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The magnetic properties of copper(II) maleate monohydrate, $Cu(C_4H_2O_4)\cdot H_2O$, have been measured in the 1.8-66 K range. Magnetic susceptibility and isothermal magnetization measurements on powdered samples reveal a transition to a ferromagnetic state in this layered compound. The transition to the ordered state occurs at T_c = 4.0 K. The positive value of 8.0 K for the Weiss constant in the paramagnetic region confirms the ferromagnetic nature of the interactions. The magnetic susceptibility data above $3T_c$ may be fit with a Heisenberg quadratic layer expression with the best-fit parameters $J = +4.3/cm^{-1}$, g = 2.0, and $zJ' = +0.42/cm^{-1}$. The e.p.r. spectrum at room temperature indicates orthorhombic symmetry. A complex containing the isomeric form of the ligand, copper(II) fumarate dihydrate, has also been prepared and its magnetic properties determined in the 80-300 K range. The data may be accomodated by two-dimensional theory with $J = +1.05\ cm^{-1}$, g = 2.09, and $zJ' = +0.10\ cm^{-1}$.

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Ferromagnetic Long Range Ordering in Copper(II) Maleate Monohydrate

by

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Ferromagnetic long range ordering in copper(II) maleate monohydrate

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Summary

The magnetic properties of copper(II) maleate monohydrate, $Cu(C_4H_2O_4)\cdot H_2O$, have been measured in the 1.8-66 K range. Magnetic susceptibility and isothermal magnetization measurements on powdered samples reveal a transition to a ferromagnetic state in this layered compound. The transition to the ordered state occurs at T_c = 4.0 K. The positive value of 8.0 K for the Weiss constant in the paramagnetic region confirms the ferromagnetic nature of the interactions. The magnetic susceptibility data above $3T_c$ may be fit with a Heisenberg quadratic

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layer expression with the best-fit parameters J = +4.3 cm^{-1}, g = 2.0,and zJ' = +0.42 cm^{-1}. The e.p.r. spectrum at room temperature indicates orthorhombic symmetry. A complex containing the isomeric form of the ligand, copper(II) fumarate dihydrate, has also been prepared and its magnetic properties determined in the 80-300 K range. The data may be accomodated by two-dimensional theory with J = +1.05 cm^{-1}, g = 2.09, and zJ' = +0.10 cm^{-1}.

Introduction

As part of our program devoted to the identification and characterization of insulating ferromagnets, we have studied the magnetic properties of copper(II) maleate monohydrate (Cu-Mal), since the x-ray structural results[2] show carboxylate bridging of the type found in copper(II) compounds with ferromagnetic interactions. The copper(II) ion in Cu-Mal has square pyramidal coordination. As shown in Figure 1, the apical ligand is a water molecule located at 2.26 A, and the donor atoms in the square plane are two oxygen atoms of a single maleate chelating ligand, and two oxygen atoms from two additional different maleate groups. The copper-oxygen bonds in the square plane are equal in length (1.99 A), and the cis-oxygen atoms are at right angles. Each maleate group is bonded to three different copper atoms forming a polymeric sheet. The copper maleate sheets are linked by hydrogen bonds (2.79 A) between the water molecule and the carboxyl oxygen atom[2], as indicated by the dashed lines in Figure 1. The shortest Cu-Cu distance within a layer is 5.12 A, and the shortest Cu-Cu distance between two adjacents layers is 5.98 A. Results of our magnetic studies on Cu-Mal, which show interactions in

the plane followed by long range ordering, are described here. Magnetic data for copper(II) fumarate dihydrate (Cu-Fum), are also presented. This latter compound was included in the study since the fumarate and maleate ligands are related by being trans- and cis- isomers, respectively.

Experimental

Synthesis

Maleic acid (10 g) (Chemical Dynamics Corp., South Plainfield, N.J.) was dissolved in distilled H_2O (200 mL) and NaOH (6.9 g) was added slowly with stirring. Copper(II) chloride dihydrate (14.7 g), dissolved in hot water (200 mL), was then slowly added to the sodium maleate solution with vigorous stirring. After cooling to room temperature the blue precipitated product was isolated by filtration, washed with a little cold water and air dried. The yield was almost quantitative. Copper(II) fumarate dihydrate was prepared in a similar way. Elemental analysis was done by Galbraith Laboratories, Knoxville, TN. Found: C, 24.3; H, 2.2; Cu, 32.6. $CuC_4H_4O_5$ (Cu-Mal) calcd.: C, 24.6; H, 2.1; Cu, 32.5%. Found: C, 22.5; H, 3.0; Cu, 30.0. $CuC_4H_6O_6$ (Cu-Fum) calcd.: C, 22.5; H, 2.8; Cu, 29.8.

Magnetic measurements

Magnetic susceptibility measurements for Cu-Mal were determined in the 1.8 to 66 K range by using a vibrating sample magnetometer (VSM)[3,4] at different magnetic fields (10,000, 100, and 2 Oe). The VSM magnetic field was calibrated against n.m.r. resonances (^1H and ^3Li). Magnetization was measured as a function of the applied magnetic field (50 to 15,000 Oe) both above and below the transition temperature to

the ordered state. Magnetic susceptibility measurements for Cu-Fum were determined in the 80-300 K range using a Faraday type balance consisting of a Cahn 2000 electrobalance equipped with an Anac 3472 HA electromagnet and 502 Lewis coils from George Associates, Berkeley, CA. E.p.r. spectra were recorded with a Varian E-109 spectrometer operated at X-band frequency. The free-radical DPPH ($g = 2.0023$) was used as a field marker. The TIP correction for copper(II) was taken as $+ 60 \times 10^{-6}$ cgsu, and the diamagnetic corrections for constituent atoms in Cu-Mal and Cu-Fum were obtained by using Pascal's constants[5-7].

Results

The effective magnetic moment data as a function of temperature for a powdered sample of Cu-Mal are shown in Figure 2. The magnetic moment increases from 1.9 B.M. at 66 K to almost 4.4 B.M. at 5.9 K. The increase at low temperatures strongly suggests a ferromagnetic interaction. The magnetic susceptibility data collected at 10,000 Oe in the 12 K range (i.e. $3T_C$) to 66 K may be fit by the Curie-Weiss law $X = C/(T - \theta)$, where $C = Ng^2\mu_B{}^2S(S+1)/3k$, with the best fit parameters $g = 2.11$ and $\theta = 8.0$ K. The positive value for the Weiss constant is also indicative of ferromagnetic interactions in Cu-Mal.

Since small values of the applied magnetic field affect the ferromagnetic properties, the transition temperature was determined from data collected with a very low applied magnetic field ($H = 2$ Oe). The Curie temperature, 4.0 K, was estimated to be the point of maximum slope in the plot of the magnetic susceptibility versus temperature data given in Figure 3.

The temperature dependence of the magnetization is shown in Figure

4 at 11 K, 4.2 K, and 2.5 K. The plot shows saturation of the magnetization at very low applied magnetic fields and confirms the presence of ferromagnetism.

In view of the layered structure of Cu-Mal and the nearly quadratic lattice (a/b = 1.11), it was anticipated that the magnetic properties could be explained by using the theoretical expansion developed by Baker for a square lattice structure[8]. The exchange Hamiltonian for a two-dimensional Heisenberg ferromagnet is given by Equation (1)

$$H = -2J \sum S_i \cdot S_j \qquad (1)$$

where Σ runs over all pairs of nearest neighbor spins i and j. The Baker power series expansion is given by Equation (2)

$$X_B = Ng^2\mu_B^2 / kT \left[1 + \sum (\alpha_n / 2^n n!)\, x^n \right] \qquad (2)$$

where $x = J/kT$, and the coefficients α_n for $i < 11$ for the square lattice have been tabulated[8].

The expression for two-dimensional interactions was modified to account for the presence of magnetic interlayer interactions, by adding a mean-field term to give the expression (3)

$$X_M = X_B/(1-2zJ'X_B/Ng^2\mu_B^2) \qquad (3)$$

where X_M is the corrected molecular field value of the molar susceptibility, X_B is the calculated magnetic susceptibility of an isolated layer given by Baker's expansion, z is the number of nearest neighbors in the layer, and J' is the interlayer exchange-coupling

constant. Expression (3) was fit to the experimental data by using a non-linear Simplex fitting numerical technique[9,10]. During the fitting the parameters J, J', and g were allowed to vary freely.

The Baker expansion is valid only at temperatures well above the transition to the ordered state. This common limitation of high-temperature series expansions is a direct consequence of having a limited number of coefficients in the expansion. The experimental data were fitted for the temperature range with $J/kT < 0.6$ (11-66 K). The best fit to the Baker expansion model was obtained with an intralayer exchange coupling constant of $J = +4.3\ cm^{-1}$, an interlayer exchange coupling constant of $zJ' = +0.42\ cm^{-1}$, and $g = 2.00$. Fitting of the data without the mean-field term zJ' resulted in an even lower g-value (1.98).

The e.p.r. spectrum of Cu-Mal, shown in Figure 5, reveals anisotropic g-values with $g_1 = 2.25$, $g_2 = 2.18$, and $g_3 = 2.07$. These values are similar to those frequently seen in copper(II) compounds with orthorhombic symmetry. The average g-value of 2.17 from the e.p.r. measurements is somewhat larger than the g-value obtained from the analysis of the magnetic data with the Baker's expression or with the Curie-Weiss law.

When the average e.p.r. g-value of 2.17 was kept constant during the fitting routine, and J and J' were allowed to vary freely, J was found to be very close to zero, and zJ' was unreasonably high. Similarly, keeping constant the g-value of 2.11, obtained from the Curie-Weiss fit, led to the unreasonable result of interlayer interactions stronger than intralayer interactions.

The experimental data could also be fitted to a three-dimensional Heisenberg model[11] for body-centered and face-centered cubic lattices. When zJ' was not included, for the body-centered lattice the best fitting parameters were $J = 1.75\ cm^{-1}$ and $g = 2.05$, and for the

face-centered lattice J = 1.11 cm^{-1} and g = 2.06. Addition of the interlayer interaction parameter resulted in negative values for zJ': -1.31 cm^{-1} for the body-centered, and -1.54 cm^{-1} for the face-centered lattice. However, the magnetic data reflect ferromagnetic interlayer interactions.

We also measured the magnetic susceptibility of copper(II) fumarate dihydrate in the temperature range 80-300 K using the Faraday balance. Fitting of the data to a Curie-Weiss equation yielded the best fitting parameters g= 2.09 and θ = +3.2 K. Fits to a square lattice using Baker's expansion yielded values for the parameters J = +1.15 cm^{-1}, and g = 2.09. Inclusion of the mean-field term zJ' resulted in the following parameters: J = +1.05 cm^{-1}, g = 2.09, and zJ' = +0.10 cm^{-1}. The positive values for θ and J are indicative of ferromagnetic exchange interactions. The e.p.r. spectrum of Cu-Fum shows axial symmetry with $g_{|}$ = 2.12 and $g_{||}$ = 2.21 at room temperature, and $g_{|}$ = 2.09 and $g_{||}$ = 2.18 at 77 K. Unfortunately, no crystal structure has been reported for Cu-Fum and we will not speculate on the nature of the exchange.

Discussion

Fittings of the magnetic susceptibility data can not distinguish between the two-dimensional quadratic lattice and three-dimensional cubic lattices for Cu-Mal; however, the structural information favors the two-dimensional assignment. The ferromagnetism observed can be accounted for in terms of the square basal plane around copper and the carboxylate groups of the maleate chelate ligand. The σ^* ($d_{x^2-y^2}$) in-plane orbital on copper is properly situated for interactions with the orbitals of the carboxylate bridges permitting ferromagnetic spin-spin

coupling by superexchange. The dihedral angle between the planes defined by the carboxylate oxygen atoms bonded to copper ions within a layer, which is designated τ in Figure 1, was calculated by using a computer program[12] and found to be 77.5°. This angle allows an almost orthogonal orbital pathway, and promotes the ferromagnetic nature of the interactions. There is a second pathway for superexchange through the 7-membered rings, but it is unlikely to make a significant contribution to the superexchange.

Ferromagnetic intralayer interactions predominate in the layer of Cu-Mal, but there are additional interlayer ferromagnetic couplings. These weaker interlayer interactions, which were accounted for through the addition of a mean-field term to the magnetic susceptibility expression, lead to the long range three-dimensional ordering.

From molecular field theory for ferromagnets[13] the effective intralayer exchange field may be calculated from the following expression

$$H_{ex} = 2zJS/g\mu_B \qquad (4)$$

Using the parameters determined in this study, an exchange field equal to 0.92×10^5 Oe was obtained. With data from a powdered sample, it is not possible to determine the in-plane anisotropy. Assuming axial symmetry, the out-of-plane anisotropy may be estimated from the magnetization data. By plotting dM/dH versus field[14], this was determined to be about 1000 Oe.

Superexchange by carboxylate bridges

Copper(II) oxydiacetate hemihydrate[3] is an example of a compound with carboxylate bridges that shows ferromagnetic behavior. The intralayer exchange coupling constant J = +4.66 cm^{-1} is very close to the exchange coupling constant obtained for Cu-Mal. This indicates that the oxydiacetate ligands transmit ferromagnetic intralayer interactions as effectively as the maleate groups.

By contrast, the out-of-plane anisotropy field (which is largely the interlayer exchange field) of copper(II) oxydiacetate hemihydrate is only 250 Oe. The much greater value of 1000 Oe for Cu-Mal reflects the presence of additional interlayer ferromagnetic interactions. This is in agreement with the structural features of both compounds. In copper(II) oxydiacetate hemihydrate the atoms in adjacent layers are held together by van del Waal's forces only, whereas in Cu-Mal the copper sheets are linked by hydrogen bonds. Hydrogen bonds provide a better pathway for interlayer exchange.

Copper(II) formate diureate dihydrate (CuFUH)[15] provides an example of a nearly Heisenberg layer antiferromagnet with carboxyl bridges. CuFUH has two unequivalent copper(II) ions in the unit cell linked by formate molecules to make up a square lattice structure. This compound with J = -22.9 cm^{-1} shows a magnetic phase transition to an antiferromagnetically long-range ordered state at 15.5 K. The exchange parameter was obtained from a fit of the data to a high-temperature series expansion using a two-dimensional Heisenberg model for a quadratic lattice. It is clear the formate ligands provide a strong pathway for antiferromagnetic exchange as evidenced by the large exchange coupling constant and the transition temperature.

Copper(II) hydrogen maleate tetrahydrate (Cu-MTH)[16] is a closely related compound that also shows ferromagnetic interactions. Even

though the maleate ion is common to both compounds, the magnetic parameters are substantially different in magnitude as a result of significant structural differences. Cu-MTH has an intralayer constant $J = 0.6\ cm^{-1}$ with $zJ' = -0.2\ cm^{-1}$. The intralayer constant in Cu-MTH is notably weaker compared to Cu-Mal because the maleate ligands are not directly bonded to the copper ions in Cu-MTH. Instead, the copper ion is bonded to six water molecules, and bonding to maleate ions occur through hydrogen bonds. While the water bridges provide a superexchange pathway, they are not as effective as the maleate bridges in Cu-Mal.

In conclusion, we have found the short-range ordering effects within the layer structure in Cu-Mal dominate the magnetic susceptibility down to the transition temperature, where the effects of the interlayer exchange and the anisotropy initiate the three-dimensional ferromagnetic ordering that occurs below 4.0 K. Below the transition temperature, the ferromagnetically ordered layers are coupled ferromagnetically through the hydrogen bonds.

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Captions for the figures

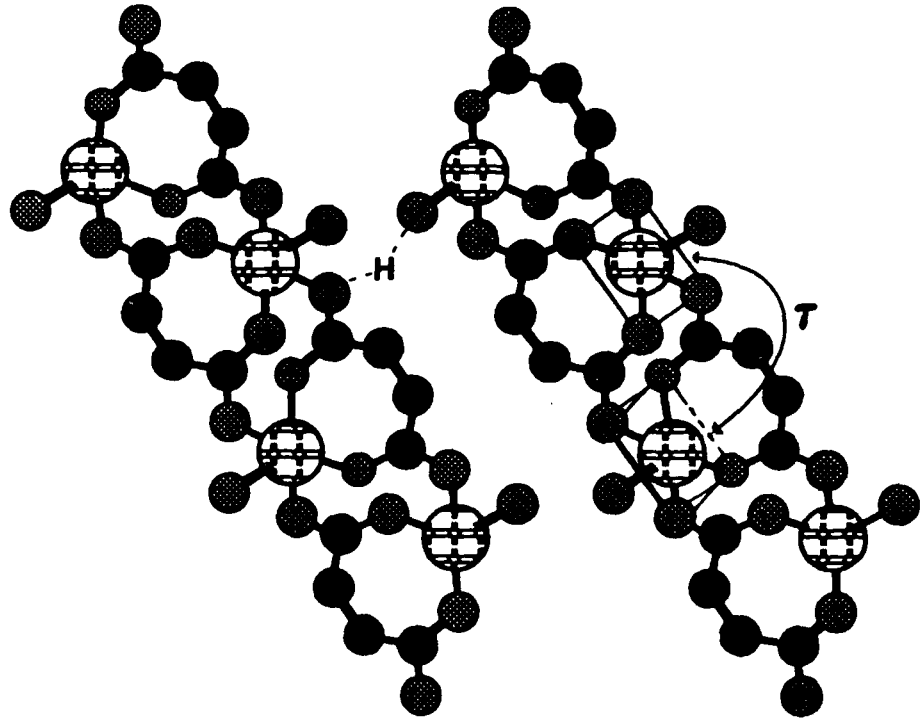
Figure 1. Crystal structure of copper(II) maleate monohydrate showing the hydrogen bond (dashed lines) between adjacent layers. Note that this is an eight-formula unit segment of a polymeric sheet.

Figure 2. Temperature dependence of the effective magnetic moment per copper atom for Cu-Mal. The solid curve represents the best-fit to the experimental data using the parameters $J = +4.3\ cm^{-1}$, $zJ' = +0.42\ cm^{-1}$, and $g = 2.00$.

Figure 3. Plot of the magnetic susceptibility versus temperature for Cu-Mal taken at 100 Oe (squares), and 2 Oe (plus signs).

Figure 4. Isothermal magnetization data collected above and below the transition temperature to the ordered state. Squares, T = 11 K; plus signs, T = 4.2 K; rhombs, T = 2.5 K.

Figure 5. E.p.r. spectrum of Cu-Mal in the solid state recorded at room temperature.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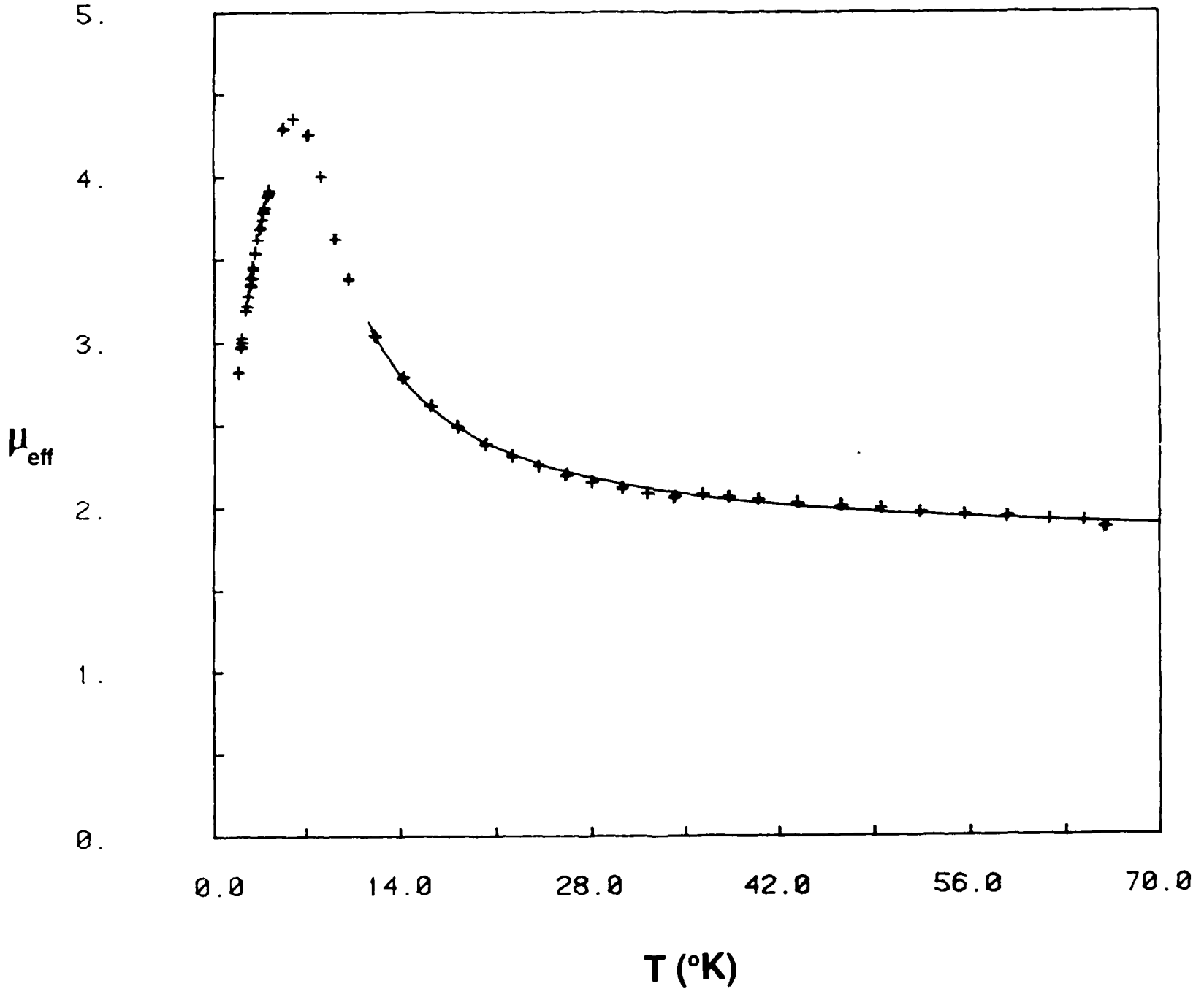
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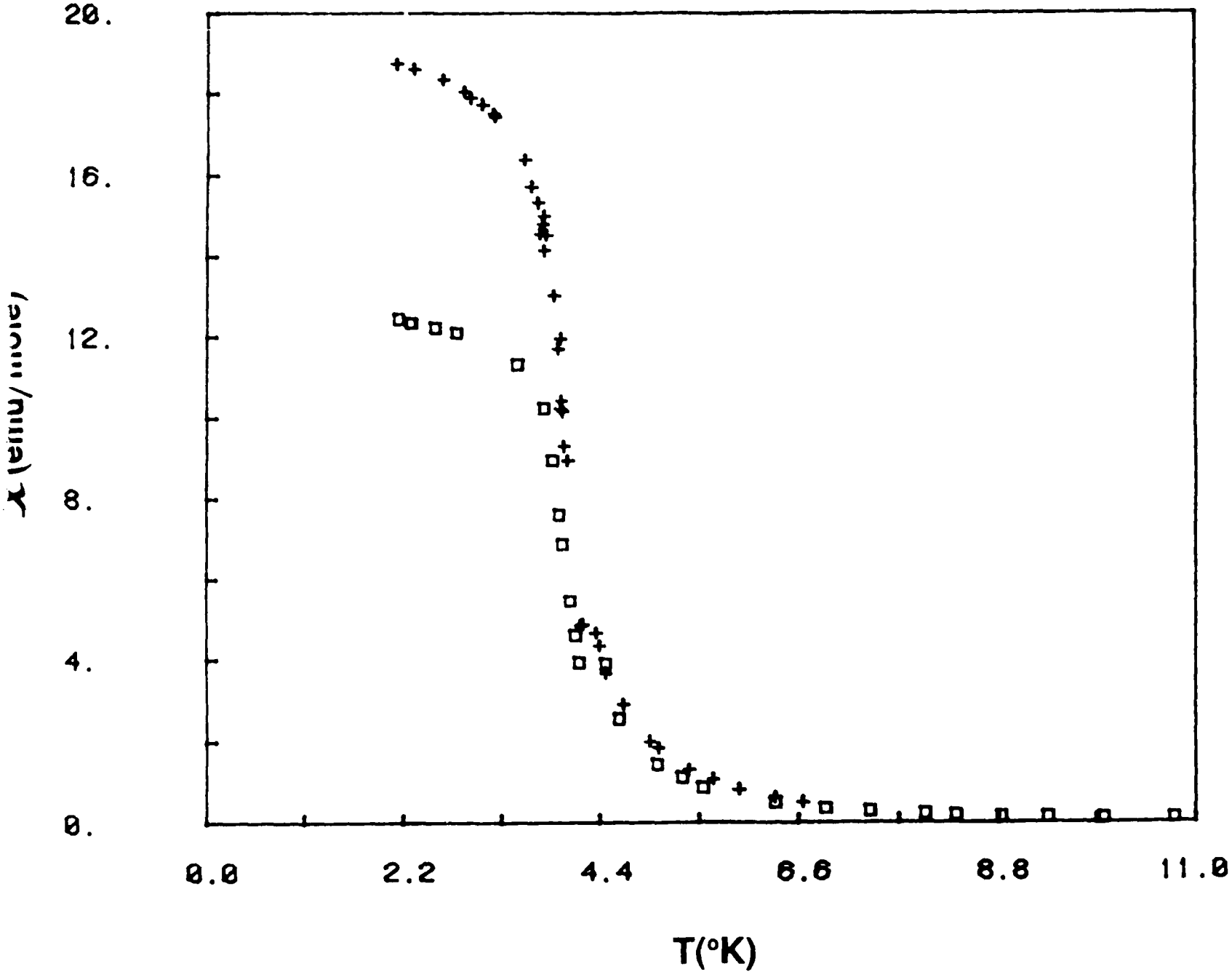
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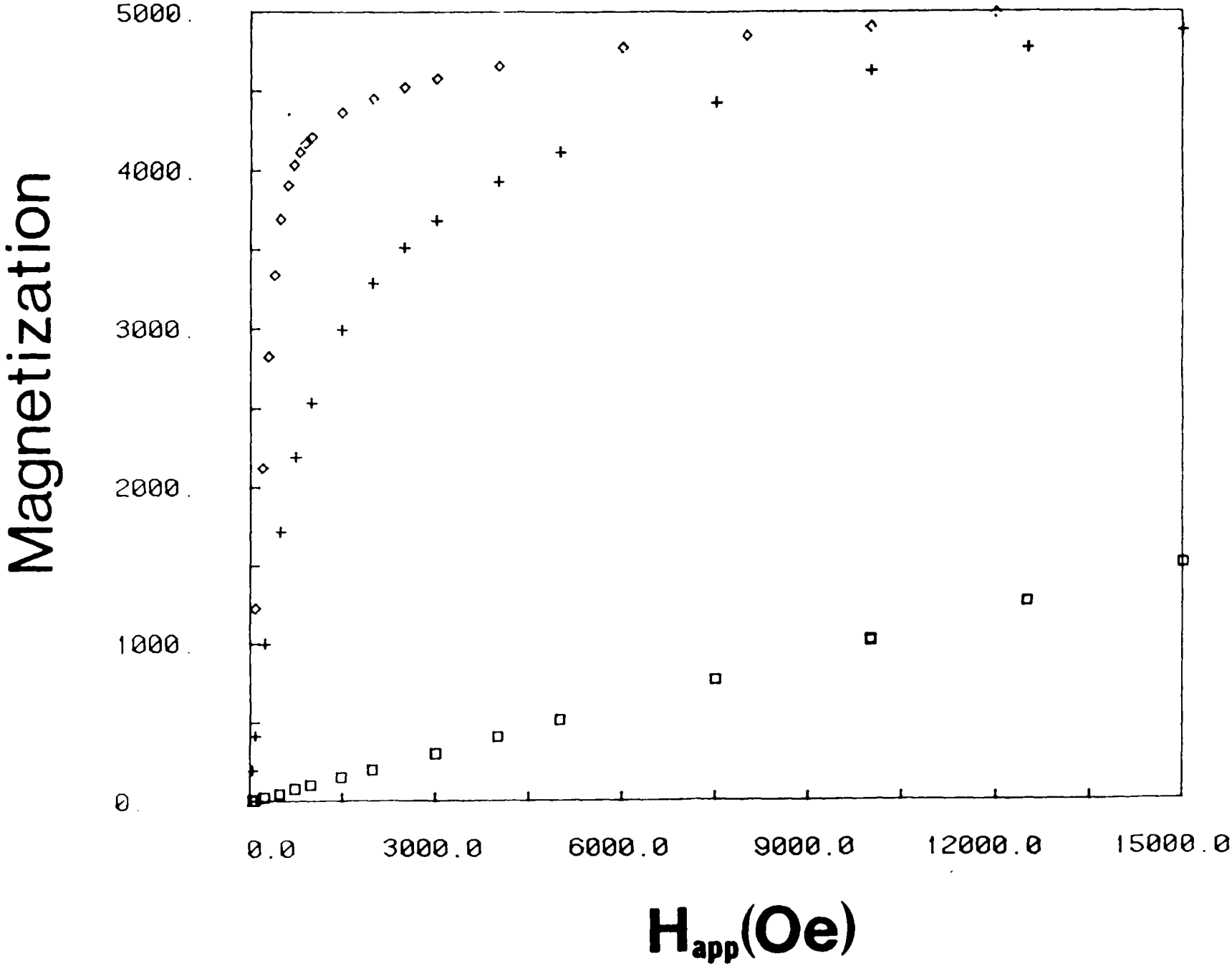
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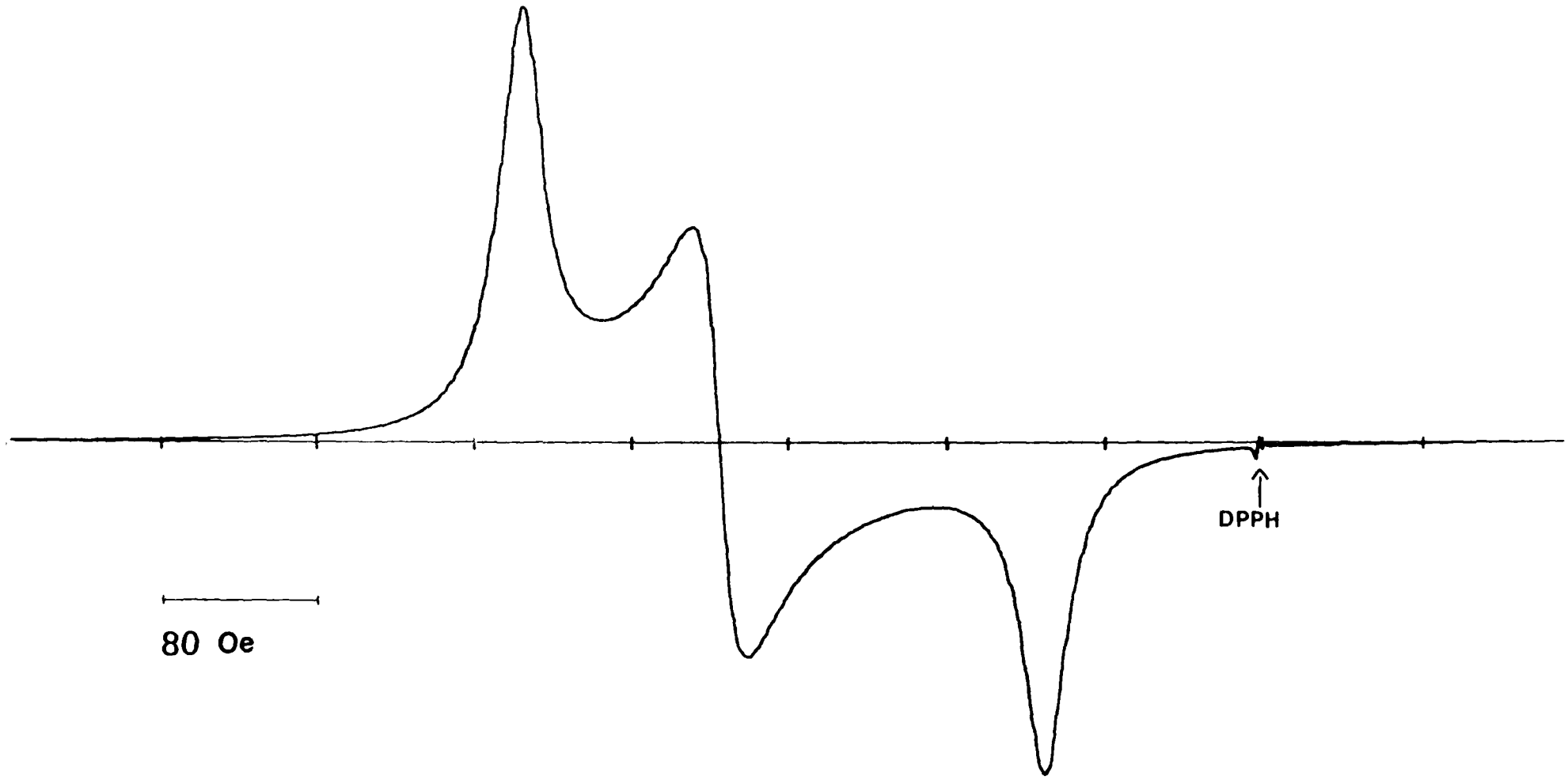
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