A Neutron Scattering Study of Magnetic Excitations in the Spin Ladder (VO)$_2$P$_2$O$_7$

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Abstract

In this letter we report results from inelastic neutron scattering experiments on powder samples of vanadyl pyrophosphate, (VO)$_2$P$_2$O$_7$. We see evidence for three magnetic excitations, at 3.5 meV, 6.0 meV and 14 meV. The intensity of the 3.5 meV mode is strong near $Q = 0.8\ \text{Å}^{-1}$, consistent with the one-magnon gap mode reported previously and predicted by the spin-ladder model. The magnetic scattering at 14 meV may be due to the top of the one-magnon band or the two-magnon continuum predicted in the ladder model. The 6.0 meV mode also peaks in intensity at $Q = 0.8\text{Å}^{-1}$. This mode has not been reported previously and was not anticipated by existing theoretical treatments of the spin ladder.
I. INTRODUCTION

The antiferromagnetic insulator (VO)$_{2}$P$_{2}$O$_{7}$ (abbreviated VOPO) is widely considered to be the prototypical realization of a Heisenberg spin ladder. Heisenberg spin ladders are interesting theoretically as intermediaries between 1D Heisenberg antiferromagnets and 2D systems. Theoretical studies of this model have concluded that S=1/2 spin ladders have a gap if they have an even number of chains, but are gapless for an odd number of chains. This is reminiscent of the Haldane result that half-integer-spin chains are gapless but integer-spin chains have energy gaps. Studies of hole-doped 2-chain ladders in the t-J model find that strong d-wave hole pairing takes place on equal-strength ladders, so analogues of the high-Tc superconductors might be found in hole-doped spin ladders. Additional examples of spin ladders are known, including a Sr-Cu-O series, a La-Cu-O series and Cu$_{2}$(C$_{5}$H$_{12}$N$_{2}$)$_{2}$Cl$_{4}$. The subject of spin ladders was reviewed recently by Dagotto and Rice.

The magnetic properties of VOPO have been interpreted in terms of the 2-chain Hamiltonian

\[ H = J_{∥} \sum_{<ij>} \vec{S}_{i} \cdot \vec{S}_{j} + J_{⊥} \sum_{<ij>} \vec{S}_{i} \cdot \vec{S}_{j}. \]

(1)

This model gives an accurate fit to the VOPO magnetic susceptibility with the parameters $J_{∥} = J_{⊥} = 7.8$ meV. Theoretical studies of the ladder model predict an energy gap of $E_{\text{gap}} = 0.5037J$, hence the susceptibility results imply $E_{\text{gap}} = 3.9$ meV. A pulsed neutron scattering experiment on VOPO powder observed a gap of 3.7(2) meV, consistent with this expectation.

Although these aspects of VOPO are consistent with the Hamiltonian (1), many unanswered questions remain concerning the dispersion relations, spectral weight, and detailed band structure of the magnetic excitations. We have carried out triple-axis inelastic neutron scattering studies of VOPO to investigate these issues in more detail. In addition to confirming the existence of the gapped spin wave mode observed previously, our results
reveal a new and unexpected magnetic excitation.

The spectrum of low-lying spin waves in the 2-chain ladder model has been discussed at length in the literature \[2, 4, 11\]. The one-magnon band can be approximately described by the function \[7\]

\[
\omega(k) = \left[ \omega(0)^2 \cos^2(k/2) + \omega(\pi)^2 \sin^2(k/2) + c_0^2 \sin^2(k) \right]^{1/2}
\]

where \(k\) is the one-dimensional wavevector in units in which the intrachain ion separation is set to unity. This dispersion relation for the one-magnon band \(\omega(k)\) is shown in Fig.1 together with the lower edges of the two-magnon and three-magnon continua. (These are the lowest-energy sums \(\omega(k_1) + \omega(k_2)\) and \(\omega(k_1) + \omega(k_2) + \omega(k_3)\) with the constraint \(\sum_i k_i = k\).) The value \(J=7.0\) meV was chosen to match the energy gap observed in the present work, as discussed below, and our dispersion relation parameters for \(J_\perp = J_\parallel = J\) are \(\omega(0) = 1.72J, \omega(\pi) = 0.5037J\) and \(c_0 = 1.38J\); these values were motivated by finite lattice results.

The lowest-lying one-magnon excitation occurs at the one-dimensional antiferromagnetic point \(k = \pi\), with an energy of \(\omega(\pi) = E_{\text{gap}}\). Exact calculations of the dynamical structure factor \(S(k, \omega)\) on a \(2 \times 8\) lattice \[11\] indicate that the spectral weight of the one-magnon band peaks strongly near \(k = \pi\), so this should be the most prominent feature seen in neutron scattering.

The two-magnon states span a continuum of levels, beginning at \(2E_{\text{gap}}\) at \(k = 0\). As seen in Fig.1, the lower edge of the two-magnon continuum crosses the one-magnon band at an intermediate \(k \approx 0.3\pi\), and for smaller \(k\) these two-magnon states are the lowest-lying excitations. Similarly there is a three-magnon continuum, also shown in Fig.1, which contains the first \(k = \pi\) excitation above \(E_{\text{gap}}\) in the ladder model; this three-magnon continuum begins at \(3E_{\text{gap}}\).

Numerical calculations of the structure factor \(S(k, \omega)\) \[11\] indicate that the two-magnon states have moderate spectral weight near \(k = \pi\), at \(E \approx 2.2J\) (\(\approx 15\) meV in VOPO), and the one-magnon band may also be observable near \(k = 0\), at \(E \approx 1.7J\)
(≈ 12 meV in VOPO). Both excitations have $S(k, \omega) \approx 0.1 S(k = \pi, \omega = E_{\text{gap}})$ of the gap mode. The $S(k, \omega)$ of the three-magnon continuum at $k = \pi$, $E \approx 1.5J$ (≈ 11 meV) is about a factor of five weaker than these higher excitations.

II. EXPERIMENTAL DETAILS

To prepare $(\text{VO})_2\text{P}_2\text{O}_7$, stochiometric amounts of $\text{V}_2\text{O}_5$ (44.1 grams, 99.99%) and $\text{NH}_4\text{H}_2\text{PO}_4$ (55.9 grams, 99.9%) powder were mixed and loaded into a platinum crucible. The mixed powders were heated to 500°C at 1°C/minute and held at 500°C for 6 hours. This initial heating allowed the ammonia and water to escape from the crucible during the decomposition of $\text{NH}_4\text{H}_2\text{PO}_4$ to $\text{P}_2\text{O}_5$. The prereacted mixture was heated further to a temperature of 1100°C and the resulting liquid was allowed to homogenize for several hours. The platinum crucible was removed from the furnace at 1100°C and the liquid was “cast” into a cold, shallow platinum crucible measuring 10 x 4 x 2 cm$^3$. The rapid cooling of the $\text{V}_2\text{O}_5 \cdot \text{P}_2\text{O}_5$ liquid resulted in the formation of a homogenous glass with a dark green to black color. The above steps were all performed in air.

The shallow platinum crucible containing the $\text{V}_2\text{O}_5 \cdot \text{P}_2\text{O}_5$ glass was then placed in a tube furnace in which the oxygen partial pressure could be controlled. A mixture of 0.1% oxygen and 99.9% argon was passed through the furnace at 40 cc/minute, and this rate and oxygen partial pressure were maintained during the entire crystal growth procedure. An independent oxygen sensor was used to monitor the oxygen partial pressure in the furnace. The $\text{V}_2\text{O}_5 \cdot \text{P}_2\text{O}_5$ glass was heated to 1050°C, maintained at that temperature for 6 h, and then cooled at 1°C/hr to 480°C, after which the furnace was turned off. The resulting material consisted of several hundred mm sized single crystals of $(\text{VO})_2\text{P}_2\text{O}_7$ embedded in a polycrystalline matrix of $(\text{VO})_2\text{P}_2\text{O}_7$ and a small amount (≈ 5-15%) of an additional phase, tentatively identified as $\text{V(PO}_3)_3$ by neutron powder diffraction [12].

Inelastic neutron scattering measurements were carried out using the HB1A and HB3 triple-axis spectrometers at the High Flux Isotope Reactor at Oak Ridge National Lab-
oratory [13]. The sample consisted of 18 grams of finely ground VOPO powder sealed in a 0.5” diameter thin-walled Al cylinder under a helium atmosphere. For the initial experiment on the HB1A spectrometer the sample was mounted in a standard He closed-cycle refrigerator with a temperature range of 10K to 300K. A double-crystal pyrolytic graphite (PG) (002) monochromator (M) was used to provide an incident neutron beam with a fixed energy of 14.7 meV. The incident beam intensity was monitored using a fission counter, and contamination of the beam by higher-order Bragg reflections was removed using the standard PG filter method. Neutrons scattered by the sample (S) were reflected by a PG(002) analyzer (A) into a $^3$He detector (D). Effective beam collimations pre-M, M-S, S-A and A-D of 40'-40'-40'-70' or 40'-20'-20'-70' were used.

A second experiment utilizing the HB3 spectrometer employed PG(002) monochromator and analyzer crystals with a fixed scattered neutron energy of 14.7 meV. The collimation was 40'-40'-40'-120', and the scattered beam was passed through a PG filter. For this experiment the sample was mounted in a pumped He cryostat capable of reaching 1.5K.

VOPO has a slightly monoclinic unit cell which has lattice parameters $a = 7.728$ Å, $b = 16.589$ Å, $c = 9.580$ Å, and $\beta = 89.98^\circ$ at $T=296K$ [14]. The ladder is composed of $S=1/2 V^{4+}$ ions in -(VO)-(VO)- chains along the $a$ axis and $V^{2-O^-}_O$ V rungs oriented along the $b$ axis. The average V-V distance along the chain is approximately equal to $a/2$. Therefore in VOPO the one-dimensional antiferromagnetic points $k = n\pi$ (with $n$ odd) occur in sheets with $Q_a = nQ_\pi$, where $Q_\pi = 0.8134$ Å$^{-1}$.

In scattering from powder the experimental momentum transfer to the sample $Q = |\vec{Q}|$ does not correspond to the one-dimensional momentum $k$ of the excitations observed. Instead $k$ is the projection of $\vec{Q}$ on the ladder axis, and in the powder one averages over all orientations. Therefore for a given $Q$ one can excite all ladder modes with the observed energy transfer and $k \leq Q$. In particular, one expects the gap mode, which has $k = \pi$, to appear first at $Q = Q_\pi$ as $Q$ is increased, and to persist to higher $Q$ due to the powder
average. The scattering intensity observed at \((Q, \omega)\) involves the density of states as well as \(S(k, \omega)\).

III. RESULTS

Figure 2 shows the results of constant-\(Q\) scans at \(Q = Q_\pi\), taken at HB1A. The data have been scaled to a fixed monitor value and have been corrected for the constant-\(E_i\) kinematical factor \(k_f \cot \theta_A\). The data at 10.8K clearly show two peaks in the scattering. The peak intensities are drastically reduced at higher temperatures, as illustrated by the data at 41K, which provides strong evidence that they are both magnetic in origin. The lower peak at 3.48(3) meV is interpreted as the one-magnon gap mode previously reported at 3.7(2) meV by Eccleston et al. [9]. Their slightly higher energy may arise from their broader \(Q\) acceptance. The 3.5 meV peak observed in the present work has a FWHM significantly greater than the instrumental resolution limit of 0.7 meV.

The mode at \(E = 6.0\) meV has not been reported previously. It appears narrower in energy than the 3.5 meV mode, with a FWHM only slightly larger than the instrumental resolution; this suggests that the band is rather flat, possibly corresponding to a relatively localized mode. Comparison with the theoretical magnon bands in Fig.1 shows that this excitation is not predicted by the simple Heisenberg spin-ladder model (1).

Figure 3 shows the \(Q\) dependence of the intensities of the 3.5 meV and 6.0 meV modes. Both modes show very similar behavior, with an onset and peak at \(Q = Q_\pi\). This is precisely what one would expect for the lowest-energy one-magnon gap mode. The fact that the 6.0 meV mode peaks at a value of \(Q\) characteristic of VOPO provides strong evidence that it actually is a VOPO excitation, and does not arise from a contaminant phase in the sample. Both modes also show a slight increase in intensity in the vicinity of \(Q = 2Q_\pi\), and a gradual falloff in intensity at higher \(Q\). The scattering intensity from a powder sample is not easily interpreted beyond the first Brillouin zone, but the diminishing intensity with increasing momentum transfer is expected because of the reduction of the magnetic
form factor at large $Q$. For the $V^{4+}$ ion in VOPO, $|f_{\text{mag}}(3Q_\pi)|^2 \approx 0.5|f_{\text{mag}}(Q_\pi)|^2$.

Constant $Q$ scans at $Q = 3Q_\pi$ and $Q = 5Q_\pi$ at a temperature of 1.6K are presented in the lower panel of Fig.4. The scans were carried out at HB3 with fixed scattered neutron energy, which allowed measurements over a wide range of energy transfers. The scattering at both wavevectors exhibits a broad maximum near 13 meV. The origin of this broad scattering is unknown, but since magnetic scattering at $Q = 5Q_\pi$ is expected to be weak, it is probably not magnetic in origin. Unfortunately, kinematic restrictions did not allow the same energy scan at lower $Q$, particularly at $Q = Q_\pi$ where the magnetic features should be most prominent. Inspection of the lower panel of Fig.4 shows that the $3Q_\pi$ scan has features which were not observed in the $5Q_\pi$ scan. In repetitions (not shown) of the $3Q_\pi$ scan at $T=45K$ and $T=100K$ the extra features are not clearly visible, and only the broad maximum remains. The $Q$ and $T$ dependence of the scattering strongly suggests that the additional features seen at $3Q_\pi$ arise from magnetic excitations. To investigate this possibility further the $Q = 5Q_\pi$ intensity was subtracted from the $Q = 3Q_\pi$ intensity on a point-by-point basis; the result is shown in the upper panel of Fig.4. The solid line is a fit of this difference spectrum to a sum of three Gaussians and a constant background, revealing features near 3.6 meV and 6.5 meV, approximately consistent with the magnetic excitations observed at $Q_\pi$. The fit also shows a higher-energy peak at 14.3 meV. Eccleston et al. [9] also saw indications of enhanced scattering near 15 meV at $Q = 0.8 \, \text{Å}^{-1}$, although the details were unclear.

Since 14 meV is close to the predicted onset of the $k = \pi$ two-magnon continuum as well as the maximum energy of the one-magnon band (see Fig.1) in the ladder model, it is tempting to associate the magnetic scattering with either or both of these features. However, a detailed comparison of experiment with the excitation spectrum predicted by (1) is problematical, since it did not anticipate the 6 meV mode.
IV. DISCUSSION

In this experiment we have found clear evidence for two low-lying magnetic excitations in VOPO, at energies of approximately 3.5 meV and 6.0 meV, and some additional magnetic scattering near 14 meV. The low-lying excitations peak strongly at \( Q = 0.8 \, \text{Å}^{-1} \), near the \( k = \pi \) point in VOPO, which suggests that these are \( k = \pi \) modes of the VOPO spin ladder. Although the 3.5 meV mode and the 14 meV scattering are similar to expectations in the spin-ladder model, the presence of two low-lying modes at \( k = \pi \) is unexpected, and appears to argue against the validity of the simple spin-ladder model (1) for VOPO.

Of course one possibility is that the theoretical predictions we associate with the spin ladder, such as the spectrum and continua shown in Fig.1, are inaccurate. Although it appears highly unlikely that two low-lying excitations arise at \( k = \pi \) in the model, these conclusions are based on Lanczos studies of relatively small clusters (up to \( 2 \times 12 \)), and careful theoretical investigations of larger spin ladders would be useful.

Assuming that the theoretical interpretation of the model is accurate, we conclude that there are magnetic excitations in VOPO which are not consistent with the Heisenberg spin-ladder Hamiltonian (1). In view of this apparent disagreement we should recall the assumptions which led to (1) and determine whether they are justified. The Heisenberg model assumes that each magnetic ion can be treated as a localized spin with \( S = 1/2 \), interacting with neighboring ions through an isotropic interaction. Whether or not this is true in practice depends on the ground state and low-lying excited states of the magnetic ion.

An isolated \( V^{4+} \) ion in an octahedral field, as is approximately realized by the O octahedra in VOPO, has a \( t_{2g} \) triplet ground state \cite{15}; this triplet is split by the spin-orbit interaction and by departures from octahedral symmetry, including the formation of \( \text{(VO)} \) units along the chains \cite{14}. For isolated \( V^{4+} \) ions in \( \text{Al}_2\text{O}_3 \) the single-ion excited \( t_{2g} \) levels are known to lie just 3.5 meV and 6.6 meV above the ground state \cite{16}, comparable
to the energy scales of the magnetic modes observed in VOPO.

This raises the possibility that one or more of the magnetic modes observed in VOPO could be crystal field levels, which might explain the peak at 6 meV. There is experimental evidence against this possibility, however; a single-ion excitation should not display a strong dependence of the scattering intensity on $Q$ (other than the magnetic form factor). Instead we have observed a strong peak at $Q_\pi$ in the constant-E scan (Fig. 3), which suggests that the 6 meV excitation is part of a band of delocalized ladder excitations. Of course this band could involve excited $t_{2g}$ orbital levels, in which case VOPO should be described by a multiband Hubbard model with $t_{2g}$ $V^{4+}$ orbitals interacting through intermediate O levels.

Although low-lying $t_{2g}$ $V^{4+}$ orbitals may indeed be present in VOPO, magnetic susceptibility measurements argue against their presence at these low energy scales. The observed high temperature limit of the susceptibility (known to about 350 K [1]) is consistent with that expected for paramagnetic spins with $S=1/2$ and $g=2$. The presence of additional $t_{2g}$ degrees of freedom would give a much larger susceptibility as the temperature approached the orbital gap. Thus there is no indication of excited $t_{2g}$ levels in VOPO to $\approx 30$ meV. Further experimentation, including infrared spectroscopy and EPR measurements, is desirable to locate the excited $t_{2g}$ orbital levels of $V^{4+}$ in VOPO.

A more likely possibility is that VOPO actually can be modelled accurately as an $S=1/2$ spin ladder at low energy scales (hence the susceptibility is approximately correct), but there are important additional spin interactions that split the one-magnon band into two bands, which we observe at 3.5 meV and 6.0 meV at $k=\pi$. There are various possibilities for such terms, including ladder-ladder interactions, chain dimerization, departures from isotropy, next-nearest-neighbor interactions and so forth. Two examples of such interactions are well known. Planar anisotropy in the spin Hamiltonian could lead to different gaps for “in-plane” and “out-of-plane” spin-wave modes; this has been observed in $S=1$ antiferromagnetic chain materials [4]. A large next-nearest-neighbor
interaction is believed to be present in the CaV$_4$O$_9$ spin lattice [18]; similar interactions may be present in VOPO as well.

In conclusion, it appears that a generalization of the simple Heisenberg spin-ladder Hamiltonian (1) will be required to describe the magnetic properties of VOPO. The correct interpretation of the magnetic excitations would be facilitated by more detailed experimental studies of the low-lying bands, in particular by inelastic neutron scattering studies of VOPO single crystals. The technical difficulties of growing adequate single crystals of VOPO have precluded such studies to date. We hope to carry out such an experiment in the near future.
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Figure 1. Predicted spin waves in (VO)$_2$P$_2$O$_7$ with $J_\perp = J_\parallel = 7.0$ meV. The one-magnon band (solid) and the onset of the two-magnon and three-magnon continua are shown.

Figure 2. Inelastic scattering from (VO)$_2$P$_2$O$_7$ at $Q = 0.8134$ Å$^{-1}$, at temperatures of T=10.8K and 40.8K, measured at HB1A with $E_i = 14.728$ meV and collimation 40′-20′-20′-70′. The gap mode at 3.5 meV and the 6.0 meV mode are clearly visible in the 10.8K data. The corrected neutron counts (see text) are normalized to counts observed in 1250 monitor units; each monitor unit corresponds to about one second of counting time.

Figure 3. Constant energy scans of scattering intensity versus momentum transfer $Q$ at $\omega = 3.5$ meV (upper panel) and $\omega = 6.0$ meV (lower panel), normalized to 500 monitor units. The dashed vertical lines show the values of $Q = nQ_\pi$ corresponding to one-dimensional nuclear ($n$ even) and magnetic ($n$ odd) zone centers. Note the strong peaking at $Q = Q_\pi$ in both cases. Measurements were done at HB1A with fixed incident energy $E_i = 14.728$ meV and collimation 40′-40′-40′-70′.

Figure 4. Constant $Q$ scans at $Q = 3Q_\pi$ and $Q = 5Q_\pi$ at T=1.6K(lower panel) and the subtracted difference scattering (upper panel). The scattering intensity is normalized to 100 monitor units. The line in the lower panel is a guide to the eye through the $5Q_\pi$ data. The solid line in the upper panel is a least-squares fit of the difference to a sum of three Gaussians and a constant background, with fitted centers equal to 3.62(13) meV, 6.46(16) meV and 14.32(22) meV. The measurements were carried out at HB3 using a fixed scattered neutron energy $E_f = 14.7$ meV and collimation 40′-40′-40′-120′.
Figure 1.
Figure 2.

(VO)$_2$P$_2$O$_7$

$Q=0.8134$ Å$^{-1}$

$T=11K$

$T=41K$

intensity (corrected) / M1250

energy transfer (meV)
Figure 3.
Figure 4.