Fluctuations and correlations in a frustrated $S=\frac{1}{2}$ square lattice with competing ferromagnetic and antiferromagnetic interactions studied by muon-spin relaxation

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Zero and longitudinal field μSR measurements in $Pb_2VO(PO_4)_2$ and $BaCdVO(PO_4)_2$, two prototypes of the frustrated $S=1/2$ square lattice model with competing ferromagnetic and antiferromagnetic interactions, are presented. Both systems are observed to undergo a phase transition to a long-range magnetic order at $T_N \simeq 3.46$ K, for $Pb_2VO(PO_4)_2$, and at $T_N \simeq 0.99$ K, for $BaCdVO(PO_4)_2$. In $Pb_2VO(PO_4)_2$ both the temperature dependence of the order parameter and the longitudinal relaxation rate above T_N are consistent with a two-dimensional XY model, as it is found for $Sr_2CuO_2Cl_2$. On the other hand, for $BaCdVO(PO_4)_2$, which lies very close to the magnetically disordered region of the phase diagram where a bond-nematic order was predicted, a peculiar logarithmic increase in the relaxation is observed above T_N. In both systems a rather broad distribution of internal fields at the muon sites is noticed below T_N. The origin of this distribution is discussed in the light of the μSR experiments already performed on $S=1/2$ frustrated antiferromagnets on a square lattice.

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I. INTRODUCTION

Strongly correlated electron systems with competing interactions are known to show rather rich phase diagrams, with crossovers or phase transitions which depend on the relative magnitude of the competing energy scales.[1] In certain cases rather complex scenarios are observed together with the insurgence of novel phases of matter, a situation typically found in frustrated magnets where, for instance, spin ice or spin liquid ground states arise.[2] In these insulating systems either the geometry of the underlying spin lattice or the geometry of the interactions causes the suppression of the long-range magnetic order and the onset of exotic phases. For instance, for a frustrated $S=1/2$ square lattice with competing ferromagnetic and antiferromagnetic interactions[3] (hereafter QFFMSL for short) it has been proposed that for exchange couplings yielding to the disappearance of long-range magnetic order, a nematic order could be present.[4] QFFMSL are characterized by a ferromagnetic nearest-neighbor (n.n.) exchange coupling J_1, along the sides of the square spin lattice, competing with the antiferromagnetic next-nearest-neighbor (n.n.n.) coupling J_2, along the diagonals. For a frustration ratio $-0.4 \geq J_2/J_1 \geq -0.7$ long-range magnetic order should disappear and a nematic order of spin bonds should arise.[4] In this phase, the individual spins do not carry any orientational order but the traceless tensor

$$\mathcal{O}^{\alpha\gamma} = \frac{1}{2}(S_i^\alpha S_j^\gamma + S_i^\gamma S_j^\alpha) - \frac{1}{3}\delta^{\alpha\gamma}\langle \vec{S}_i \vec{S}_j \rangle, \qquad (1)$$

with $\alpha, \gamma = x, y, z$ and i, j running over spin-lattice sites, does order and gives rise to stripy correlations in the plane. This interesting theoretical result lacks of any experimental confirmation, basically because there is no material characterized by a frustration ratio $J_2/J_1 \simeq -0.5$. Nevertheless, in the last years there has been a considerable effort to synthesize novel vanadates, containing V^{4+} $S=1/2$ ions, which have exchange couplings in this region of the J_1-J_2 phase diagram. Several prototypes QFFMSL have been synthesized,[5] with ratios J_2/J_1 ranging from -1.8 for $Pb_2VO(PO_4)_2$ to -0.9 for $BaCdVO(PO_4)_2$,[6] very close to the boundary between the long-range magnetic order and the nematic order. On the other hand, it has been recently pointed out by Tsirlin and Rosner[7] that in these compounds the n.n. and n.n.n. exchange couplings along different sides and diagonals of the square lattice, respectively, might be inequivalent due to the buckling and stretching of the magnetic layers. The difference between the exchange couplings appears to be less pronounced for $BaCdVO(PO_4)_2$ than for $Pb_2VO(PO_4)_2$.

In order to study the evolution of the local microscopic properties of QFFMSL upon decreasing the ratio J_2/J_1 toward the critical value $J_2/J_1 \simeq -0.7$ and how they compare to the ones of frustrated $S=1/2$ square lattice systems with competing antiferromagnetic interactions[8,9] (QFAFSL, i.e., for $J_2/J_1 > 0$), we have performed zero field (ZF) and longitudinal field (LF) μSR experiments on $BaCdVO(PO_4)_2$ and $Pb_2VO(PO_4)_2$ powders. It was found that although both systems are characterized by a magnetically ordered ground state below T_N, significant differences with respect to the behavior observed for QFAFSL are present. The correlated spin dynamics in $Pb_2VO(PO_4)_2$ for $T > T_N$ and the temperature dependence of the magnetic order parameter are both consistent with a two-dimensional (2D) XY model. On the other hand, in $BaCdVO(PO_4)_2$ a rather peculiar behavior is observed, with a logarithmic increase in the longitudinal muon relaxation rate (λ) on cooling for $T > T_N$. This behavior is reminiscent of the one observed in one-dimensional systems and might suggest the onset of bond-nematic correlations above T_N.

II. TECHNICAL ASPECTS AND EXPERIMENTAL RESULTS

A polycrystalline sample of $Pb_2VO(PO_4)_2$ was synthesized by solid-state reaction technique using PbO (99.99%),

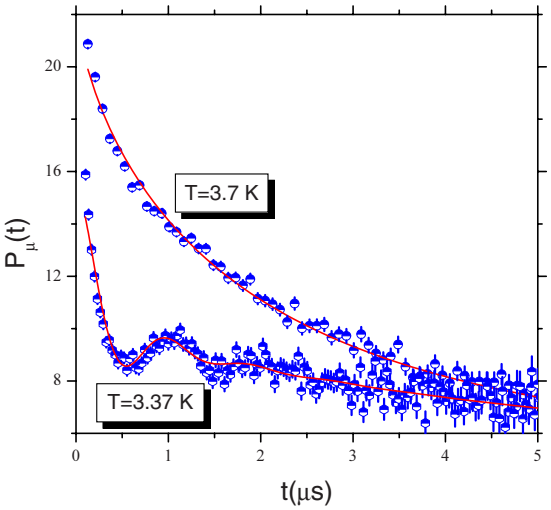

FIG. 1. (Color online) Time evolution of the ZFμSR asymmetry in $Pb_2VO(PO_4)_2$ at two representative temperatures, above and below T_N. The solid lines are best fits according to Eq. (2) (data at $T=3.7$ K) and Eq. (3) (data at $T=3.37$ K) in the text.

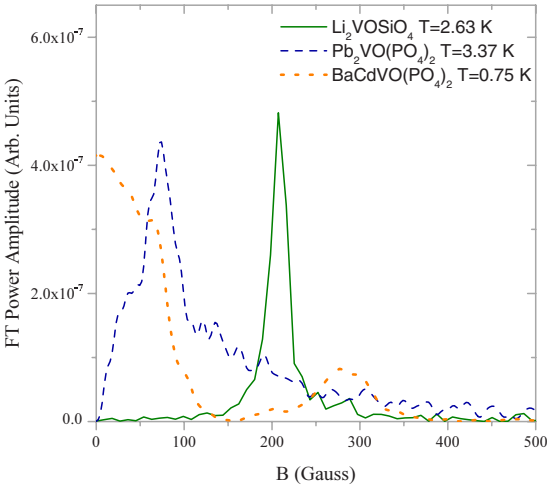

FIG. 2. (Color online) Power of the Fourier transform of the oscillating component of the ZFμSR asymmetry for $Pb_2VO(PO_4)_2$, $BaCdVO(PO_4)_2$ and Li_2VOSiO_4, for $T<T_N$. It is evident that while in Li_2VOSiO_4 (Ref. 12) a well-defined internal field is present, in $Pb_2VO(PO_4)_2$ and $BaCdVO(PO_4)_2$ a broad distribution of internal fields is probed by the muons.

$(NH_4)H_2PO_4$ (99.9%), and VO_2 (99.99%) as starting materials. In the first step, the intermediate compound $Pb_2P_2O_7$ was prepared firing the stoichiometric mixtures of PbO and $(NH_4)H_2PO_4$ in air at 750 °C for 24 h. In the second step, the intermediate product was mixed with VO_2 in appropriate molar ratio and heated for 48 h at 680 °C in dynamic vacuum with one intermediate grinding and pelletization. Synthesis of $BaCdVO(PO_4)_2$ was done following the same procedure reported in Ref. 6. Single phase materials were confirmed by x-ray diffraction performed with a STOE powder diffractometer (Cu K_α radiation). Both materials, $Pb_2VO(PO_4)_2$ and $BaCdVO(PO_4)_2$, contained a minor (2%–3%) fraction of unreacted diamagnetic phosphates $Pb_2P_2O_7$ and $BaCdP_2O_7$, respectively. These impurities are nonmagnetic and, therefore, are irrelevant for the discussion to be presented in the following.

ZF and LFμSR measurements were performed on EMU and MUSR beam lines at ISIS pulsed muon facility, using 29 MeV/c spin-polarized muons. The powders of $Pb_2VO(PO_4)_2$ and $BaCdVO(PO_4)_2$ were pressed and attached to a silver sample holder, whose background contribution (B) to the muon asymmetry was determined from the slowly decaying part of the longitudinal polarization. We checked that there were no history-dependent effects associated with a poor sample thermalization onto the dilution fridge cold plate.

In $Pb_2VO(PO_4)_2$ at temperatures above $T_N = 3.46 \pm 0.01$ K the decay of the muon polarization $P_\mu(t)$, either in zero (Fig. 1) or in a longitudinal field, could be fitted by the expression

$$P_\mu(t) = A \exp(-(\lambda t)^\beta) + B. \quad (2)$$

In ZF, for $T \gg T_N$, β was found to increase slightly above unity which indicates that at high temperature a non-negligible contribution to the relaxation arises from the field distribution generated by the dipolar interaction with the nuclei, which typically gives a Gaussian decay.[10] On the other hand, when a LF of 1 kGauss was applied in order to quench the nuclear dipole contribution, β was found to decrease from unity to about 0.7 on approaching T_N, possibly due to the presence of μ^+ inequivalent sites.

Below T_N ZF experiments showed oscillations in the muon asymmetry (Fig. 1) which clearly indicate the presence of a long-range magnetic order, which generates an internal field at the muon site B_μ. Accordingly one observes a precessional signal at a frequency $\omega_\mu = \gamma_\mu B_\mu$, with γ_μ the muon gyromagnetic ratio. Nevertheless, the initial decay of the muon polarization can hardly be fitted by assuming a well-defined field at the muon. On the other hand, one can fit $P_\mu(t)$ over a broad temperature range below T_N with the expression

$$P_\mu(t) = A_1 \exp(-\sigma t) J_0(\gamma_\mu B_\mu t) + A_2 \exp(-(\lambda t)^\beta) + B, \quad (3)$$

where $J_0(x)$ is the zeroth order Bessel function of the first kind, which characterizes the decay of the muon polarization in the presence of a distribution of internal fields,[11] which is further broadened by the decay constant σ. In fact, if one performs the Fourier transform of the oscillating component of $P_\mu(t)$ one finds quite a broad spectrum (Fig. 2). Upon decreasing the temperature below T_N one observes a loss of the initial asymmetry, possibly due to a further broadening of the field distribution, yielding a decrease in the ratio A_1/A_2 below 2. The temperature dependence of B_μ, derived by fitting the ZF decay of the muon asymmetry with Eq. (3) is reported in Fig. 3. In Fig. 4 the temperature dependence of the longitudinal relaxation rate λ is reported. A clear divergence of the relaxation rate is observed at T_N.

Also for $BaCdVO(PO_4)_2$ at temperatures above $T_N = 0.99 \pm 0.01$ K the decay of the muon polarization can be nicely fitted with Eq. (2), with an exponent decreasing from $\beta \simeq 1$ for $T \simeq 10$ K to $\beta \simeq 0.6$ on approaching T_N (Fig. 5).

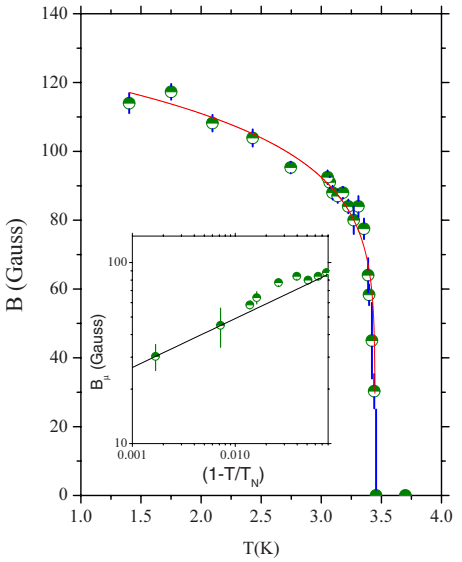

FIG. 3. (Color online) Temperature dependence of the internal field at the muon in $Pb_2VO(PO_4)_2$ as derived from ZFμSR experiments. In the inset the same data are reported as a function of the reduced temperature for $T \rightarrow T_N$. The solid line is the critical behavior expected for a 2D *XY* model.

On the other hand, although in ZF below T_N oscillations in $P_\mu(t)$ are observed, confirming the onset of a long-range magnetic order,[6] the decay of the muon polarization can hardly be fitted by Eq. (3) and a much broader distribution of internal fields is present. In fact, by looking at the Fourier transform of the oscillating component of the muon asymmetry (Fig. 2) one clearly notices that a significant distribution of internal fields is present, much broader than in QFAFSL.[12] Still, it is possible to fit the slowly decaying nonoscillating component of $P_\mu(t)$ with a stretched exponential in order to derive the temperature dependence of λ over all the explored temperature range. Again a peak is present at T_N (Fig. 6). However it is noticed that for $T > T_N$ the increase in λ on cooling is rather different from the one observed in $Pb_2VO(PO_4)_2$.

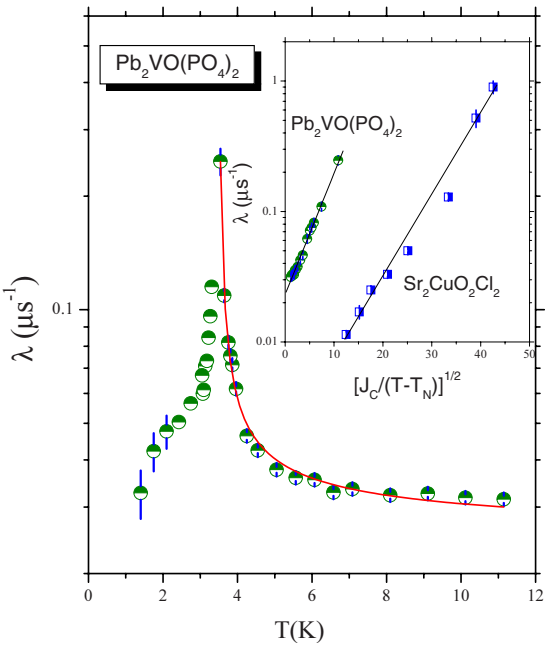

FIG. 4. (Color online) Temperature dependence of the muon longitudinal relaxation rate λ in $Pb_2VO(PO_4)_2$. Above T_N λ was derived in a field of 1000 Gauss, while below T_N it was estimated from the nonoscillating component of the muon asymmetry in zero field. The solid line shows the behavior expected for a 2D *XY* model. In the inset the same data are reported for $T \rightarrow T_N$ as a function of $\sqrt{J_c/(T-T_N)}$, together with the data derived for $Sr_2CuO_2Cl_2$, for a LF $H=800$ Gauss along the *c* axes. $J_c=10.4$ K for $Pb_2VO(PO_4)_2$ and $J_c=1450$ K for $Sr_2CuO_2Cl_2$.[17]

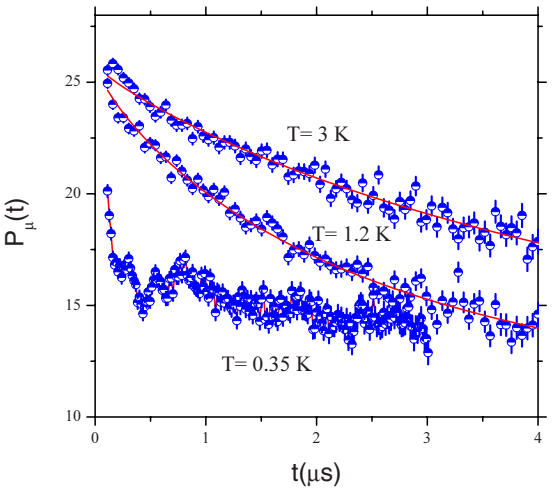

FIG. 5. (Color online) Time evolution of the ZFμSR asymmetry in $BaCdVO(PO_4)_2$ at three representative temperatures. For the data above T_N the solid lines are best fits according to Eq. (2), while for the data at $T=0.35$ K the solid line is just a guide to the eye.

III. DISCUSSION

First we shall address the temperature dependence of the muon longitudinal relaxation rate and the temperature depen-

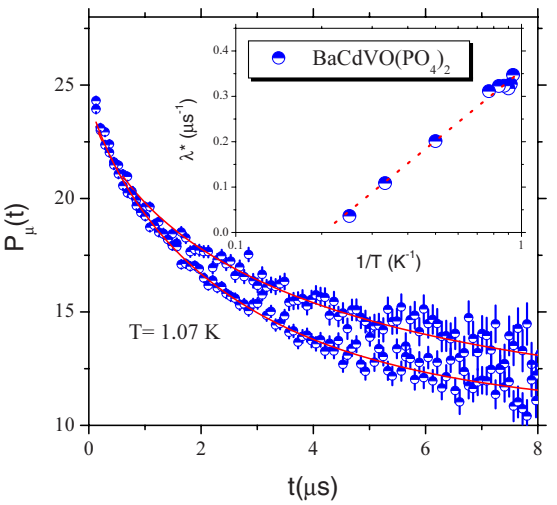

FIG. 6. (Color online) Time evolution of the muon asymmetry in $BaCdVO(PO_4)_2$ at $T=1.07$ K in zero field (bottom curve) and in a longitudinal field of 20 Gauss (top curve). In the inset λ^*, as derived from Eq. (6) is reported as a function of $1/T$.

dence of B_μ in $Pb_2VO(PO_4)_2$. The temperature dependence of B_μ corresponds to the one of the order parameter. In fact, in zero-field the local field at the muon can be written as $\vec{B}_\mu = \mathcal{A}\langle\vec{S}\rangle$,[10] where $\mathcal{A}$ is the hyperfine coupling tensor describing the coupling of the muon with the surrounding V^{4+} $S=1/2$ spins and $\langle\vec{S}\rangle$ the corresponding spontaneous spin polarization. In spite of the distribution of local fields evidenced in Fig. 2, which yields some uncertainty in the estimate of B_μ, the basic trend of $B_\mu(T)$ is reminiscent of the one observed for Li_2VOSiO_4 QFAFSL.[12] In this latter compound it has been observed that for $T \rightarrow T_N$ the order parameter increases according to the critical power law $B_\mu \propto (1 - T/T_N)^{\beta_c}$, with a critical exponent $\beta_c \simeq 0.235$, the one predicted for a 2D *XY* system.[13] If one reports B_μ data for $Pb_2VO(PO_4)_2$ in the proximity of T_N as a function of the reduced temperature $(1-T/T_N)$ (see the inset of Fig. 3), one realizes that also for $Pb_2VO(PO_4)_2$ $\beta_c \simeq 0.235$.

The temperature dependence of the muon longitudinal relaxation rate above T_N originates from the one of the in-plane correlation length. In fact, if we neglect the nuclear contribution to the relaxation which is temperature independent and in anyway, quenched by the application of a longitudinal field, the critical increase in λ on approaching T_N is of dynamical nature and associated with spin-lattice relaxation processes.[10] Then one can write

$$\lambda = \frac{\gamma_\mu^2}{2N}\sum_{\vec{q},\alpha} |A_{\vec{q}}|^2 S^{\alpha\alpha}(\vec{q},\omega_\mu), \tag{4}$$

where $S^{\alpha\alpha}(\vec{q},\omega_\mu)$ are the components of the dynamical structure factor at the muon Larmor frequency and $A_{\vec{q}}$ is the form factor describing the hyperfine coupling of the spin excitations at wave-vector $\vec{q}$ with the nuclei. By resorting to scaling arguments, which are expected to be valid for $T \rightarrow T_N$, one finds that for a 2D spin system $\lambda \propto \xi^z$, with ξ the in-plane correlation length and z the dynamical scaling exponent.[14] Considering that $Pb_2VO(PO_4)_2$ lyes in the sector of the J_1-J_2 phase diagram where the antiferromagnetic J_2 coupling is larger, one could at first take for ξ the temperature dependence expected for a $S=1/2$ 2D Heisenberg antiferromagnet on a square lattice. Namely, $\xi \propto \exp(2\pi\rho_s/T)$,[15] with ρ_s the spin-stiffness constant, which is possibly reduced with respect to the value $\rho_s = 1.15J/2\pi$ expected for a nonfrustrated systems. Accordingly one should observe that $\lambda \propto \exp(2z\pi\rho_s/T)$. However, we have found that this expression can fit the data only over a limited temperature range, no matter which value one takes for ρ_s. On the other hand, the temperature dependence of the order parameter below T_N suggests considering for ξ the form predicted for a 2D *XY* model,[16] namely $\xi \propto \exp[B_{XY}/(T-T_N)]^{1/2}$, where B_{XY} is of the order of magnitude of the exchange coupling constant. Then, one should find

$$\lambda = c \times e^{[z^2 B_{XY}/(T-T_N)]^{1/2}}. \tag{5}$$

Indeed, $Pb_2VO(PO_4)_2$ data can be nicely fitted with Eq. (5) over almost all the explored temperature range (Fig. 4). It is worth comparing the behavior observed for $Pb_2VO(PO_4)_2$ with the one found for a prototype of the $S=1/2$ 2D *XY* model, $Sr_2CuO_2Cl_2$.[17] In Fig. 4 we compare the temperature dependence of λ for $Pb_2VO(PO_4)_2$ and for $Sr_2CuO_2Cl_2$,[18] reported as a function of $\sqrt{J_c/(T-T_N)}$, with $J_c = \sqrt{J_1^2+J_2^2}$ an effective exchange coupling. One notices that for both systems Eq. (5) is followed very well.

Now we shall address the behavior of the local field at the muon below T_N in $BaCdVO(PO_4)_2$. A broad distribution of local fields at the muon site, even larger than the one found for $Pb_2VO(PO_4)_2$, is noticed (Fig. 4). This field distribution should not be associated with sample inhomogeneities since the phase transitions detected by λ or specific-heat measurements[6] appear rather sharp. On the other hand, it should be mentioned that in QFAFSL as Li_2VOSiO_4, where the buckling along the *a*-axis is not present and the unit cell is tetragonal, such a broad distribution is absent[12,19] (Fig. 4). Thus, at first sight one could think that the different structure could yield a distribution of muon sites, leading to a distribution of hyperfine coupling constants which could justify the broadening of the Fourier transform spectra. However, it is rather difficult to associate such a broad distribution with the presence of a discrete number of μ^+ sites and moreover the distribution is larger in $BaCdVO(PO_4)_2$ where the *ab* plane is less distorted.[7] Furthermore, even if *a priori* the presence of several inequivalent muon sites cannot be excluded, we mention that in oxides with superstructures and much larger unit cells, as $Bi_2Sr_2YCu_2O_{8-\delta}$ (Ref. 20)and $Sr_{14}Cu_{24}O_{41}$ (Ref. 21) for instance, just up to two muon sites are observed.

If the significant broadening in the Fourier transform is not due to a distribution of inequivalent probes it means that $\langle\vec{S}\rangle$ varies from lattice cell to lattice cell, namely either the underlying magnetic lattice is not commensurate with the crystal lattice structure or domains are present at the microscopic level. In this respect it is instructive to compare our results to neutron-scattering ones.[22] Although neutron-scattering experiments in $Pb_2VO(PO_4)_2$ indicate the presence of a columnar order, a broad background distribution of the energy integrated dynamical structure factor is noticed in *q*-space, even well below T_N.[22] The origin of this broad distribution could suggest the presence of small size domain like structures. Indeed $Pb_2VO(PO_4)_2$ and $BaCdVO(PO_4)_2$ are in a sector of the J_1-J_2 phase diagram where a twofold-degenerate magnetic ground state is present[23] and one could speculate that under certain conditions the coexistence of domains of both phases, characterized by different magnetic wave vectors, could be observed. Although, we cannot draw a clear conclusion on the origin of the broadening of the spectra, it is clear that in $BaCdVO(PO_4)_2$ and $Pb_2VO(PO_4)_2$ a larger degree of structural and possibly of magnetic disorder is present with respect to QFAFSL as Li_2VOSiO_4.

In $BaCdVO(PO_4)_2$ the application of a moderate magnetic field is observed to affect significantly the spectrum of the excitations and the specific heat.[6] Therefore, in order to probe the intrinsic low-energy excitations of this system we have performed zero-field relaxation experiments. Under these conditions an additional contribution to the decay of the muon polarization, associated with the dipolar interaction with the nuclei, might be present for $T > T_N$. Nevertheless, the stretched exponential form of $P_\mu(t)$ suggests that in $BaCdVO(PO_4)_2$ this contribution is not significant for T

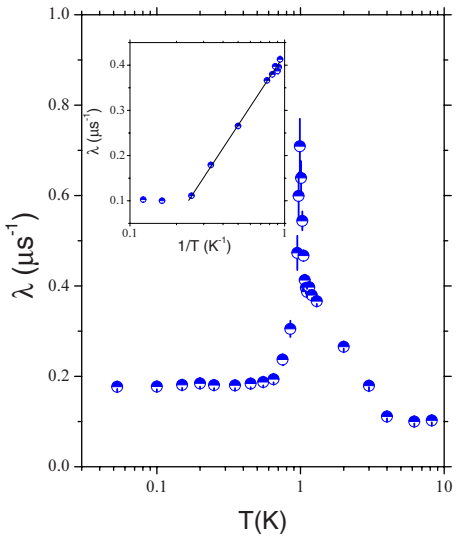

FIG. 7. (Color online) Temperature dependence of the zero-field muon longitudinal relaxation rate λ in $BaCdVO(PO_4)_2$. In the inset the same data are reported vs $1/T$ in a linear-log scale in order to evidence the logarithmic increase in λ above T_N.

≤ 4 K. In fact, if a longitudinal field of tens of Gauss is applied in order to quench the nuclear dipole contribution, only a certain decrease in λ is noticed (Fig. 6). Both this decrease and the high temperature ($T \simeq 10$ K) value of the zero-field relaxation rate (Fig. 7) are consistent with a nuclear contribution to the decay rate $\sigma \leq 0.1\ \mu s^{-1}$. In order to check if nuclear dipolar contribution to the relaxation does not significantly affect the temperature dependence of λ driven by the spin fluctuations, one can fit $P_\mu(t)$ zero-field data by including a static Kubo-Toyabe function[10] $KT(\sigma = 0.1\ \mu s^{-1})$ to account for the nuclear dipole contribution. Namely, the data can be fit with

$$P_\mu(t) = A\ \exp(-(\lambda^* t)^\beta) \times KT(\sigma) + B, \qquad (6)$$

where λ^* is the contribution of the spin excitations to the relaxation rate. The temperature dependence of λ^* above T_N, reported in the inset of Fig. 6, is very similar to the one of λ reported in Fig. 7. Therefore, the nuclear contribution to the relaxation rate does not affect significantly the temperature dependence of λ, to be discussed in the following paragraph.

The temperature dependence of the zero-field relaxation rate in $BaCdVO(PO_4)_2$(Fig. 7) shows a rather peculiar behavior, not observed in $Pb_2VO(PO_4)_2$. At high temperature, for $T > 4$ K, λ is practically temperature independent, as expected for a noncorrelated spin system. On decreasing the temperature toward T_N, one would expect for a 2D system, on the basis of Eq. (4), an exponential growth of λ. Remarkably λ is found to increase logarithmically, i.e., $\lambda \propto \ln(1/T)$ (Fig. 7), a trend which is rather characteristic of one-dimensional systems.[24] This behavior should not be related to the filtering of the critical fluctuations by the form factor in Eq. (4).[14] In fact, if this was the case one should expect the partial cancellation of the magnetic field at the muon site below T_N, at variance with the experimental findings (Fig. 2). On the other hand, since $BaCdVO(PO_4)_2$ should lie very close to the boundary with the bond-nematic phase,[4,6] one could expect that nematic correlations of stripy character arise above T_N and yield a logarithmic increase in λ with decreasing temperature. This occurs until the XY anisotropy and/or the interlayer coupling cause the crossover to a three-dimensional long-range order at a finite temperature resulting in an abrupt increase in λ very close to T_N (see Fig. 7). It is interesting to notice that for $T \ll T_N$, in contrast to what is observed for $Pb_2VO(PO_4)_2$ (Fig. 4), λ is constant at low temperature. This indicates a low temperature persistent dynamic, which is characteristic of frustrated systems with a degenerate ground state.[25]

IV. CONCLUSIONS

In this manuscript we have presented μSR measurements in two prototypes of QFFMSL: $Pb_2VO(PO_4)_2$ and $BaCdVO(PO_4)_2$. In both compounds a broad distribution of local fields at the muon site is evidenced and is tentatively associated either with an incommensurate magnetic order or with the formation of mesoscopic domains. In $Pb_2VO(PO_4)_2$ the overall temperature dependence of the longitudinal relaxation rate and of the order parameter below T_N are both consistent with a 2D XY model. On the other hand, in $BaCdVO(PO_4)_2$ a rather peculiar behavior of the longitudinal relaxation rate was evidenced, which can be hardly associated with conventional 2D spin correlations. The logarithmic increase in λ on cooling suggests the onset of one-dimensional correlations which might be related to novel type of correlations which arise on approaching the magnetically disordered phase of these QFFMSL.

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