Variation of antiferromagnetic Pr ordering in the Tl(Ba$_{1-x}$Sr$_x$)$_2$PrCu$_2$O$_{7-\delta}$ system

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Structural, magnetic, calorimetric, and transport studies have been made on a synthesized Tl(1212) system of Tl(Ba$_{1-x}$Sr$_x$)$_2$PrCu$_2$O$_{7-\delta}$ ($0 \le x \le 1$). Based on x-ray Rietveld refinement data, the bond length between Pr and oxygen in the adjacent CuO$_2$ layers increases with increasing x, even though the tetragonal lattice parameters decrease with progressive Sr substitution. This may result in a lower degree of hybridization between extended Pr $4f$ and O $2p\pi$ orbitals and a corresponding reduction of superexchange magnetic coupling strength among Pr moments. Indeed, the anomalously high Néel temperature T_N(Pr) decreases from 8.0–8.2 K for $x = 0$ to 3.6–4.2 K for $x = 1$. The magnetic entropy associated with this antiferromagnetic Pr ordering is estimated from specific-heat data, in which a heavy-fermion-like contribution also prevails.

I. INTRODUCTION

PrBa$_2$Cu$_3$O$_7$ is the only nonsuperconducting member of the orthorhombic RBa$_2$Cu$_3$O$_7$ system with $R =$ Y and most rare-earth elements.[1–4] The dramatic suppression of superconducting transition temperature from above 90 K and the anomalously high antiferromagnetic Pr ordering temperature T_N(Pr) of 17 K remain to be among the most intriguing topics in high-T_c research.[1–6] The strong hybridization between the Pr $4f$ and the eight O $2p\pi$ orbitals in the adjacent CuO$_2$ layers is believed responsible for both the suppression of superconductivity and the anomalous Pr ordering. The proposed T_c suppression mechanisms of either hole filling by Pr^{4+} ions or hole localization by Pr^{3+} ions is presumably associated with the hybridization.[4–7] For the entire oxygen-deficient orthorhombic-tetragonal semiconducting system PrBa$_2$Cu$_3$O$_{7-y}$ ($0.0 \le y \le 1.0$), Cu^{2+} moments become ordered antiferromagnetically above room temperature.[8] Meanwhile, T_N(Pr) decreases from 17 K for PrBa$_2$Cu$_3$O$_7$ ($y \cong 0.0$) to 10 K for PrBa$_2$Cu$_3$O$_6$ ($y \cong 1.0$).[9] In comparison, the next highest $T_N(R)$ for the RBa$_2$Cu$_3$O$_{7-y}$ system is 2.2 K for GdBa$_2$Cu$_3$O$_{7-y}$.[10,11] Since Pr is one of the lighter elements of rare-earth series, its $4f$ wave functions should be more extended. The anomalously high T_N(Pr) indicates the importance of quasi-two-dimensional Pr-O-Pr superexchange mechanism through the strong hybridization between the Pr $4f$ and the eight O $2p\pi$ orbitals in the adjacent CuO$_2$ layers.[12] The degree of hybridization is expected to follow the bond length d[Pr-O]. Crystallographic data indicate that this bond length increases with decreasing T_N(Pr) value, from approximately 2.451 Å for orthorhombic PrBa$_2$Cu$_3$O$_7$ with T_N(Pr)$= 17$ K to 2.472 Å for tetragonal PrBa$_2$Cu$_3$O$_6$ with T_N(Pr)$= 10$ K.[9,13]

A similar Pr anomaly occurs in other two-CuO$_2$-layered systems.[14,15] A recent report on the new Tl(1212) compound of TlBa$_2$PrCu$_2$O$_{7-\delta}$ has revealed a broad Pr ordering near 8–10 K from magnetic-susceptibility and specific-heat measurements.[14] This is corroborated by the magnetic ($\frac{1}{2},\frac{1}{2},\frac{1}{2}$) Bragg line in neutron diffraction, which also yields magnetic lattice parameters of $a_m = 2a$ and $c_m = 2c$. The extrapolated Pr-sublattice saturation moment along the c axis is $1.1\mu_B$/Pr. It is smaller than the calculated Pr^{3+} ($4f^2,{}^3H_4$) value of $gJ = 3.2\mu_B$. The discrepancy may be originated from the complex crystal-field effect on the more extended Pr $4f$ wave functions, which are not completely shielded by the $5s^2 5p^6$ outer shells, in addition to a strong quasi-two-dimensional quantum spin fluctuation.[16] The tetragonal TlBa$_2$CaCu$_2$O$_{7-\delta}$-type structure (space group $P4/mmm$) of TlBa$_2$PrCu$_2$O$_{7-\delta}$ is very similar to the orthorhombic-tetragonal lattice (space group P/mmm or $P4/mmm$) of the PrBa$_2$Cu$_3$O$_{7-y}$ system. In fact, by rewriting the formula of TlBa$_2$PrCu$_2$O$_7$ to PrBa$_2$(TlCu$_2$)O$_7$, its only difference from PrBa$_2$Cu$_3$O$_7$ is the replacement of the Cu-O chains by the Tl-O planes.[14,17] Cu^{2+} moments also become ordered antiferromagnetically around T_N(Cu)$\cong 370$ K.[16] Again, in comparison, the next two highest $T_N(R)$ values for the TlBa$_2$$RCu_2O_{7-\delta}$ system are 2.2 K for TlBa$_2$GdCu$_2$O$_{7-\delta}$, 1.5 K for TlBa$_2$NdCu$_2$O$_{7-\delta}$ and 0.94 K for TlBa$_2$DyCu$_2$O$_{7-\delta}$.[18–20] Preliminary Cu NMR study yields additional support to the strong hybridization between CuO$_2$ layers and Pr orbitals.[20] The anomalous unit-cell volume variation as compared with other R^{3+} rare-earth compounds TlBa$_2$$RCu_2O_{7-\delta}$ also supports this argument.[21] To further explore this mechanism, the Tl-1212 study has been extended to the newly found TlSr$_2$PrCu$_2$O$_{7-\delta}$ compound and the Tl(Ba$_{1-x}$Sr$_x$)$_2$PrCu$_2$O$_{7-\delta}$ solid solution system. The smaller Sr^{2+} radius (1.12 Å) as compared to that of Ba^{2+} (1.34 Å) should lead to a modification of such hybridization.

II. EXPERIMENTS

Seven $Tl(Ba_{1-x}Sr_x)_2PrCu_2O_{7-\delta}$ samples ($x = 0$, 0.2, 0.4, 0.5, 0.6, 0.8, and 1) were prepared by solid-state-reaction techniques. Synthesis of the $x = 0$ sample followed the same procedures as given in Ref. 14. For each Sr-substituted sample, high-purity $SrCO_3$, $BaCO_3$, Pr_6O_{11}, and CuO powders in a stoichiometric ratio were thoroughly mixed, ground, and calcined at 900 °C for 12 h. After an appropriate amount of Tl_2O_3 was added, the powders were pressed into pellets, wrapped in gold foils, and individually placed in a gold-foil-covered alumina crucible. The pellets were reacted and fully oxygenated by annealing in flowing oxygen at 880–980 °C for 5–10 h, followed by furnace cool ($x < 1.0$) or liquid nitrogen quench ($x = 1$).

Structural analysis was obtained from a Rigaku Rotaflex rotating anode powder x-ray diffractometer using Cu $K\alpha$ radiation. Rietveld refinement method provided detailed crystallographic data.

Magnetic-susceptibility data were obtained using a Quantum Design MPMS superconducting-quantum-interference-device magnetometer down to 2 K in an applied magnetic field of 1 T. Specific-heat data between 2 and 15 K were generated using an adiabatic calorimeter with pulsed joule heating, the heat capacity of which was separately determined for addenda correction. ac electrical-resistivity data were obtained using the standard four-probe method with a 1 mA current of 16 Hz down to 10 K in a closed-cycle refrigerator.

III. RESULTS AND DISCUSSION

$TlSr_2PrCu_2O_{7-\delta}$ is successfully synthesized for the first time. Powder x-ray-diffraction patterns in Fig. 1 indicate that this tetragonal compound with lattice parameters $a = 3.854(2)$ Å, $c = 12.108(4)$ Å, and unit-cell volume V of 179.8(1) Å^3 is isostructural to $TlBa_2PrCu_2O_{7-\delta}$ [$a = 3.918(2)$ Å, $c = 12.550(4)$ Å, and $V = 192.7(1)$ Å^3]. An homogeneous single-phase solid solution appears in the substitutional compounds $Tl(Ba_{1-x}Sr_x)_2PrCu_2O_{7-\delta}$ with $x = 0.2$, 0.4, 0.6, and 0.8. The compositional variations of a, c, and V are shown in Fig. 2. The lattice pa-
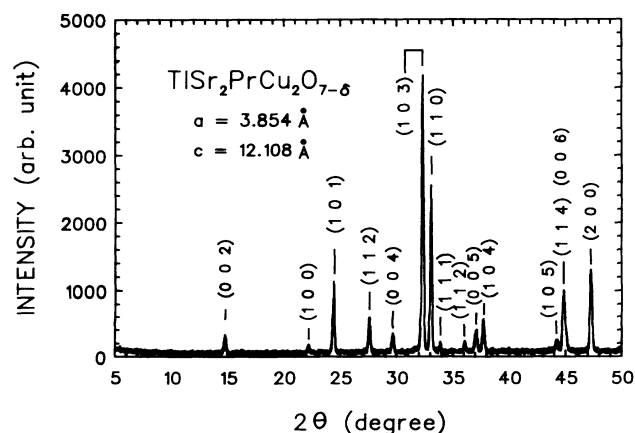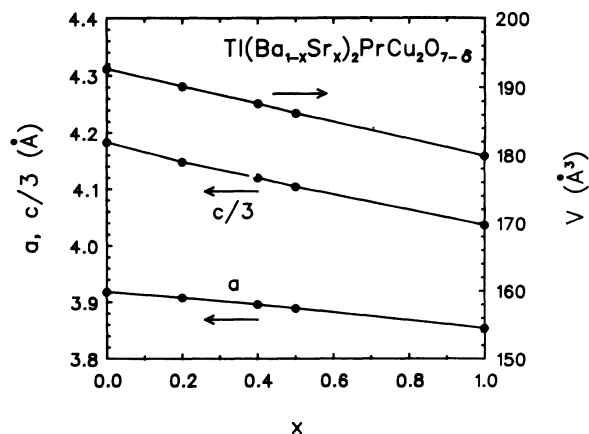

FIG. 2. Compositional variation of tetragonal lattice parameters a, c, and unit-cell volume V for the $Tl(Ba_{1-x}Sr_x)_2PrCu_2O_{7-\delta}$ system.

rameters decrease steadily with increasing x. This is expected, considering the relative size of Ba^{2+} and Sr^{2+} ions. However, volume reduction of 12.88 Å^3 due to full Sr substitution is much larger than the estimated 8.38 Å^3 from simple ionic volume calculation. Further in-depth analysis on four samples ($x = 0$, 0.5, 0.6, 1) from detailed x-ray Rietveld refinement[22,23] becomes relevant. Typical crystallographic parameter refinement data for the parent compound $TlBa_2PrCu_2O_{7-\delta}$ ($\delta = 0.07$) with refinement factors $R_P = 5.09\%$ and $R_{WP} = 6.89\%$ are listed in Table I. The small Tl deficiency (6%) arises from the high Tl volatility during the sample preparation process. The Pr-O(3) bond lengths

$$d[\text{Pr-O}(3)] = \{(a/2)^2 + [(0.5-z)c]^2\}^{0.5} ,$$

where O(3) refers to oxygen ions in the CuO_2 layers, and z is a crystallographic parameter as listed in Table I. Of particular importance is the fact that, in spite of the steady decrease of lattice parameters a and c, the bond length $d[\text{Pr-O}(3)]$ increases slightly from 2.478(5) Å for $x = 0$ to 2.514(2) Å for $x = 1$ due to a decrease in z value (from 0.379 to 0.368).
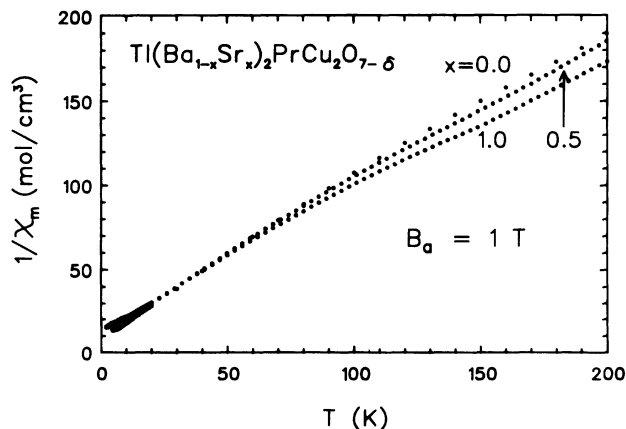
Figure 3 presents the temperature dependence of in-

FIG. 1. Powder x-ray-diffraction patterns for new tetragonal compound $TlSr_2PrCu_2O_{7-\delta}$.

FIG. 3. Temperature dependence of inverse molar magnetic susceptibility χ_m^{-1} for three $Tl(Ba_{1-x}Sr_x)_2PrCu_2O_{7-\delta}$ samples ($x = 0.0$, 0.5, and 1.0).

TABLE I. Crystallographic parameter refinement data for $TlBa_2PrCu_2O_{7-\delta}$ ($\delta = 0.07$). $R_P = 5.09\%$, $R_{WP} = 6.89\%$.

Atom	Site	x	y	z	B	Occupancy
Tl	$1a$	0	0	0	1.64(1)	0.94(3)
Ba	$2h$	$\frac{1}{2}$	$\frac{1}{2}$	0.2078(2)	0.13(1)	1.0
Pr	$1d$	$\frac{1}{2}$	$\frac{1}{2}$	$\frac{1}{2}$	0.13(1)	1.0
Cu	$2g$	0	0	0.3622(2)	0.13(1)	1.0
O(1)	$1c$	$\frac{1}{2}$	$\frac{1}{2}$	0	0.65(6)	0.93(5)
O(2)	$2g$	0	0	0.167(2)	0.33(3)	1.0
O(3)	$4i$	0	$\frac{1}{2}$	0.379(3)	0.27(3)	1.0

verse molar magnetic susceptibility χ_m^{-1} in an applied magnetic field of 1 T for three typical samples with $x = 0$, 0.5, and 1. Their Cu^{2+} moments are already ordered antiferromagnetically above room temperature.[16,24] Because of the extended $4f$ wave functions of light rare-earth Pr, the crystal-field effect is comparable to the exchange interaction. Therefore, no simple Curie-Weiss behavior is observed even in the relatively high applied field. However, the data can be roughly fitted by the relation $\chi(T) = \chi_0 + C/(T + \theta_P)$, with a negative paramagnetic intercept of $|\theta_P| \cong 7$ K for $x = 0$, 7.5 K for $x = 0.5$, and 10 K for $x = 1$. The effect moment μ_{eff} derived from the fitted Curie constant is $2.70\mu_B$ for $x = 0$, $2.77\mu_B$ for $x = 0.5$, and $2.73\mu_B$ for $x = 1$. For reference, the calculated crystal-field configuration in the orthorhomic $PrBa_2Cu_3O_7$ compound gives a quasitriplet ground state from the inelastic-neutron-scattering data.[7,25,26] Also to be noted is that a preliminary neutron-diffraction study on $Tl(Ba_{1-x}Sr_x)_2PrCu_2O_{7-\delta}$ with $x = 0.5$ indicates a complicated Cu spin reoriented structure below room temperature.[24]

Low-temperature derivatives of molar magnetic susceptibility $d\chi_m/dT$ for the same three samples ($x = 0$, 0.5, 1) are presented in Fig. 4. The minimum in $d\chi_m/dT$ corresponds to the inflection point in $\chi_m(T)$, which is generally taken as the antiferromagnetic Pr ordering temperature $T_N(Pr)$. The broad transitions vary monotonically from approximately 8.0 K for $x = 0$, 5.0 K for $x = 0.5$, to 3.6 K for $x = 1$. Compositional variations of $T_N(Pr)$ and bond length $d[Pr-O(3)]$ are summarized in Fig. 5 and listed in Table II. The correlation between x dependence of $T_N(Pr)$ and $d[Pr-O(3)]$ is evident, and is

consistent with a similar observation for the $PrBa_2Cu_3O_{7-y}$ system. For the latter, the Pr-O bond length increases from 2.451 Å for orthorhombic $PrBa_2Cu_3O_7$ with $T_N(Pr) = 17$ K to 2.479 Å for tetragonal $PrBa_2Cu_3O_6$ with $T_N(Pr) = 10$ K.[9,13]

Figure 6 shows the temperature dependence of molar specific heat C for $x = 0$, 0.5, and 1. Also included are data for a nonmagnetic reference compound $TlBa_2YCu_2O_{7-\delta}$.[14] The latter has only a lattice contribution $C_l = \beta T^3$ equivalent to a Debye temperature $\Theta_D = 250$ K. The specific-heat anomaly in each sample is certainly a corroboration to the magnetic data as an indication of the antiferromagnetic Pr ordering around 8.2 K for $x = 1$, 5.4 K for $x = 0.5$, and 4.2 K for $x = 1$, even though the broad peak makes it difficult to unambiguously identify $T_N(Pr)$. It is of interest to point out that, for $x = 0$ and 0.5, $T_N(Pr)$ values of 8 K and 6 K, respectively, have been confirmed by neutron diffraction.[16,23] The enhanced transition widths may result from the quasi-two-dimensional short-range magnetic correlation above $T_N(Pr)$ and the strong hybridization of Pr $4f$ and adjacent oxygen states.[25,26]

Above 10 K, the specific-heat data for all samples can be fitted to the sum of a T^3-dependent lattice contribution $C_l = \beta T^3$ and a linear term γT. The Debye temperature Θ_D as derived from β varies from 280 K for $x = 0$, 290 K for $x = 0.5$, to 350 K for $x = 1$. Figure 7 shows the temperature dependence of $\Delta C/T = (C - C_l)/T$. The γ values thus derived above T_N are included in Table II. These exceptionally large, heavy-Fermion-like linear terms seem to prevail in many other Pr-containing high-T_c systems.[6,27,28] Their origin, however, remains to be

TABLE II. Pr Néel temperature $T_N(Pr)$, Debye temperature Θ_D, linear term coefficient of molar specific heat γ, effective magnetic moment μ_{eff} and Pr-O(3) bond length $d[Pr-O(3)]$ for the $Tl(Ba_{1-x}Sr_x)_2PrCu_2O_{7-\delta}$ system.

x	T_N (K)	Θ_D (D)	γ J/mol K)	$\rho(300$ K) (Ω cm)	$d[Pr-O(3)]$ (Å)
0.0	8.0,8.2[a]	280	0.57	9600	2.478(2)
0.2	6.5				
0.4	5.7				2.498(2)
0.5	5.0,5.4[a]	290	0.34	157	2.506(2)
0.6	5.0				
0.8	4.5				
1.0	3.6,4.2[a]	350	0.42	0.189	2.514(2)

[a]From specific-heat data.

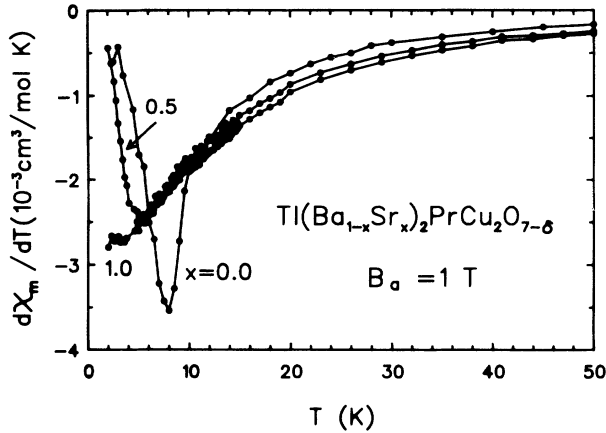FIG. 4. Low-temperature derivative molar magnetic susceptibility $d\chi_m/dT$ for $Tl(Ba_{1-x}Sr_x)_2PrCu_2O_{7-\delta}$ ($x=0.0$, 0.5, and 1.0). Antiferromagnetic Néel temperature $T_N(Pr)$ is defined as the minimum in $d\chi_m/dT$.

identified. The area integral below each set of data equals the entropy change $\Delta S = \int [(C - \beta T^3)/T]dT$ with the lattice contribution excluded. A question remains as to what fraction of ΔC is associated with the ordering process. Basically, the yet-to-be-understood γT term extends to above T_N, and its presence at lower temperatures should be excluded from the integration toward estimating the magnetic ordering-related entropy ΔS_m. Nevertheless, the γ value may not remain constant below T_N, because of possible change in magnetic correlations. Without such information at present, the horizontal dashed line simply represents the constant γT extrapolation from higher temperatures. Accordingly, the ΔS_m values between 2 and 10 K are derived from $\int (C_m/T)dT$ with $C_m = C - \beta T^3 - \gamma T$. The estimated magnetic entropy values are only fractions of $R \ln 2$ or $R \ln 3$ corresponding to a doublet ground state of Pr^{4+} or a quasitriplet ground state of Pr^{3+}.[7] The extra contribution below 2 K as well as the uncertainty in $\gamma(T < T_N)$ could enhance the ΔS_m estimates.

Electrical-resistivity data show a temperature dependence characteristic of a semiconducting variable-range
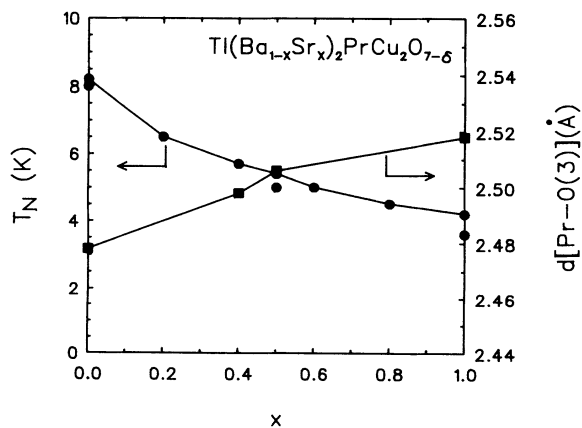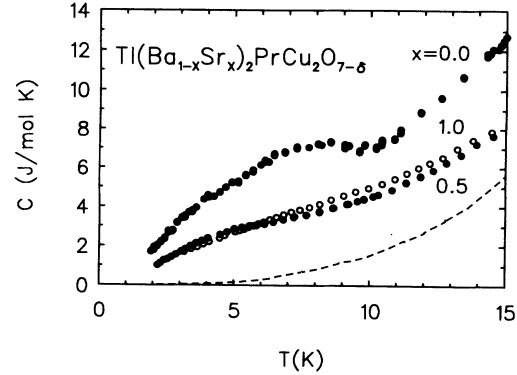

FIG. 6. Temperature dependence of molar specific heat C for $Tl(Ba_{1-x}Sr_x)_2PrCu_2O_{7-\delta}$ ($x=0.0$, 0.5, and 1.0). Nonmagnetic $TlBa_2YCu_2O_{7-\delta}$ data are shown as dashed line.

hopping behavior for all samples. There is, however, very dramatic variation of room-temperature resistivity, which decreases sharply from 9600 Ω cm for $x=0$ to 157 Ω cm for $x=0.5$ and 0.189 Ω cm for $x=1$.

It seems that these anomalous magnetic and electrical properties can be understood through the strong hybridization between the Pr $4f$ orbital and the adjacent eight O $2p\pi$ orbitals in the CuO_2 layers. As mentioned earlier, this strong hybridization is originated from the extended $4f$ wave function of the light rare earth Pr. The degree of hybridization can be reflected in the bond length $d[Pr-O(3)]$. Progressive replacement of larger Ba^{2+} ions by smaller Sr^{2+} ions decreases the bucking angle between Cu and O(3) ions in the CuO_2 layers, which in turn may lengthen the net bond length between Pr and O(3). As a consequence, the superexchange coupling strength through Pr-O-Pr and, therefore, the Pr ordering temperature $T_N(Pr)$ decrease with increasing Sr substitution. Meanwhile, an effective coupling occurs between Cu and Pr moments through oxygen $2p\sigma/2p\pi$ as revealed by preliminary neutron and NMR studies.[20,24] This competition should be taken into account before a final conclusion can be reached.
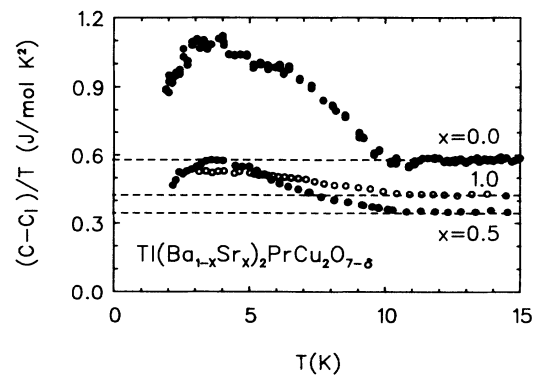

FIG. 5. Compositional variation of $T_N(Pr)$ and Pr-O(3) bond length $d[Pr-O(3)]$ for the $Tl(Ba_{1-x}Sr_x)_2PrCu_2O_{7-\delta}$ system.

FIG. 7. $(C-C_1)/T$ versus T for $Tl(Ba_{1-x}Sr_x)_2PrCu_2O_{7-\delta}$ ($x=0.0$, 0.5, and 1.0). Area integral below the data points provides an estimate of entropy change (lattice contribution excluded).

IV. CONCLUSION

The structural, magnetic, calorimetric, and transport studies have been made on the newly synthesized Tl(1212) system of $Tl(Ba_{1-x}Sr_x)_2PrCu_2O_{7-\delta}$ ($0 \leq x \leq 1$). X-ray Rietveld refinement data indicate an increasing Pr-O(3) bond length between Pr and eight oxygen ions in the adjacent CuO_2 layers, even though the tetragonal lattice parameters decrease with progressive Sr substitution. Consequently, the degree of hybridization between the extended $4f$ orbital of light rare-earth Pr and O $2p\pi$ orbitals is reduced. This may result in a corresponding reduction of superexchange magnetic coupling strength among Pr moments. Indeed, an anomalously high antiferromagnetic Pr ordering temperature $T_N(Pr)$ decreases from 8 K at $x = 0$ to 4 K at $x = 1$. The magnetic entropy associated with this transition is estimated from specific-heat data, in which a heavy-Fermion-like contribution also prevails. More diverse and detailed studies are in progress.

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