)$$

where the J_n are Bessel functions of the first kind, Eq. (5) can be written

$$\vec{V}(\vec{q}) = \sum_{n=-\infty}^{+\infty} \left(\sum_{i, j} b_i J_n(\vec{q} \cdot \vec{A}(i)) e^{i\vec{u}(i, j) \cdot (\vec{q} + n\vec{Q})} \right) \times \sum_{\vec{L}} e^{i\vec{T}_{\vec{L}} \cdot (\vec{q} + n\vec{Q})}. \quad (7)$$

The translation vectors of the direct lattice are $\vec{T}_{\vec{L}}$, and $\vec{u}(i, j)$ denotes the average atomic position within a unit cell such that

$$\vec{R}_{\vec{L}}(i, j) = \vec{T}_{\vec{L}} + \vec{u}(i, j). \quad (8)$$

The sum over \vec{L} in Eq. (7) is only large when

$$\vec{q} + n\vec{Q} = \vec{G}, \quad (9)$$

where \vec{G} is a reciprocal-lattice vector of the underlying average crystal structure. If we define the n th-order CDW structure factor as

$$F_n(\vec{G}) = \sum_{i, j} b_i e^{i\vec{G} \cdot \vec{u}(i, j)} J_n(\vec{q} \cdot \vec{A}(i)), \quad (10)$$

then we obtain for the scattering cross section

$$\frac{d\sigma}{d\Omega} \equiv \vec{V}(\vec{q}) \vec{V}^*(\vec{q}) = \frac{(2\pi)^3 N}{V_{\text{cell}}} \sum_{n, \vec{G}} |F_n(\vec{G})|^2 \delta(\vec{q} + n\vec{Q} - \vec{G}). \quad (11)$$

The volume of the unit cell is V_{cell} , and N is the number of unit cells in the crystal.

Thus, we obtain a series of Bragg peaks located at the positions $\vec{q} = \vec{G} - n\vec{Q}$. The $n=0$ terms in the sum are the Bragg peaks of the underlying average structure. If the amplitudes are small, then the factor $J_0(\vec{q} \cdot \vec{A}(i))$ in Eq. (10) can be absorbed into the Debye-Waller factor; and the intensities of the first order ($n=\pm 1$) CDW satellites become quadratic in the scattering vector \vec{q} . For constant and small

amplitudes, $\vec{A}(i) = \vec{A}$, the cross section is

$$\left. \frac{d\sigma}{d\Omega} \right|_{n=1} \cong \frac{(2\pi)^3 N}{V_{\text{cell}}} \frac{N}{4} (\vec{q} \cdot \vec{A})^2 |F_0(\vec{G})|^2 \delta(\vec{q} - \vec{Q} - \vec{G}). \quad (12)$$

We see that the intensity of the progenic satellite depends upon the structure factor of its parent Bragg reflection at the point \vec{G} , and not upon the structure factor at the point \vec{q} where the peak is observed. An analogous result is obtained in the flexible response of a magnetic system to a spin-density wave (SDW), where the magnetic diffraction peaks depend on the atomic magnetic form factor at \vec{G} and not at \vec{q} .

Since the displacements of Eq. (2) do not preserve the fourfold rotation axis about \vec{c} and the mirror planes of the underlying tetragonal structure, the CDW satellites will not exhibit the same symmetries as their parent reflections, in contradiction with our measurements on KCP single crystals. If the displacements given by Eq. (2) are to be viable, they must be accompanied by the assumption that KCP single crystals have a tendency to grow with four equally populated \vec{Q} domains. An alternative approach is to only consider displacements which preserve the tetragonal symmetry. This can be done by writing the displacements in terms of four \vec{Q} vectors such that

$$\vec{r}_{\Gamma}(i, j) = \vec{R}_{\Gamma}(i, j) + \frac{\vec{A}(i)}{4} \sum_{\alpha=1}^4 \sin \vec{Q}_{\alpha} \cdot \vec{R}_{\Gamma}(i, j), \quad (13)$$

with

$$\vec{Q}_{\alpha} = (\pi/a)(\pm \hat{x} \pm \hat{y}) + Q_z \hat{z}. \quad (14)$$

By employing the Jacobi-Anger expansion four times, we find that Eq. (9) must be generalized to

$$\vec{q} + \sum_{\alpha=1}^4 n_{\alpha} \vec{Q}_{\alpha} = \vec{G}. \quad (15)$$

Since any $\vec{G}(h', k', l) = \vec{G}(h, k, l) + \sum n_{\alpha} \vec{Q}_{\alpha}$ for a suitable choice of the n_{α} , we reach the conclusion that the intensity of a given satellite depends on a weighted sum of parent structure factors over the plane of constant l from which it is derived. A similar conclusion holds for the Bragg peaks of the underlying structure. Although this approach provides a useful and intriguing picture, the problem can be circumvented by writing Eq. (13) as

$$\vec{r}_{\Gamma}(i, j) = \vec{R}_{\Gamma}(i, j) + \vec{B}(i) \sin \vec{Q} \cdot [\vec{T}_{\Gamma} + \vec{u}_z(i)], \quad (16)$$

with

$$\vec{B}(i) = \vec{A}(i) \cos[\pi u_x(i, j)/a] \cos[\pi u_y(i, j)/a]. \quad (17)$$

This leads to the scattering cross section

$$\frac{d\sigma}{d\Omega} = \frac{(2\pi)^3 N}{V_{\text{cell}}} \sum_{n, \vec{G}} |F_n(\vec{q})|^2 \delta(\vec{q} + n\vec{Q} - \vec{G}), \quad (18)$$

with the generalized structure factors

$$F_n(\vec{q}) = \sum_{i,j} b_i e^{i\vec{q} \cdot \vec{u}(i,j)} e^{inQ_z u_z(i)} J_n(\vec{q} \cdot \vec{B}(i)). \quad (19)$$

We note that the explicit dependence of the structure factor on \vec{G} has been lost due to the implicit sum involved in writing Eq. (16).

B. Molecular or rigid response

This model is appropriate when the molecular bonding between atoms is much stronger than their individual interaction with the CDW or, equivalently, when the response of one atom in the molecular unit to the CDW predominates. We let the index m denote both the m th molecular unit and the coordinates of the reference position in the m th molecule which determines its response to the CDW. The indices i and j run over all atoms in the m th molecule. Considering only displacements which preserve the tetragonal symmetries, we write

$$\vec{r}_{\Gamma}(m, i, j) = \vec{R}_{\Gamma}(m, i, j) + \vec{A}(m) \sin \vec{Q} \cdot [\vec{T}_{\Gamma} + \vec{u}_z(m)]. \quad (20)$$

From Eq. (20) one obtains the cross section Eq. (18), with the generalized structure factor

$$F_n(\vec{q}) = \sum_m J_n(\vec{q} \cdot \vec{A}(m)) e^{inQ_z u_z(m)} f_m(\vec{q}). \quad (21)$$

The $f_m(\vec{q})$ are the molecular structure factors

$$f_m(\vec{q}) = \sum_{i,j} b_i e^{i\vec{q} \cdot \vec{u}(i,j)}. \quad (22)$$

In the limit of one molecule per unit cell with small amplitude, the CDW satellite cross section is given by

$$\left. \frac{d\sigma}{d\Omega} \right|_{n=1} \cong \frac{(2\pi)^3 N}{V_{\text{cell}}} \frac{N}{4} (\vec{q} \cdot \vec{A})^2 |f(\vec{q})|^2 \delta(\vec{q} - \vec{Q} - \vec{G}), \quad (23)$$

where $f_m = f$. Here we see that the intensity depends upon a structure factor evaluated at the point \vec{q} where the peak is observed, in analogy with the rigid response of a magnetic system to a SDW.

III. KCP AND ITS RESPONSE TO THE CDW

The $(1, \bar{1}, 0)$ reciprocal-lattice plane of KCP is shown in Fig. 2. The indexing is based on a tetragonal unit cell in which there are two formula units. The solid circles lying at integral values of the indices (h, k, l) are the Bragg peaks of the underlying structure. The fundamental CDW satellites (crosses in the figure) occur at values of the scattering vector

$$\vec{q} = \vec{G}(h, k, l) \pm \vec{Q}. \quad (24)$$

The z component of \vec{Q} is observed to be twice the Fermi wave vector k_F , as calculated by removing 0.60 electrons per unit cell from the "one-dimensional" d_{z^2} band formed from the Pt atoms and placing them on the 0.60 Br atoms per unit cell. Thus,

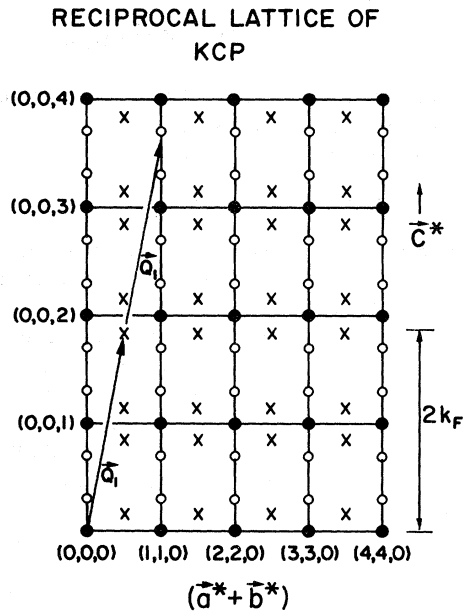


FIG. 2. Reciprocal lattice of KCP. The solid circles are the Bragg peaks of the average structure, the k 's are the first-order ($n = \pm 1$) CDW satellites, and the open circles locate the second-order ($n = \pm 2$) CDW satellites.

$$Q_z = 2k_F = 1.70(2\pi/c). \quad (25)$$

The fact that the observed satellite reflections lie along the lines $(h + 0.5, k + 0.5, z)$ necessitates the conclusion that the displacements associated with adjacent strands of $\text{Pt}(\text{CN})_4$ must be 180° out of phase.

In Fig. 3 we show the result of a scan on a deuterated crystal at 7 K through the two satellite peaks at $(0.5, -0.5, 4.30)$ and $(0.5, 0.5, 4.30)$ along the line in reciprocal space connecting these points. The widths of these peaks are somewhat broader than the instrumental resolution, indicating that three-dimensional long-range order is not complete in this crystal at 7 K. In order to determine the displacement amplitudes, one needs the integrated

TABLE I. Experimentally determined neutron structure factors of various first-order CDW satellites in a deuterated single crystal at 7 K. The $\sim 10\%$ uncertainty in the scaling factor has not been included.

Reflection	$ F_{\text{exp}} ^2$ (barns)
$(0.5, 0.5, 1.7)$	$(3.88 \pm 0.24) \times 10^{-2}$
$(0.5, 0.5, 2.3)$	$(10.52 \pm 0.34) \times 10^{-2}$
$(0.5, 0.5, 3.7)$	$(23.14 \pm 0.56) \times 10^{-2}$
$(0.5, 0.5, 4.3)$	$(36.50 \pm 0.80) \times 10^{-2}$
$(0.5, 0.5, 5.7)$	$(46.81 \pm 0.98) \times 10^{-2}$
$(0.5, 0.5, 6.3)$	$(51.11 \pm 1.16) \times 10^{-2}$
$(0.5, 2.5, 4.7)$	$(9.78 \pm 0.67) \times 10^{-2}$
$(0.5, 5.5, 5.7)$	$(16.63 \pm 0.82) \times 10^{-2}$

intensities of the satellites, I , and ultimately, the structure factors, which are related by

$$I = C |F_{\text{exp}}|^2 E, \quad (26)$$

where C is an instrumental constant and E is the absorption factor. The experimental structure factors for a series of satellite peaks in deuterated KCP at 7 K are listed in Table I. The errors are based on counting statistics only and do not reflect additional systematic uncertainties. The satellite intensities at $(0.5, 0.5, 4.3)$ and $(0.5, 0.5, 6.3)$ were also measured in a hydrogenous sample at 7 K. Within experimental error, the integrated intensities agreed with those obtained in the deuterated sample.

In principle, one could decide on the appropriate model for describing the atomic response to the CDW by measuring a large number of satellite reflections and employing the standard techniques of crystallography. Since our number of observations is limited, we postulate a solution and use the experimental data as justification. Since the CDW is presumably in a d -electron band derived from the Pt atoms, the Pt atoms must respond. From the fact that the satellite peaks become noticeably asymmetric at elevated temperatures, one can conclude that there must be a response of off- c -axis atoms.¹⁴ The simplest response consistent with this observation is to allow the CN ligands to displace with the Pt atoms. As previously mentioned, the satellite intensities are nearly the same for hydrated and deuterated samples; thus, the waters cannot play a major role in the displacement. As most of the waters are strongly bound to the K, it seems plausible to assume that the total response of the ionic lattice (K, Br, and H_2O) is of secondary importance. We are thus led to postulate a displacement involving only the nearly planar $\text{Pt}(\text{CN})_4$ complexes. Whether the response of each complex

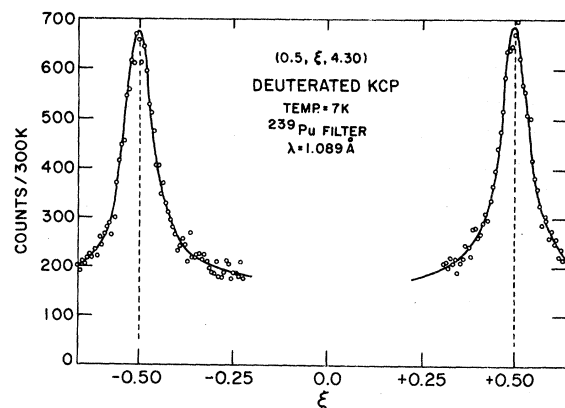


FIG. 3. Scan through the first-order CDW satellites located at $(0.5, -0.5, 4.30)$ and $(0.5, 0.5, 4.30)$ along the line in reciprocal space connecting these points.

TABLE II. The coordinates \vec{u} , and rms thermal displacements $\vec{\rho}$ of the $\text{Pt}(\text{CN})_4$ complexes. The quoted distances are in fractional parts of the appropriate lattice parameter.

m	Atom	u_x	u_y	u_z	ρ_x	ρ_y	ρ_z
1	Pt	0	0	0	0.0071 ± 0.0013	0.0071 ± 0.0013	0.0117 ± 0.0015
1	C	0.2033 ± 0.0002	0	-0.0052 ± 0.0008	0.0093 ± 0.0006	0.0108 ± 0.0005	0.0174 ± 0.0020
1	N	0.3212 ± 0.0001	0	-0.0133 ± 0.0008	0.0081 ± 0.0006	0.0135 ± 0.0004	0.0188 ± 0.0015
2	Pt	0	0	0.5003 ± 0.0008	0.0071 ± 0.0013	0.0071 ± 0.0013	0.0117 ± 0.0015
2	C	0.1438 ± 0.0003	0.1438 ± 0.0008	0.5013 ± 0.0008	0.0095 ± 0.0008	0.0095 ± 0.0008	0.0174 ± 0.0020
2	N	0.2273 ± 0.0002	0.2273 ± 0.0002	0.5095 ± 0.0008	0.0106 ± 0.0007	0.0106 ± 0.0007	0.0188 ± 0.0015

is rigid or partly flexible is difficult to determine. We will assume a totally rigid response and look for any resulting inconsistencies.

With the assumption of a small constant amplitude for both complexes, the CDW satellite structure factor, Eq. (21), takes the form

$$F_{\pm 1}(\vec{q}) = J_{\pm 1}(\vec{q} \cdot \vec{A}) [f_1(\vec{q}) + f_2(\vec{q}) e^{\pm i k_F c}] \\ \cong \pm \frac{1}{2} (\vec{q} \cdot \vec{A}) S(\vec{q}). \quad (27)$$

The coordinates and thermal parameters used to calculate the reduced structure factor, $S(\vec{q})$, are given in Table II, and were determined by a standard crystallographic refinement on the underlying Bragg reflections.¹⁵ From Eq. (27), one expects a plot of $|F_{\text{exp}}(\vec{q})|^2 / |S(\vec{q})|^2$ to be quadratic in qz . Such a plot is shown in Fig. 4. The solid curve is parabolic and exemplifies the good agreement between model and experiment. We find for the value of the corresponding displacement amplitude,

$$\vec{A} = (0.0047 \pm 0.0005) \vec{c}. \quad (28)$$

We would like to point out the significance of the peak at $(0.5, 2.5, 4.7)$, which is derived from a plane where l is odd. If one were to assume that only the Pt atoms displaced, the reduced structure factor would be zero for this peak. Since the rms value for A is an appreciable fraction of the c -axis rms thermal displacements listed in Table II, one should examine the errors incurred due to the omission of the $J_0(\vec{q} \cdot \vec{A})$ factor in the crystallographic refinement. The depression of the $(0, 0, 8)$ structure factor by J_0 is 2%, while the thermal motion depresses it 30% at 7 K; hence, the thermal parameters obtained from the standard crystallographic refinement are not expected to be significantly altered by the CDW response. Since the rms CDW displacements are smaller than the thermal displacements, there is no contradiction to our assumption of a totally rigid response for the $\text{Pt}(\text{CN})_4$ complexes.

In conclusion, we have shown that the satellite reflections in KCP can be adequately explained by a rigid response of the $\text{Pt}(\text{CN})_4$ complexes to the

electronic CDW, with a displacement amplitude of $(0.0047 \pm 0.0005)c$, or $0.027 \pm 0.003 \text{ \AA}$. Although the interaction of the $\text{Pt}(\text{CN})_4$ chains with the ionic lattice of K, Br, and H_2O ensures that displacements on adjacent chains will be 180° out of phase, the ionic lattice does not appear to respond appreciably to the CDW.

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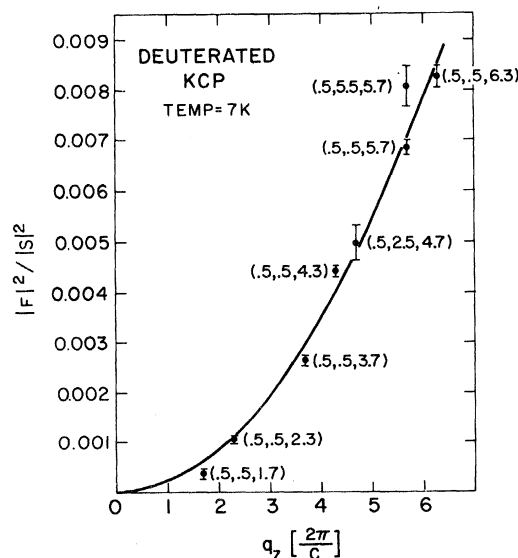


FIG. 4. The quadratic dependency of the first-order CDW satellites on q_z . The solid curve is drawn for a displacement amplitude of $0.0047c$.

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