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# Anisotropic magnetocaloric properties of the ludwigite single crystal $Cu_2MnBO_5$

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ABSTRACT

We report upon the specific heat and magnetocaloric properties of $Cu_2MnBO_5$ over a temperature range of 60–350 K and in magnetic fields up to 18 kOe. It is found that at temperatures below the Curie temperature ($T_C \sim 92$ K), $C_P(T)/T$ possesses a linear temperature-dependent behavior, which is associated with the predominance of two-dimensional antiferromagnetic interactions of magnons. The temperature independence of $C_P/T = f(T)$ is observed in the temperature range of 95–160 K, which can be attributed to the excitation of the Wigner glass phase. The magnetocaloric effect [i.e., the adiabatic temperature change, $\Delta T_{ad}(T,H)$] is assessed through a direct measurement or an indirect method using the $C_P(T,H)$ data. Owing to its strong magnetocrystalline anisotropy, an anisotropic magnetocaloric effect (MCE) or the rotating MCE [$\Delta T_{ad}^{rot}(T)$] is observed in $Cu_2MnBO_5$. A deep minimum in the $\Delta T_{ad}^{rot}(T)$ near the $T_C$ is observed and ascribed to the anisotropy of the paramagnetic susceptibility.

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Ludwigite $Cu_2MnBO_5$ belongs to the group of quasi-low-dimensional transition metal oxyborates, which is a prominent representative of systems with strongly correlated properties.[1–3] Macroscopic magnetic and specific heat studies have shown that this compound undergoes a magnetic phase transition into the ferrimagnetic phase at $T_C \sim 92$ K.[1–3] The crystal-orientational dependences of magnetization and magnetic susceptibility revealed the anisotropic magnetic characteristic both below the $T_C$ and in the paramagnetic regime (associated with the anisotropy of g-tensor due to monoclinic distortions introduced by Jahn–Teller $Cu^{2+}$ and $Mn^{3+}$ ions[3]). The microscopic magnetic structure of the ludwigite was experimentally studied using powder neutron diffraction.[2] It was determined that out of four nonequivalent positions occupied by magnetic ions, three positions are predominantly occupied by $Cu^{2+}$ ions, and one by $Mn^{3+}$ ions. It was also found that in the compound under study there was only a partial ordering of the magnetic moments in the ferrimagnetic phase due to the small moment of $Cu^{2+}$ ions in position $2a$. The magnetic moments of $Cu^{2+}$ and $Mn^{3+}$ ions in the ferrimagnetic phase are antiparallel and

their directions do not coincide with the main crystallographic directions in the crystal.[2] The structural, magnetic, and thermodynamic properties of $Cu_2MnBO_5$ were studied.[1–3] However, the magnetothermal response of the material has not been investigated to date.

The utilization of the magnetocaloric effect (MCE) in magnetic cooling technology is of current interest as it has the potential to replace conventional gas compression techniques.[4,5] In addition to its perspective cooling application, the MCE has recently been used as a useful research tool for the analysis and interpretation of competing magnetic phases and the collective magnetic phenomena in a wide range of exotic magnetic materials.[6–10] In this regard, we present here results of the first comprehensive study of the anisotropic magnetocaloric properties of the ludwigite crystal $Cu_2MnBO_5$.

Single crystals of $Cu_2MnBO_5$ were synthesized by the flux method with the ratio of the initial components $Bi_2Mo_3O_{12}$:1.3$B_2O_3$:0.7$Na_2CO_3$:0.7$Mn_2O_3$:2.1CuO by spontaneous nucleation.[1] The oxyborate $Cu_2MnBO_5$ has a monoclinic distorted structure and is characterized by the P21/c space group due to the presence of $Cu^{2+}$

and $Mn^{3+}$ cations in Jahn–Teller cations.[1,2] The $Cu_2MnBO_5$ sample has a plate shape, with a dimension of $3 \times 3 \times 0.3\,mm^3$ in the $a^*b^*c$ configuration. Since the sample has a plate shape and the magnetic field was applied parallel to the plane of the plate, the effect of the demagnetizing factor on the MCE is less significant and negligible. The specific heat was measured by ac-calorimetry. Direct measurements of the adiabatic temperature change $\Delta T_{ad}$ were carried out by the modulation method[11] in the direction of the magnetic field in the $ab$-plane. The application of an alternating magnetic field to the sample induced a periodic change in the temperature of the sample, due to the MCE. This temperature change was recorded by a differential thermocouple. The frequency of the alternating magnetic field in this experiment was 0.2 Hz. An alternating magnetic field with an amplitude of up to 4 kOe was generated using an electromagnet and an external control power unit. The control alternating voltage was supplied to the power unit from the output of the (lock-in) amplifier SR 830. An alternating magnetic field of 18 kOe was created by a source of permanent magnetic field of an adjustable intensity manufactured by AMT & CLLC.

Figure 1 shows the temperature dependence of the specific heat $C_p(T)$ for $Cu_2MnBO_5$. The inset of Fig. 1 demonstrates the temperature dependence of $C_P/T$ in the range of 70–150 K and at $H = 0$, 6.2, and 11 kOe. The $C_P(T)$ dependence of $Cu_2MnBO_5$ was reported in Ref. 2, and our results are in good agreement with these data. Note that $C_P(T)$ dependence exhibits a pronounced lambda anomaly at $T_C = 92$ K, associated with the ferromagnetic-paramagnetic (FM-PM) phase transition, which is suppressed upon the application of a magnetic field, while the maximum of the heat capacity shifts by 4 K toward a higher temperature at $H = 11$ kOe. Two features of the $C_P(T,H)$ behavior are worthy of note. Below the $T_C$, the $C_P/T(T)$ is linear and unchanged with temperature in the temperature range of 95–160 K. A similar behavior was observed for a number of ferroborates.[12,13] At $T < T_C$, the $C_P/T = f(T)$ is well described by the expression $C_P = \alpha T^2$ (the dashed line in Fig. 1). This behavior has been explained by the predominance of two-dimensional antiferromagnetic interactions of magnons.[12]
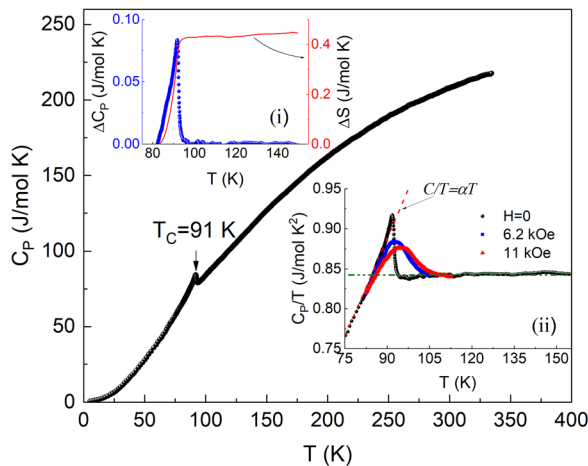


**FIG. 1.** Temperature dependence of $C_P/T$ for $Cu_2MnBO_5$ at $H = 0$, 6.2, and 11 kOe. Inset (i) shows the temperature dependences of $\Delta C_P$ (blue) and the entropy change $\Delta S$ (red line). Inset (ii) shows the temperature dependence of an enlarged anomalous part of the $C_P/T$ (points).

Using expressions, $C_P = \alpha T^2$ and $\alpha = 7.2k_B^3/2\pi D(N/A)$, where $\alpha$ is determined from the experimental data (Fig. 1) to be $\sim 0.01118$ J/ mol $K^3$ and $(N/A) = 3 \cdot 10^{-9}$ mole/$cm^2$—the number of magnetic ions per unit area,[12] we can estimate the spin wave stiffness $D = 7.2k_B^3/2\pi\alpha(N/A) = 8.9810^{-59}$ $J^2cm^2$. Then using $\sqrt{D} = zJSa$, we have obtained the numerical value of the exchange integral from the $C_p(T)$ data at $T < T_C$ as $J = \sqrt{D}/zSa = 5.2610^{-23}$ J or $J/k_B = 3.8$ K. Here, we suppose $S = 2$ for $Mn^{3+}$ ions, $z = 3$ and $a = 3$ Å as the medium distance of local ions. We have neglected little variations in the distance between the local ions and the magnetic arrangements of the walls. This value of $J/k_B = 4$ K, which is compared to that reported for $Fe_3BO_5$ ($J/k_B = 2.1$ K), which orders near 70 K.[12] We can also obtain the magnons speed in the walls of $Cu_2MnBO_5$, $v = 8.9910^2$ m/s, by considering a linear dispersion relation with k $\hbar w = ck$. Just to compare, 2D antiferromagnetic magnons were also reported in hulsite with $v = 1.1810^3$ m/s.[14] This reinforces the presence of 2D antiferromagnetic magnons in our compound.

The temperature-independent $C_P/T = f(T)$ behavior is observed in the temperature range of 95–160 K (Fig. 1), which is associated with a precursor effect of the charge ordering in a Wigner glass phase.[12] It may also arise from the presence of short range magnetic ordering in a low dimensional structure persisting just above the magnetic phase transition.[13] It is interesting to point out that the coefficients of the linear terms, $C/T = 773$ mJ/mol $K^2$ obtained for $Fe_3BO_5$[12] and $C/T = 843$ mJ/mol $K^2$ for the present $Cu_2MnBO_5$, are of similar magnitude.

The inset of Fig. 1 also shows the temperature dependence of the magnetic contribution to the specific heat, $\Delta C_p(T) = C_P - C_{Ph}$ ($C_{Ph}$ is the background contribution/lattice contribution). The value of the heat capacity jump $\Delta C_p$ in the phase transition region is $\approx 8$ J/mol K. The temperature dependence of $\Delta S$ is also shown in Fig. 1, associated with the disordering of the magnetic system during the phase transition, as determined using the expression: $\Delta S(T) = \int(\Delta C_P/T)dT$. According to our data, the $\Delta S$ is 0.44 J/mol K, which is slightly smaller than that obtained by the authors of work[2] ($\Delta S \sim 0.6$ J/mol K). Both values are much smaller compared with the theoretical value $\Delta S^* = \Delta S_{Mn} + \Delta S_{Cu} = n_{Mn}R\ln(2S(Mn^{3+}) + 1) + n_{Cu}R\ln(2S(Cu^{2+}) + 1) = 25.2$ J/mol K. Such discrepancy between the experimental and theoretical estimates of $\Delta S^*$ is characteristic of many magnetic materials, due to various reasons such as magnetic inhomogeneity[15–17] or it is indicative of the absence of a complete ordering of the magnetic moments at the magnetic phase transition, which agrees with the results from the neutron magnetic scattering data.[2]

Direct measurements of MCE ($\Delta T_{ad}$) were carried out in magnetic fields up to 18 kOe. In work,[18] for $Co_{4.76}Al_{1.24}(O_2BO_3)_2$, the maximum value of the MCE was estimated from the $C_P(T,H)$ data. According to the data,[18] at $T = 80$ K (far from the phase transition temperature $T_N = 57$ K), the maximum $\Delta S$ of 7.5 J/kg K was observed for a field change of 9 T. Figure 2(a) compares values of $\Delta T_{ad}$ in a magnetic field of 18 kOe for two orientations of the crystal [see the inset of Fig. 2(a)]. The maximum value of $\Delta T_{ad}$, obtained from direct measurements in a magnetic field of 18 kOe, is $\Delta T_{\parallel} = 0.45$ K at $T \approx 95$ K. A rotation of 90 degrees leads to a decrease to $\Delta T_{\perp} = 0.30$ K, i.e., the magnitude of $\Delta T_{ad}$ decreases by 1.5 times. In addition, the maximum of $\Delta T_{ad}$ also shifts toward a low temperature $T = 93$ K. As can be seen in Fig. 2(b), the strong anisotropy of the MCE appears below the $T_C$. So, at $T = 82$ K, the ratio $\Delta T_{\parallel}/\Delta T_{\perp} \sim 6.6$, while at $T = T_C$, it is $\sim 1.5$. The observed anisotropy of the MCE is in
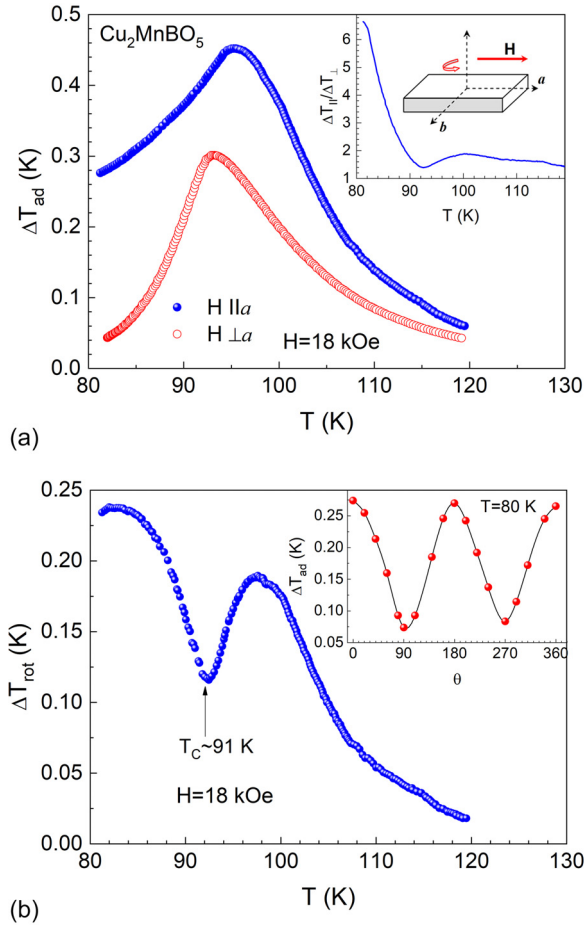
**FIG. 2.** (a) Temperature dependence of MCE ($\Delta T_{ad}$) in a magnetic field of 18 kOe for two orientations of the crystal with respect to the applied magnetic field. The inset of Fig. 2(a) shows the $\Delta T_{||}/\Delta T_{\perp}(T)$ dependence in a field of 18 kOe and the orientation of the crystal in a magnetic field. (b) The adiabatic temperature change $\Delta T_{rot}$ of Cu$_2$MnBO$_5$ caused by a 90° rotation of the crystal as shown in the inset of Fig. 2(a). The inset of Fig. 2(b) shows the angular dependence of $\Delta T_{ad}$ at $T = 80$ K in a field of 18 kOe.

good agreement with the anisotropy of the magnetization of Cu$_2$MnBO$_5$.[2]

Figure 2(b) shows the change in temperature $\Delta T_{rot}$ of Cu$_2$MnBO$_5$ due to a 90° rotation of the crystal within the $ab$ plane, which is defined as the rotating MCE (RMCE). These values of $\Delta T_{rot}$ are obtained by subtracting the curves shown in Fig. 2(a), measured along the two directions of the magnetic field at $H||a$ and at $H\perp a$, i.e., $\Delta T_{rot} = \Delta T_{||a} - \Delta T_{\perp a}$. As one can see from Fig. 2(b), the $\Delta T_{rot}(T)$ dependence shows a minimum near the $T_C$, and the $\Delta T_{rot}$ reaches a maximum at $T = 82$ K, which is 0.24 K at $H = 18$ kOe. Although the $\Delta T_{rot}$ value is not gigantic, it is comparable with the $\Delta T_{H\perp a}$ value for this sample, as well as with that of the RMCE for Gd ($\Delta T_{rot} \sim 0.3$ K at 15 kOe).[19,20] The inset of Fig. 2(b) shows the angular dependence of $\Delta T_{rot}$ at $T = 80$ K in a field of 18 kOe. At present, the record values of $\Delta T_{rot}$ are −4.4 K at $H = 20$ kOe for DyNiSi,[21] 5.2 K at $H = 50$ kOe for HoMn$_2$O$_5$,[22] and –1.6 K at $H = 13$ kOe for NdCo$_5$.[23] It is also

important to note that the RMCE is reversible [see the inset of Fig. 2(b)], which is a necessary condition for a magnetocaloric material to be used in magnetic cooling technology. Balli *et al.* proposed[22,24] a rotating magnetic refrigerator in which the regenerator consists of single-crystal blocks of HoMn$_2$O$_5$ or TbMn$_2$O$_5$, and is rotated in a constant magnetic field. The idea of creating a thermomagnetic generator based on the RMCE was proposed at an earlier time though.[25]

The observed anomaly in $\Delta T_{rot}(T)$ near the $T_C$ is also worthy of note. This anomalous behavior was not observed previously when studying the anisotropy of the MCE in magnetic single crystals. According to work,[26] an equation for determining the adiabatic temperature change of a ferrimagnet taking into account the contribution of two magnetic sublattices can be written in the following form:

$$\Delta T_{ad} = \frac{T}{C_{P,H}} \left( \frac{\partial \overrightarrow{M_1}}{\partial T} d\overrightarrow{H} + \frac{\partial \overrightarrow{M_2}}{\partial T} d\overrightarrow{H} \right),$$

where $M_1$ is the magnetization of the first sublattice, $M_2$ is the magnetization of the second antiparallel sublattice, d$H$ is the magnetic field increment, and $C_{P,H}$ is the heat capacity. For Cu$_2$MnBO$_5$, these are the Mn$^{3+}$ and Cu$^{2+}$ sublattices, which determine positive and negative exchange interactions in the system. It is suggested that the minimum in $\Delta T_{rot}(T)$ near the $T_C$ is a combination of several mechanisms. First, magnetostriction can also make a significant contribution to the MCE along with the paraprocess.[26] It was shown in work[5] that the magnetostriction anisotropy of Cu$_2$MnBO$_5$ has an unusual form; for a parallel configuration of $H||c$, the crystal in the fields under consideration up to 20 kOe is compressed along the $c$-axis. At the same time, for the $H\perp c$ configuration, the magnetostriction has a usual quadratic form (the crystal expands). It is generally known that magnetostrictive contributions to MCE, depending on whether the crystal is compressed or expanded, will have opposite signs.[27] Another mechanism causing the anomaly in $\Delta T_{rot}(T)$ near the $T_C$ could be related to the temperature shift of the MCE maxima at different orientations of the crystal in a magnetic field [see Fig. 2(b)], due to the competition of positive and negative exchange interactions in Cu$_2$MnBO$_5$.

As is known, the energy of magnetocrystalline anisotropy (MCA) is determined through the MCA constants, which determine the RMCE. According to work,[25] the magnetic contribution to the entropy caused by the rotation of the magnetization vector is equal to

$$\Delta S_{anis} = -\frac{\partial \Delta E_{anis}}{\partial T}, \tag{1}$$

where $\Delta E_{anis}$ is the change in the anisotropy energy upon rotation of the sample in a magnetic field from the $b$-axis to the $a$-axis. For rotation of the magnetization vector in the $ab$-plane near the Curie temperature, taking into account the first anisotropy constant, the change in the anisotropy energy can be written in the following form:

$$\Delta E_{anis} = K_1 \left( \sin^2 \Theta_H - \sin^2 \Theta_0 \right), \tag{2}$$

where $\Theta_H$ is the angle between the $a$-axis and the magnetization vector in the field and $\Theta_0$ is the angle between the $a$-axis and the magnetization vector without the field. Thus, the magnetic contribution to the entropy caused by the rotation of the magnetization vector in the prismatic plane is

$$\Delta S_{anis} = -\frac{\partial K_1}{\partial T}\left(\sin^2\Theta_H - \sin^2\Theta_0\right). \qquad (3)$$

As a rule, in the temperature region near the Curie temperature, the anisotropy field is small and the magnetization vector completely rotates in the direction of the magnetic field. Therefore, in our case, from Eq. (3), we can drive an equation for the RMCE,

$$\Delta S_{rot} = -\frac{\partial K_1}{\partial T}. \qquad (4)$$

As can be seen from Eq. (4), the value of the rotational entropy is determined by the temperature behavior of the first anisotropy constant. For this reason, the observed minimum cannot be explained within the framework of the classical theory of magnetocrystalline anisotropy. Earlier study on the RMCE near the Curie temperature was carried out.[28] One broad maximum of the RMCE was observed at a temperature well below the Curie temperature. An increase in the magnetic field led to an expansion of the RMCE. In this case, the existence of a complex temperature dependence of the RMCE can be associated with the anisotropy of the paramagnetic susceptibility. In work,[3] at temperatures above the Curie temperature, it was shown that the magnetic susceptibility has a strong dependence on the orientation of the single crystal in a magnetic field. In work,[29] the influence of the orientation of a monoclinic $Nd_2Ti_2O_7$ single crystal in the magnetic field on the value of the paramagnetic Curie point was found. This phenomenon itself could lead to the appearance of two maxima in the temperature dependence of the RMCE in our sample.

To use expression (4), it is necessary to know the magnetocrystalline anisotropy constants, which can be obtained from the magnetization vs magnetic field curves along the hard axis of magnetization. In this paper, by definition, we consider

$$\Delta T_{rot} = \frac{-T}{C_P(T,H)}\Delta S_{rot}. \qquad (5)$$

Therefore, we can use the MCE data obtained from direct measurements (Fig. 2) and the $C_P(T)$ data measured at the same magnetic field
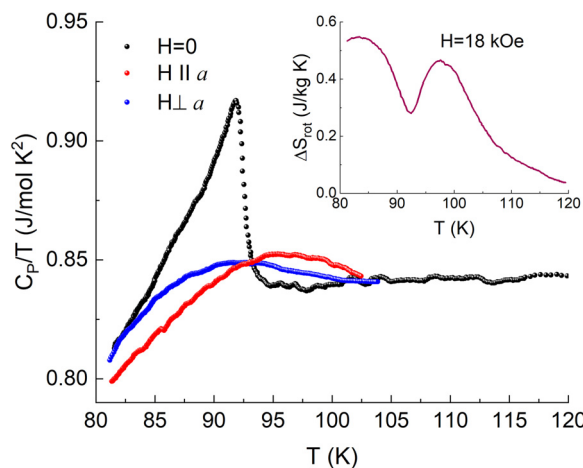
configurations (Fig. 3). Figure 3 shows the $C_P(T)/T$ dependence at $H = 0$ and at 18 kOe for two orientations of the crystal with respect to the applied magnetic field. It can be observed that the application of a 18 kOe field almost completely suppresses the anomaly of $\Delta T_{rot}(T)$. The orientation of the magnetic field in $H \perp a$ caused a change in the temperature dependence of $\Delta T_{rot}$ and a shift of the maximum temperature by 4 K toward low temperatures in comparison with the $\Delta T_{rot}(T)$ curve at $H \| a$, i.e., the slight anisotropy is observed. By taking into consideration of the data shown in Fig. 3, $\Delta S_{rot}(T)$ can be written in the following expression:

$$\Delta S_{rot}(T) = \Delta S_{H\|a}(T) - \Delta S_{H\perp a}(T)$$
$$= \frac{1}{T}\left(C_P^{H\|a}(T,H)\cdot\Delta T_{H\|a}(T) - C_P^{H\perp a}(T,H)\cdot\Delta T_{H\perp a}(T)\right). \qquad (6)$$

The result of the estimated rotating magnetic entropy change ($\Delta S_{rot}$) as a function of temperature in a magnetic field of 18 kOe using expression (6) is presented in the inset of Fig. 3. The temperature dependence trend of $\Delta S_{rot}$ (the inset of Fig. 3) is similar to that of $\Delta T_{rot}$ [Fig. 2(b)].

In summary, we have studied the specific heat and magneto-caloric properties of $Cu_2MnBO_5$ over the temperature range of 60–350 K in magnetic fields up to 18 kOe. At temperatures below the $T_C$, $C_P(T)/T$ shows a linear temperature-dependent behavior, which is associated with the predominance of two-dimensional antiferromagnetic interactions of magnons with a linear dispersion relation propagating in the walls of the ludwigite. The temperature-independent behavior $C_P/T = f(T)$ observed in the temperature range of 95–160 K is likely associated with the excitation of the Wigner glass phase. Due to its strong magnetocrystalline anisotropy, the MCE anisotropy, and consequently the RMCE, is observed in $Cu_2MnBO_5$. The deep minimum of the RMCE is observed near the $T_C$, which is likely associated with the anisotropy of the paramagnetic susceptibility.

## AUTHORS' CONTRIBUTIONS

All authors contributed equally to this work.

## DATA AVAILABILITY

The data that support the findings of this study are available from the corresponding author upon request.



**FIG. 3.** $C_P(T)/T$ dependence at $H = 0$ and 18 kOe for two orientations of the crystal with respect to the magnetic field direction, $H\|a$ and $H\perp a$. The inset shows the $\Delta S_{rot}(T)$ at $H = 18$ kOe.

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