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Magnetic phase transition and magnetic structure of ground state in $CsDyW_2O_8$

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Abstract. Magnetic phase transition in the $CsDyW_2O_8$ magnet has been studied by means of low temperature specific heat $C(T)$ measurements. The magnetic ordering temperature of the Dy^{3+} sublattice was established to be 1.34 K. The experimental results indicate on the antiferromagnetic character of interactions between Dy^{3+} ions. The behavior of the $C(T)$ dependencies above and below T_N is discussed in frames of different theoretical models. The measurements data on temperature and field dependencies of magnetization are used to calculate the exchange and dipole-dipole interactions energy and to determine the possible magnetic structure of the ground state.

PACS. 65.40.-b Thermal properties of crystalline solids – 75.30.Et Exchange and superexchange interactions – 75.30.Cr Saturation moments and magnetic susceptibilities

1 Introduction

The alkaline-rare-earth double tungstates of the general formula $AReW_2O_8$ ($ARe(WO_4)_2$, AReW), where A and Re are the alkaline and rare-earth ions, respectively, are of special interest because of manifestation of the cooperative Jahn-Teller effect as the origin of the structural phase transition (SPT) for these low-symmetry (monoclinic) compounds. To this time the SPT of the cooperative Jahn-Teller type from paraelastic to antiferroelastic state were found at $T_{SPT} = 6.38$ K for KDyW and at 4.9 and 9.0 K for RbDyW in thermodynamical (specific heat including measurements in magnetic field, thermal expansion), spectroscopic (optical absorption of electron states, EPR, optical absorption of phonon states, Raman scattering), magnetic (magnetization including measurements under pressure, magnetic susceptibility) and structural (neutron diffraction) experiments [1–6]. At subkelvins region of temperatures the magnetic phase transitions (MPT) were observed. The antiferromagnetic (AFM) ordering temperatures of Dy^{3+} sublattice were shown to be 0.6 and 0.82 K for KDyW and RbDyW, respectively [7,8].

The measurements presented below are a continuation of study of magnetic properties of alkaline-rare-earth double tungstates in the paramagnetic and magnetically ordered phases. As far as we know, there is no publications devoted to magnetic properties studies of $CsDyW_2O_8$ (CsDyW). Some non-magnetic data concerning the cesium mixed double tungstates have recently been published [9].

The absence of non-equivalent Dy^{3+} ions in CsDyW makes this compound a very attractive object for both magnetic measurements and interpretation of experimental results.

This paper reports on the results of specific heat and magnetization measurements in the CsDyW double tungstate as a function of temperature and magnetic field. The magnetic investigations of CsDyW are prompted by a desire to establish the character and temperature of the Dy^{3+} ions magnetic ordering, to calculate the exchange and dipole-dipole interaction energies and to determine the possible magnetic structure of the ground state.

2 Samples and experimental

The $CsDyW_2O_8$ compound was synthesized using the reaction of $Dy_2O_3 + Cs_2CO_3 + 4WO_3 + 4CsCl$ at temperature 1023–1053 K up to a complete evaporation of CsCl. At preparation of the low temperature modification of CsDyW, one of the main problem is that CsCl can enter into reaction and essentially pollute the compound. Growth of the CsDyW single crystals is difficult because at the polymorphic transformation the crystals either completely destroy or their quality sharply deteriorate. Therefore in our measurements we use the pressed powder CsDyW samples. The high-temperature modification of CsDyW has a monoclinic symmetry with the lattice

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parameters: $a = 0.9261$ nm, $b = 0.5206$ nm, $c = 0.7443$ nm and angle $\beta = 92.51°$ [10,11]. The unit cell of CsDyW contains two formula units. The Dy ion is surrounded by eight oxygen atoms and has local symmetry C_2. Detailed measurements of the CsDyW structure at low temperature have not yet been carried out.

The low temperature specific heat measurements have been performed using a computer controlled quasi-adiabatic calorimeter over a temperature range of 0.5–25 K. For these experiments a ^{3}He cryostat was used. The temperature of sample was determined by two Ru_2O thermometers. The calorimeter and experimental details have been described in [12]. In thermal experiments the pressed powder pellet with diameter of 5 mm and thickness of 0.5 mm was measured. The magnetization measurements were performed using the vibrating sample magnetometer in the temperature range from 4.2 to 240 K and in magnetic field up to 15 kOe. The pressed powder sample had the shape of cylinder whose diameter and length were approximately equal to 2.5 and 4.5 mm, respectively. The specific heat investigations described in this paper were performed up to 20 K only, due to the limitations of the experimental set-up.The accuracy of the specific heat measurements does not exceed 1%.

3 Experimental results

3.1 Heat capacity

The zero-field specific heat of CsDyW as a function of temperature is displayed in Figure 1. The $C(T)$ dependence is similar to the low temperature behaviour of the related KDyW and RbDyW double tungstates. In the (K, Rb)DyW compounds, the peak of $C(T)$ dependence has been shown to be due to the antiferromagnetic ordering of the Dy^{3+} sublattice. By analogy and on the base of the below presented results, we assume that observed in this paper the $C(T)$ anomaly with the peak at the temperature 1.34 K is also connected with AFM phase transition. This anomaly has clearly defined λ-type shape and its value at the Néel temperature (T_N) is considerably reduced in comparison with the KDyW and RbDyW compounds. A nonsymmetrical shape of the $C(T)$ anomaly in the vicinity of T_N is shown below to be due to the fact that the specific heat temperature dependence has a different character below and above T_N.

It should be noted that the contributions to $C(T)$ from both the lattice specific heat and crystal field splitting are negligible small in the temperature region studied. The estimation of the contribution of the crystal field splitting to the total specific heat of CsDyW at low temperatures shows that it is less than 1%. The Debye temperature is high (about 280 K) and therefore the lattice specific heat contributes less than 4% to the total specific heat at $T < 4.0$ K. The CsDyW tungstate is an insulator and therefore the conduction electrons do not contribute to the specific heat.

Taking into account the existence of strong anisotropy in the double tungstates we have analyzed the $C(T)$ dependence above and below T_N in the framework of the theoretical models for quadratic (2D Ising) and XY lattices as well as for simple cubic (3D Ising) (Fig. 1). The comparison between experimental data and theoretical $C(T)$ predictions has been performed up to 3.5 K only because of the influence of the tail of the structural phase transition on the $C(T)$ dependence at higher temperatures as shown below.

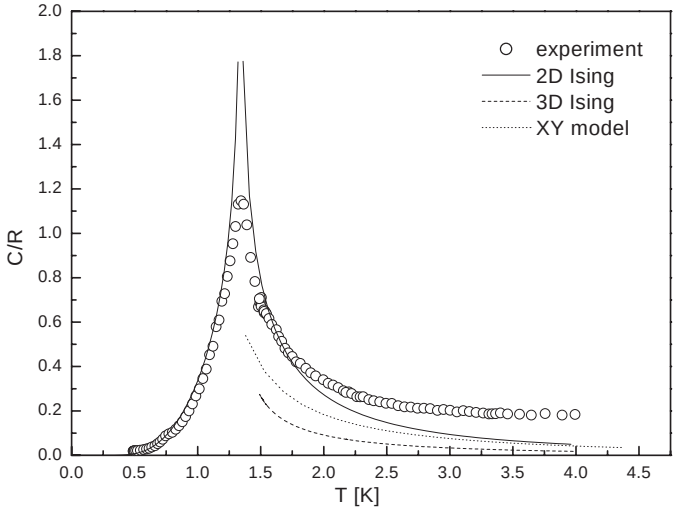

Fig. 1. Experimental and theoretical dependencies of the specific heat of the $CsDyW_2O_8$ compound near magnetic phase transition.

The analysis of the experimental results gives no definite answer which model is the best to describe the $C(T)$ dependence near T_N. As it is seen in Figure 1, the experimental data below T_N are in agreement with the 2D Ising model. At $T_N < T < 1.75$ K, the behavior of specific heat correlates with theoretical $C(T)$ dependence for 2D Ising model better than with 3D and XY ones. At $T > 1.75$ K, none of the models presented describe the experimental temperature dependence of specific heat.

The models discussed above take into account the fluctuations in the critical region (near the phase transition point) where their influence is large. At $T > 2$ K, when influence of fluctuations falls off, the high temperature expansion with accuracy of the order of T^{-2} [13,14] for the specific heat within the framework of the 3D Heisenberg model can be used. In the temperature range limited between 2.1 and 3.5 K, the specific heat is close to the T^{-2} law, namely, $C/R = 0.76\ T^{-2}$ (Fig. 2). Here, we suppose that the T^{-2} term is the tail of the Schottky anomaly resulting from the energy splitting of the lowest Kramers doublet and is caused by fluctuation of the effective spin of the lowest doublet. The maximum deviation of the experimental data from linearity in the temperature range from 2.1 to 3.5 K is of the order of 5%.

Using the experimental dependence of $C/R = 0.76\ T^{-2}$ and the relation of $8C_M T^2/R = zJ^2/k_B^2$ where $z = 6$ is the coordination number of the simple cubic lattice [13,14], the exchange interaction parameter (J/k_B) was obtained to be equal to 1.01 K.

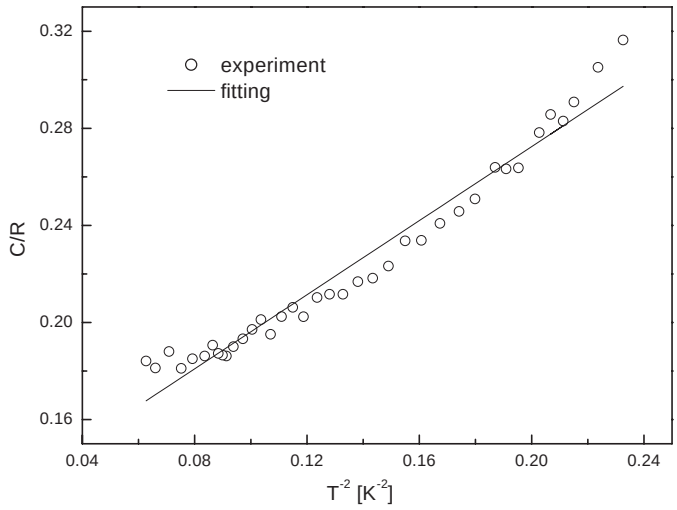

Fig. 2. Magnetic specific heat of $CsDyW_2O_8$ above T_N. In the temperature range of 2.1–3.5 K it follows a $1/T^2$ law (solid line).

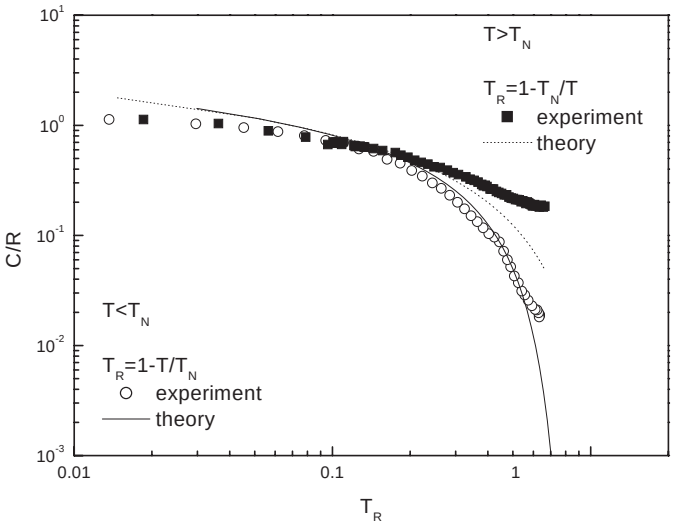

Fig. 3. Log-log plot of C/R as a function of the reduced temperature T_R.

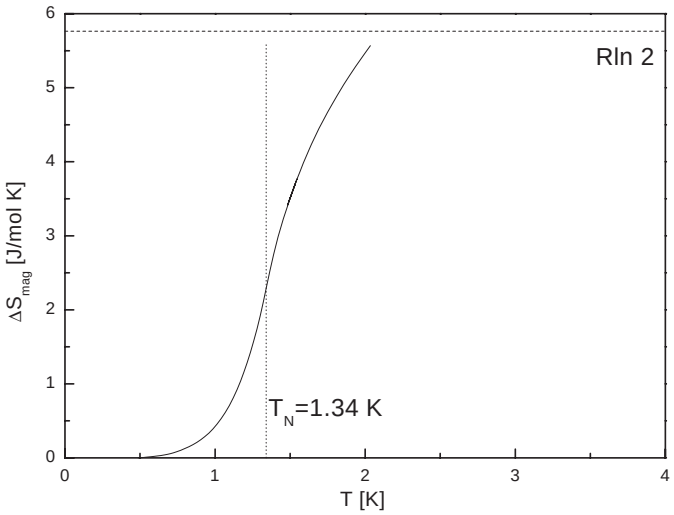

Fig. 4. Change of entropy with temperature neat T_N.

The equation of $J/k_B = -T_N(\ln\sqrt{2}-1)$ was obtained using the exact expression of the $C(T)$ dependence by Onsager [15] for 2D antiferromagnet. In this case, when $T_N/2J = 0.567$ and $z = 4$, the value of $J/k_B = 1.18$ K.

In contrast, the exchange parameter of $J/k_B = 0.59$ K was calculated using the high temperature expansion for a specific heat within the framework of the 3D Ising model [16] taking into account the relation between the Néel temperature and parameter J in the form of $T_N/zJ = 0.376$ (the coordination number of the simple cubic lattice $z = 6$ and the effective spin $S = 1/2$ were used). Using the relation of $k_BT_N/J = 2.02$ obtained for the XY model [17], the exchange parameter was determined to be $J/k_B = 0.663$ K.

The comparison of the J/k_B values obtained shows that the experimental and theoretical (in the framework of the 2D Ising model) J/k_B parameters are in reasonable agreement.

Figure 3 shows a log-log plot of the specific heat *versus* reduced temperature T_R ($T_R = 1 - T/T_N$ for $T < T_N$ and $T_R = 1 - T_N/T$ for $T > T_N$). The slope of the curves $\log(C/R)$ *vs.* $\log T_R$ gives the critical exponents α and α' directly determining the character of the magnetic phase transition in the limit $T \to T_N$ above and below T_N. As one can see in Figure 3, the critical exponents for CsDyW are close to zero that corresponds to the theoretical ones predicted by the 2D Ising model.

The experimental $C(T)$ dependence allows to determine a change of magnetic entropy with temperature $[\Delta S_{mag}(T)]$ near the magnetic phase transition (Fig. 4). The $\Delta S_{mag}(T)$ plot obtained by integrating the specific heat $\Delta S_{mag}(T) = \int_0^T [C(T)\mathrm{d}T/T]$ shows that $\Delta S_{mag} \approx 0.4\ R\ln 2$ at 1.34 K and can reach the $R\ln 2$ limit at temperature $T \approx 2.0$ K. At temperatures above 2 K, where C_{mag} obeys a T^{-2} variation, $\Delta S_{mag}(T)$ exceeds $R\ln 2$ value. By analogy with the related compounds (K, Rb)$Dy(WO_4)O_2$, the similar magnetic entropy behaviour is evidently due to an influence of high temperature structural phase transition resulting in a change of the distance between the lowest two doublets of the Dy^{3+} ion. It can also be the reason that none of the models presented properly describe the experimental temperature dependence of specific heat above 1.75 K.

We have also studied the magnetic field effect (up to 10 kOe) on the specific heat. The observed increase of the specific heat ($H = 0$) at $T > 5$ K (Fig. 5) is supposed to be due to the influence of the tail of the structural phase transition at higher temperature (see Ref. [1–4]). As can be seen in Figure 5, the peak of the specific heat corresponding to the magnetic phase transition shifts to lower temperatures and the $C(T)$ maximum decreases in magnitude with increasing magnetic field. The magnetic field of 5 kOe decreases T_N by about 0.11 K only, and the $C(T)$ anomaly is no longer observed in field of 10 kOe. Such temperature behaviour of the λ peak position in magnetic field is expected for antiferromagnetically ordered systems. It

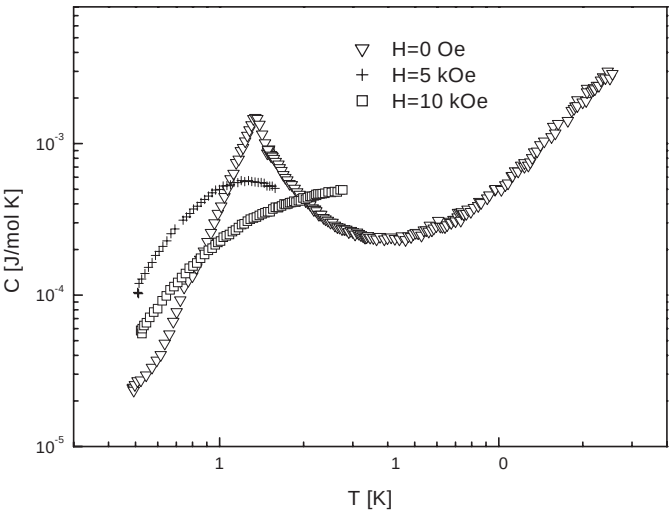

Fig. 5. Magnetic field effect on the $C(T)$ dependence in $CsDyW_2O_8$.

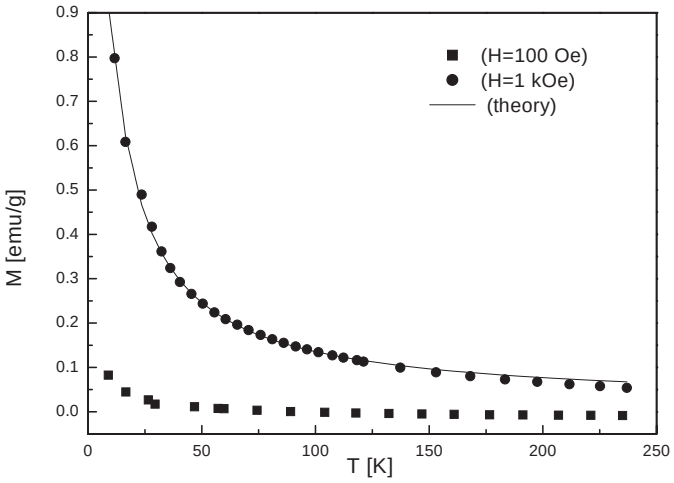

Fig. 6. Temperature dependencies of magnetization at applied magnetic fields of 0.1 (solid squares) and 1 kOe (solid circles). The line (1 kOe) corresponds to fitting by Curie-Weiss law.

is connected with that in magnetic field the system will order and the interaction energy will decrease. There is an analytic expression for T_N decrease in magnetic field: $T_N(H)/T_N(0) = \{(1 - H/H_C)^2\}^\alpha$ where α is the critical index depending on the lattice type. The rough estimation of the effective Weiss field $H_C = zJk_B/(\mu_B gS)$ where $J = 1$ K, $g = 12.6$, $S = 1/2$ gives $H_C = 14$ kOe. It is very close to the field value of 10 kOe when the phase transition destroys.

3.2 Magnetization

Figure 6 presents the temperature dependencies of magnetization at applied magnetic fields of 0.1 and 1 kOe. The fitted temperature dependence of magnetization at $H = 1$ kOe determines the Curie-Weiss temperature $T_\theta = -\theta = -4.1$ K. The sign of the T_θ temperature confirms the antiferromagnetic character of interactions.

The experimental and theoretical field dependencies of magnetization $M(H)$ at temperature $T = 5$ K are presented in Figure 7. As can be seen, the magnetization saturation is not reached up to 16 kOe at 5 K.

The Brillouin $M(H)$ dependence was calculated using the experimentally determined Curie-Weiss temperature θ which gives $J/k_B = 1.37$ K for $S = 1/2$ (see Eq. (2)). The best fitting of the theory and experiment was obtained for the spectroscopic splitting factor $g = 12.6$. In this calculation the dipolar energy E_α was neglected and hence J has meaning of mean value determined using powder sample. Then the saturation magnetization value of $M_{SAT} = 44.5$ emu/g will be reached at fields of about 40–45 kOe. It is known that the susceptibility can be described using the Curie-Weiss law:

$$M/H = \chi = C/(T + \theta), \quad (1)$$

where $C = N_A(\mu_B g)^2 S(S + 1)/3k_B$, N_A is the Avogadro number, μ_B is the Bohr magneton and k_B is the

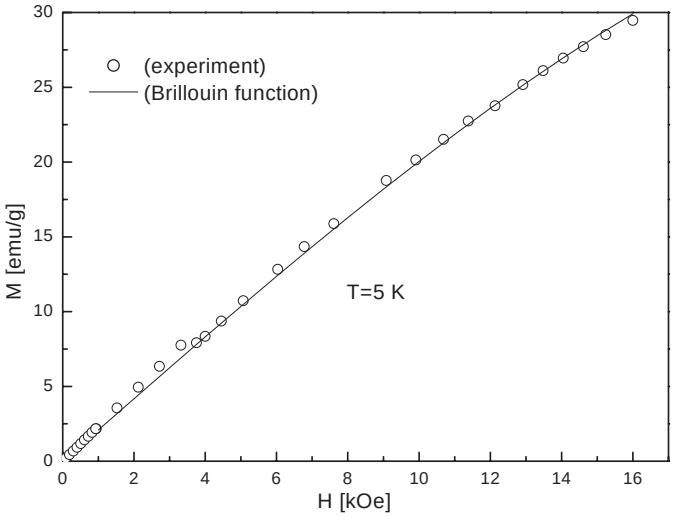

Fig. 7. Experimental (circles) and theoretical (line) magnetic field dependencies of magnetization at $T = 5$ K.

Boltzmann constant. Fitting of the experimental data to the Curie-Weiss law in the temperature interval from 30 to 120 K, the values of $C = 9.8$ cm^3 K/mol and $g = 10.2$ for $S = 1/2$ were calculated. Some difference of the g-factor values calculated at $T = 5$ K and at $T > 30$ K seems to be connected with an influence of the structural phase transition at $T \sim 30$ K.

The J/k_B parameter is known to consist of two contributions related to the exchange and dipole-dipole interactions. Taking into account that the magnetic phase transition in CsDyW takes place at low temperature, it can be assumed that the contribution of dipole-dipole interactions to spin-spin interactions may be essential.

Let us estimate the values of the exchange (J) and dipole-dipole energy (E_a) on the basis of our experimental results. The expression for the paramagnetic Curie-Weiss temperature along one of the crystallographic directions

has the form:

$$\theta_\alpha = \frac{2}{3}S(S+1)\left(zJ^\alpha + E_\alpha\right) \tag{2}$$

where $z = 6$ is the number of the nearest neighbors, index α designates the **a**, **b** and **c** axes direction in the unit cell. In the consideration the anisotropy of the exchange interactions was neglected. The total contribution E of the dipole-dipole interaction to the θ value is:

$$E_\alpha/k_\mathrm{B} = \frac{\mu_\mathrm{B}^2 g_\alpha^2}{k_\mathrm{B}} \sum_j \frac{r_{ij}^2 - 3(r_{ij}^\alpha)^2}{r_{ij}^5} = L^\alpha(1)/k_\mathrm{B} + L^\alpha(2)/k_\mathrm{B}, \tag{3}$$

where r_{ij} is the radius vector from ith to jth lattice site and r_{ij}^α is its projection on the αth axis, g_α - g-factor along the α axis. In the expression (3), $L^\alpha(1)$ is the sum over the lattice sites inside the Lorentz sphere and $L^\alpha(2)$ involves both the Lorentz term and the demagnetization factor N of sample, *i.e.*

$$L^\alpha(2) = \left(-\frac{4\pi}{3} + N\right)\frac{(\mu_\mathrm{B} g_\alpha)^2}{V k_\mathrm{B}}, \tag{4}$$

where $2V = abc$ is the volume of elementary cell of pseudo rhombic structure. We choose the Lorentz sphere of radius $R = 10$ nm.

The g-factor in the ground state of the Ising Dy^{3+} ion is highly anisotropic. Unfortunately, the EPR measurements of the g-factor values for $CsDy(WO_4)_2$ were not carried out until now because of absence of single crystals. According to the EPR data, the g-factor values along the main crystallographic directions for the related $KDy(WO_4)_2$ compound are equal to $g_a = 0 \pm 0.2$; $g_b = 1.54$ and $g_c = 14.3$ [18]. Using the relation of the g-factor values along the main crystallographic directions for $KDy(WO_4)_2$ and $g = 12.6$ calculated earlier for $S = 1/2$ by the fitting of the experimental data to the Brillouin law $M(H)$ for low temperature region as well as standard expression $g^2 = (g_c^2\cos^2\theta + g_b^2\sin^2\theta)$, the approximate g-factor values for $CsDy(WO_4)_2$ were assumed to be to $g_a = 0$; $g_b = 1.53$ and $g_c = 13.39$. An approximate analysis of possible magnetic structure of the ground state was made using the exchange parameters J^α obtained below and these g-factors.

The summation of the lattice sums $L^\alpha(1)$ gives the following constants:

$$L^a(1)/k_\mathrm{B} = 0 \text{ K}, \qquad L^b(1)/k_\mathrm{B} = 0.006 \text{ K} \qquad \text{and} \qquad L^c(1)/k_\mathrm{B} = -7.77 \text{ K}.$$

Unfortunately, the axes of crystallites are distributed randomly relative to the sample cylinder axis, and we could not exactly estimate $L^\alpha(2)$. Therefore it is impossible to calculate precisely the contribution connected with the demagnetization. However it is possible to estimate an averaged value of this contribution. Indeed, for the parallelepiped shape sample of size $4 \times 3 \times 1.5$ mm the appropriate components of demagnetization factor are equal $N_z = 2.22$, $N_y = 3.25$ and $N_x = 7.08$. The z' axes of crystallites have a casual angle ω with the fixed z axis of sample. It is also necessary to enter yet the casual angle φ for turn of $X'Y'Z'$ system of crystallite to XYZ system in which the tensor of demagnetization factors is diagonal. Carrying out a transformation of tensor of demagnetization factors with the help of turns to local axes of crystallites, and then performing an averaging over the angles of ω and φ, it is easily to calculate an average values of the demagnetization factors along the parallelepiped sample axes: $\langle N_x\rangle = 0.25(N_x + N_z) + 0.5N_y = \langle N_y\rangle, \langle N_z\rangle = 0.5(N_x + N_z)$. Here the basic ratio of $\langle N_x + N_y + N_z\rangle = 4\pi$ is carried out. Then, in spite of that the contribution in $L^\alpha(2)/k_\mathrm{B}$ connected with demagnetization is rather essential (~2.9 K), the $L^\alpha(2)/k_\mathrm{B}$ value is insignificant (the negative contribution of the Lorentz term compensates a demagnetization). As result, we obtain the following values:

$$L^a(2)/k_\mathrm{B} = 0 \text{ K} \qquad L^b(2)/k_\mathrm{B} = -0.002 \text{ K} \qquad \text{and} \qquad L^c(2)/k_\mathrm{B} = 0.287 \text{ K}.$$

Thus, the complete contribution of dipole interactions to the paramagnetic temperature θ includes mainly a contributions of lattice sums inside the Lorentz sphere which have following values along the appropriate directions:

$$E_a/k_\mathrm{B} = 0 \text{ K}, \qquad E_b/k_\mathrm{B} = 0.004 \text{ K} \qquad \text{and} \qquad E_c/k_\mathrm{B} = -7.483 \text{ K}.$$

Then, using equation (2) and experimental value of the Curie-Weiss temperature θ, we obtain the following values of the exchange interaction parameters:

$$J^a = 1.37 \text{ K}, \quad J^b = 1.37 \text{ K} \quad \text{and} \quad J^c = 2.61 \text{ K}.$$

The obtained values of exchange parameters are in agreement with the data of both specific heat measurements (see above) and analysis in frames of 2D Ising model as the average value of an pair exchange is equal to

$$\left\{J^a + J^b + J^c\right\}/3 \sim 1.78 \text{ K}.$$

The energy of the ground state was determined as a minimum energy for eight possible spin configurations which are realized in the Bravais lattices taking into account a translation invariance. In the unit cell, all magnetic ions were considered to be equivalent. The technical details of calculations are based on the Luttinger and Tisza method for finding an approximation to the ground state [19]. The results of calculation of an energy of the ground state along the **a**, **b** and **c** directions of magnetization are presented in Table 1 where 1–8 are numbers of spin configurations defined in [19]. It is seen that the minimum energy corresponds to a configuration $N = 2$, *i.e.* for a pure antiferromagnetically ordered Dy^{3+} ions with magnetic moments directed along the **c**-axis.

4 Conclusions

The magnetic phase transition in the $CsDyW_2O_8$ magnet has been studied by means of the low temperature specific

Table 1. Energy of the ground state along the **a**, **b** and **c** directions of magnetization.

N	E_a [K]	E_b [K]	E_c [K]
1	8.2	8.2	8.2
2	−8.89	−8.67	−14.46
3	−0.08	2.72	8.04
4	10.6	0.21	−0.14
5	0.21	9.88	0.58
6	−3.34	−5.13	−2.21
7	−6.57	2.65	−6.75
8	−2.11	−1.45	−7.12

heat $C(T)$ over a temperature range from 0.5 to 25 K. The magnetic ordering temperature of the Dy^{3+} sublattice was established to be 1.34 K. The peculiarities of the $C(T)$ dependencies above and below T_N are discussed in frames of different theoretical models. The analysis gives no definite answer which model is the best to describe the specific heat behaviour near T_N. The experimental data below T_N are in a good agreement with the 2D Ising model. At $T_N < T < 1.75$ K, the behaviour of specific heat correlates with theoretical $C(T)$ dependence for 2D Ising model better than with 3D and XY ones. At $T > 1.75$ K, none of the models presented properly describe the experimental temperature dependence of the specific heat. At $T > 1.8\ T_N$, the entropy exceed the value of the molar entropy R ln2 of the electronic doublet of the Dy^{3+} ion ground state. The peak of the specific heat corresponding to the magnetic phase transition shifts to lower temperatures and the height of the $C(T)$ maximum decreases with increasing magnetic field. Analyzing the magnetization data, the values of the Curie-Weiss temperature θ and g-factor were calculated. The exchange parameter J/k_B was estimated by various methods. It was shown that the experimental exchange parameter J/k_B value is in reasonable agreement with J/k_B one for 2D Ising model only. The signs of both J/k_B and θ indicate on the antiferromagnetic character of Dy^{3+} ions interactions. Data of magnetization measurements were used to calculate the exchange and dipole-dipole interactions energy, taking in account the strong anisotropy of the g-factor, and tentatively determine the magnetic structure of the ground state. Since the magnetic investigations were performed using the powder samples it was impossible to calculate precisely the contribution connected with the demagnetization. An approximate analysis of possible magnetic structure of the ground state of $CsDyW_2O_8$ was made. The dipole contribution was shown to be comparable to the value of the exchange interactions along the **c** direction. The possible magnetic structure was established to present the 3D system of pure antiferromagnetically ordered Dy^{3+} ions with magnetic moments directed along the **c**-axis, in contrast to the KDyW and RbDyW crystals wherein the antiferromagnetic moments are directed along the **b**-axis.

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