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SUBSTITUTED CADMIUM SPINELS(U) BROWN UNIV PROVIDENCE RI
DEPT OF CHEMISTRY M TELLEFSEN ET AL. 25 NOV 83 28
N00014-77-C-0387

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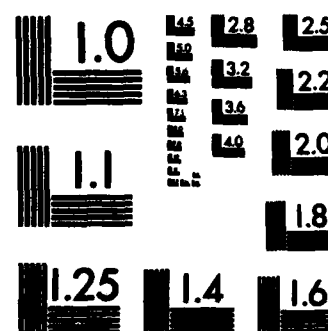
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1. REPORT NUMBER 28	2. GOVT ACCESSION NO. A135297	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) Homogeneity and Magnetic Susceptibility in Some Substituted Cadmium Spinels		5. TYPE OF REPORT & PERIOD COVERED
		6. PERFORMING ORG. REPORT NUMBER 28
7. AUTHOR(s) M. Tellefsen, L. Carreiro, R. Kershaw, K. Dwight, and A. Wold		8. CONTRACT OR GRANT NUMBER(s) N00014-77-C-0387
9. PERFORMING ORGANIZATION NAME AND ADDRESS Professor Aaron Wold Brown University, Department of Chemistry Providence, Rhode Island 02912		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS NR-359-653
11. CONTROLLING OFFICE NAME AND ADDRESS Dr. David Nelson Code 472 Office of Naval Research Arlington, Virginia 22217		12. REPORT DATE November 25, 1983
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)		13. NUMBER OF PAGES 20
		15. SECURITY CLASS. (of this report)
		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE
16. DISTRIBUTION STATEMENT (of this Report) APPROVED FOR PUBLIC RELEASE: DISTRIBUTION UNLIMITED		
17. DISTRIBUTION STATEMENT (of the abstract entered here) (If different from 16)		
18. SUPPLEMENTARY NOTES SUBMITTED TO THE JOURNAL OF SOLID STATE CHEMISTRY		
19. KEY WORDS (Continue on reverse side if necessary and identify by block number) cadmium iron spinels magnetic susceptibility cadmium rhodium spinels		
20. ABSTRACT (Continue on reverse side if necessary and identify by block number) The normal spinels CdFe_2O_4 , $\text{CdGa}_{1.8}\text{Fe}_{0.2}\text{O}_4$ and $\text{CdRh}_{1.8}\text{Fe}_{0.2}\text{O}_4$ were prepared, and their magnetic susceptibilities were measured. The low μ_{eff} value of 4.72(5) μ_B found for CdFe_2O_4 can be attributed to hybridization of the $6s$ and $4g$ wavefunctions resulting from spin-orbit interaction coupled with strong crystal fields having trigonal components at the spinel B-sites. Magnetic susceptibility was sensitive to the homogeneity of samples of $\text{CdGa}_{1.8}\text{Fe}_{0.2}\text{O}_4$ and $\text{CdRh}_{1.8}\text{Fe}_{0.2}\text{O}_4$, which was dependent upon the method of preparation. For		

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Homogeneity and Magnetic Susceptibility in Some Substituted Cadmium Spinel

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ABSTRACT The normal spinels CdFe_2O_4 , $\text{CdGa}_{1.8}\text{Fe}_{0.2}\text{O}_4$ and $\text{CdRh}_{1.8}\text{Fe}_{0.2}\text{O}_4$ were prepared, and their magnetic susceptibilities were measured. The low μ_{eff} value of $4.72(5)\mu_B$ found for CdFe_2O_4 can be attributed to hybridization of the 6S and 4G wavefunctions resulting from spin-orbit interaction coupled with strong crystal fields having trigonal components at the spinel B-sites. Magnetic susceptibility was sensitive to the homogeneity of samples of $\text{CdGa}_{1.8}\text{Fe}_{0.2}\text{O}_4$ and $\text{CdRh}_{1.8}\text{Fe}_{0.2}\text{O}_4$, which was dependent upon the method of preparation. For homogeneous samples, the magnetic susceptibility approaches the theoretical value for high-spin $\text{Fe}^{3+}(3d^5)$. The slight remaining discrepancy from spin-only moment is due to the statistical existence of a few small Fe^{3+} clusters.

Introduction

The spin-only moment observed for the $\text{Fe}^{3+}(3d^5)$ ion in a number of oxide systems (Fe_2O_3 , $\text{Fe}_2\text{O}_3/\text{Rh}_2\text{O}_3$, ZnFe_2O_4) has been reported to be unusually low compared to the theoretical value.¹⁻³ In an earlier study, Selwood¹ indicated that iron-iron interactions between adjacent cations were probably responsible for the low moment observed in $\alpha\text{-Fe}_2\text{O}_3$. Similarly, low moments were observed by Krén² in a study of the solid solution $\text{Fe}_2\text{O}_3\text{-Rh}_2\text{O}_3$. However, this system presents a further problem in that complete homogeneous solid solution is difficult to attain.⁴

Lotgering³ has also reported on the low moment observed for Fe^{3+} in the normal spinel ZnFe_2O_4 , which he attributed to the presence of a small number

of Fe^{3+} ions at tetrahedral sites. However, the reported temperature dependence of the magnetic susceptibility for ZnFe_2O_4 was linear and hence the lowering of the moment could not be attributed to the properties of A-O-B- clusters.

Another possible explanation might be interaction of t_{2g} orbitals on neighboring B-sites resulting in a trigonal distortion of the cubic field, permitting admixture of the ^6S and ^4G states. In order to study the role of such t_{2g} - t_{2g} interactions, the systems $\text{CdGa}_{2-x}\text{Fe}_x\text{O}_4$ and $\text{CdRh}_{2-x}\text{Fe}_x\text{O}_4$ were chosen. The magnetic susceptibility of the normal spinel CdFe_2O_4 has not been reported. However, a lowering of the spin-only moment of Fe^{3+} would be anticipated. In addition, members of the systems $\text{CdGa}_{2-x}\text{Fe}_x\text{O}_4$ and $\text{CdRh}_{2-x}\text{Fe}_x\text{O}_4$ are expected to crystallize with the normal spinel structure, and it may be possible to minimize the trigonal distortion and hence obtain a measured moment per Fe^{3+} approaching the theoretical value.

Experimental Section

Preparation. Polycrystalline samples of the systems $\text{CdRh}_{2-x}\text{Fe}_x\text{O}_4$ and $\text{CdGa}_{2-x}\text{Fe}_x\text{O}_4$ were prepared by the solid state reaction of the binary oxides. Samples of $\text{CdRh}_{1.8}\text{Fe}_{0.2}\text{O}_4$ were also prepared by a precursor method starting from ammonium hexachlororhodate(III) and iron(II)sulfate heptahydrate.⁴

Solid State Reaction of the Oxides. Starting materials consisted of Fe_2O_3 (Mapico Red, Columbian Carbon Co.), Ga_2O_3 (Gallard and Schlesinger, 99.999%), Rh_2O_3 , and CdO . Rhodium(III) oxide was prepared in the high temperature, ambient pressure form (space group Pbca) by heating finely divided rhodium metal (Engelhard Inc. 99.99%) under flowing oxygen at 800°C .⁵ Cadmium oxide was obtained from the decomposition of CdCO_3 at 450°C in air.⁶

Members of the system $\text{CdRh}_{2-x}\text{Fe}_x\text{O}_4$ were prepared by thoroughly grinding together stoichiometric quantities of the oxides (with a 5% by weight excess of CdO) and heating in a silica boat open to air at 800°C . Fast scan x-ray diffraction analysis indicated completion of the reaction by the absence of

Fe_2O_3 or Rh_2O_3 after two 24 hr heating intervals with intermittent grinding. After complete reaction, excess CdO was removed by washing the product with 50 ml hot 1M (aq) NH_4Cl , followed by 50 ml hot distilled water.

It was necessary to pretreat samples of the $\text{CdGa}_{2-x}\text{Fe}_x\text{O}_4$ system by "nitrating" the starting mixtures, because of the poor reactivity of Ga_2O_3 . Mixtures were ground until homogeneous and transferred to a porcelain crucible. Concentrated (16M) nitric acid was added dropwise to sufficiently wet the powder, and the crucible was heated gently until the decomposition of all nitrates was complete. The powder was ground thoroughly and transferred to a silica tube. The tube was sealed with an internal pressure of ~1 atm of air. Samples heated at 800°C required three 24 hr intervals with intermittent nitration and grindings. Samples heated at 900°C or 1000°C required two 24 hr heating intervals. Cadmium oxide is volatile above 825°C and must be heated in sealed tubes above this temperature.⁶

Preparation of $\text{CdRh}_{1.8}\text{Fe}_{0.2}\text{O}_4$ by the Precursor Method. Ammonium hexachlororhodate(III) (Engelhard Inc., 99.999%) and iron(II)sulfate heptahydrate were taken in stoichiometric quantities with respect to metal assay, and ground together. After transferring to a porcelain crucible, concentrated (18M) sulfuric acid was added dropwise, and heating proceeded as in the nitration method above. CdO was added to the resulting black powder, and the mixture was ground thoroughly and heated at 800°C. After complete reaction was achieved, the excess cadmium was removed.

Analysis of the rhodium complex precursor was performed by reduction under flowing 85% argon/15% hydrogen at 500°C employing a thermogravimetric (TGA) balance. Iron(II) sulfate heptahydrate was ignited in air at 800°C to give Fe_2O_3 .

Sample Characterization

X-ray Diffraction. Powder diffraction patterns were obtained with a Norelco diffractometer using monochromatic high-intensity $\text{CuK}\alpha_1$ radiation ($\lambda = 1.5405\text{\AA}$). Fast scans were taken at a rate of $1^\circ 2\theta$ /min in the range $12^\circ < 2\theta < 72^\circ$. Lattice parameters were determined by least-squares analysis of slow scans at $0.25^\circ 2\theta$ /min in the range $26^\circ < 2\theta < 74^\circ$.

Magnetic Measurements. Magnetic susceptibility measurements were performed from 77 to 300 K using a Faraday balance described elsewhere.⁷ Field dependent measurements were made with field strengths between 6.22 and 10.4 kOe, and the balance was calibrated with platinum wire ($\chi_g = 0.991 \times 10^{-6}$ emu/g at 275 K).

Results and Discussion

The normal spinel CdFe_2O_4 (space group $\text{Fd}\bar{3}\text{m}$) is a red powder when prepared at 800°C in air. The observed cell parameter, $a_0 = 8.708(1)\text{\AA}$, agrees with the value reported in the literature.⁸ As shown in Figure 1, the magnetic susceptibility data for CdFe_2O_4 obeys the Curie law with $\mu_{\text{eff}} = 4.72(5)\mu_B$. This value of μ_{eff} is less than the value of $5.50\mu_B$ reported for ZnFe_2O_4 ³ and is significantly lower than the theoretical value of $5.92\mu_B$ for spin-only $S=5/2$.

In fact, the high-spin ^6S configuration is the rigorous ground state only for a free $3d^5$ ion. Its energy is not affected by crystal fields or by spin-orbit interaction, whereas that of the immediately adjacent 9-fold degenerate ^4G manifold is influenced strongly by these factors. Significant hybridization of ^6S and ^4G wavefunctions belonging to the same irreducible

representation can occur if the crystal fields are sufficiently strong and of less than cubic symmetry so as to permit spin-orbit coupling.

Neighboring cations contribute a trigonal component to the crystal field at a B-site in the spinel structure. Hence a lowering of the Fe^{3+} moment by admixture of ^4G into ^6S is possible when the crystal fields are large enough. The observed reduction from 5.92 to $4.72\mu_B$ shows this to be the case for CdFe_2O_4 . The observation of a smaller reduction for ZnFe_2O_4 can be attributed to Zn^{2+} being less covalent than Cd^{2+} , since covalent bonding contributes appreciably to the strength of crystal fields.⁹

The reduction of Fe^{3+} moment by this mechanism requires a sufficiently large trigonal distortion from cubic symmetry. In the case of a dilute solution of CdFe_2O_4 in a diamagnetic host spinel, the distortion at each Fe^{3+} site depends upon the number of adjacent Fe^{3+} neighbors, i.e. upon the size of its cluster. Consequently, the magnetic moment of each Fe^{3+} ion, its contribution to the Curie constant, and hence μ_{eff} will depend upon the cluster size. The distribution of clusters characteristic of a homogeneous solution can be obtained by statistical analysis; inhomogeneous clustering has a pronounced effect upon the magnetic properties of these solid solutions. This effect is illustrated by the magnetic properties of the solid solutions $\text{CdGa}_{2-x}\text{Fe}_x\text{O}_4$ and $\text{CdRh}_{2-x}\text{Fe}_x\text{O}_4$ reported in this study.

The composition $\text{CdGa}_{1.8}\text{Fe}_{0.2}\text{O}_4$ was chosen for study since the effects of both phase homogeneity and the intrinsic magnetic behavior of Fe^{3+} should be observed at this level. Preparations of the mixed composition $\text{CdGa}_{1.8}\text{Fe}_{0.2}\text{O}_4$ were performed at temperatures of 800°, 900°, and 1000°C.

X-ray analysis indicated the formation of a single-phase spinel at each temperature. The color was yellow; unsubstituted CdGa_2O_4 is white. The cell parameter for $\text{CdGa}_{1.8}\text{Fe}_{0.2}\text{O}_4$ remained constant at a value of $8.614(2)\text{\AA}$ for all temperatures of preparation. This value represents an increase from the observed cell parameter for CdGa_2O_4 at $8.604(1)\text{\AA}$, which is consistent with the substitution of a cation of larger ionic radius, Fe^{3+} ($r = 0.645\text{\AA}$), for one of smaller ionic radius, Ga^{3+} ($r = 0.620\text{\AA}$).¹⁰

The room temperature magnetic susceptibility of $\text{CdGa}_{1.8}\text{Fe}_{0.2}\text{O}_4$ is given as a function of the temperature of preparation in Table I. The values reported represent those attained after repeated heatings produced unchanged magnetic susceptibilities. Clearly, there is an increase of the magnetic susceptibility for $\text{CdGa}_{1.8}\text{Fe}_{0.2}\text{O}_4$ with increasing temperature of preparation, which is indicative of an increase in phase homogeneity.

Preparations of the mixed composition $\text{CdRh}_{1.8}\text{Fe}_{0.2}\text{O}_4$ were performed by solid state reaction of the oxides and by a precursor method. X-ray analysis of the products in both cases indicated the formation of a single phase spinel. The measurement of cell parameters was not attempted, since unsubstituted CdRh_2O_4 and CdFe_2O_4 have similar cell parameters at $8.765(1)\text{\AA}$ and $8.708(1)\text{\AA}$, respectively. The magnetic susceptibility values given in Table I indicate that the precursor method for preparing solid solutions achieves maximum homogeneity.

Having established the effects of phase homogeneity on the magnetic susceptibilities of both solid solution systems, a comparison with unsubstituted CdFe_2O_4 is appropriate. Within the limits of the synthetic technique, the samples of $\text{CdGa}_{1.8}\text{Fe}_{0.2}\text{O}_4$ prepared at 1000°C and $\text{CdRh}_{1.8}\text{Fe}_{0.2}\text{O}_4$ prepared by the precursor method are the most homogeneous for the respective compositions. The magnetic susceptibility value of 1.37×10^{-2} emu/mol.eq.Fe observed

for these samples shows a large increase from the value of 0.96×10^{-2} emu/mol.eq.Fe for unsubstituted CdFe_2O_4 . The remaining small deviation from spin-only moment is due to the clustering inherent in a random distribution of 10% iron over the spinel B-sites. Statistical analysis for this composition predicts that 53% of the Fe^{3+} ions will be isolated and that 28% will exist in nearest-neighbor pairs, 11% in clusters of three, and a total of 8% in larger clusters. The Curie constant and magnetic susceptibility are the sums of contributions from each Fe^{3+} ion; the amount contributed per ion will depend upon the cluster size. For example, the measured value of 1.37×10^{-2} emu/mol.eq.Fe could be explained by assigning the spin-only value of 1.50×10^{-2} to isolated and paired Fe^{3+} ions, and the bulk value of 0.96×10^{-2} to clusters of three or more.

Summary and Conclusions

The normal spinels CdFe_2O_4 , $\text{CdGa}_{1.8}\text{Fe}_{0.2}\text{O}_4$, and $\text{CdRh}_{1.8}\text{Fe}_{0.2}\text{O}_4$ were prepared, and their magnetic susceptibilities were measured. Unsubstituted CdFe_2O_4 showed a μ_{eff} of $4.72(5) \mu_B$, which is lower than the value $5.92 \mu_B$ expected for high-spin Fe^{3+} ($3d^5$). The deviation from spin 5/2 behavior is attributed to a hybridization of the 6S and 4G states resulting from spin-orbit interaction coupled with strong crystal fields possessing trigonal components at the spinel B-sites. The magnetic susceptibility of $\text{CdGa}_{1.8}\text{Fe}_{0.2}\text{O}_4$ samples was found to increase with the temperature of preparation, which was attributed to an increase in phase homogeneity. Similarly, samples of $\text{CdRh}_{1.8}\text{Fe}_{0.2}\text{O}_4$ prepared by a precursor method were found to be more homogeneous than those prepared directly from the oxides,

as determined by magnetic susceptibility. For homogeneous samples of both $\text{CdGa}_{1.8}\text{Fe}_{0.2}\text{O}_4$ and $\text{CdRh}_{1.8}\text{Fe}_{0.2}\text{O}_4$, observed magnetic susceptibilities of 0.0137 emu/mol.eq.Fe approach that expected for $\text{Fe}^{3+}(3d^5)$. The small deviation from ideal spin-only moment is caused by the statistical existence of a few remaining Fe^{3+} clusters.

Acknowledgment. Acknowledgment is made to the Office of Naval Research, Arlington, Virginia, for the support of Mark Tellefsen, and to the National Science Foundation (Grant DMR-82-03667) for the support of Louis Carreiro and Kirby Dwight. Acknowledgment is also made to Brown University's Materials Research Laboratory program which is funded through the National Science Foundation.

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TABLE I

Magnetic Properties of $\text{CdGa}_{1.8}\text{Fe}_{0.2}\text{O}_4$ and $\text{CdRh}_{1.8}\text{Fe}_{0.2}\text{O}_4$

<u>Composition</u>	<u>Starting Materials</u>	<u>Temperature of Preparation</u>	<u>χ 293 K ^a</u>
CdFe_2O_4	Oxides	800°C	0.0096(1)
$\text{CdGa}_{1.8}\text{Fe}_{0.2}\text{O}_4$	Oxides	800°C	0.0122(1) ^b
		900°C	0.0131(1)
		1000°C	0.0137(1)
$\text{CdRh}_{1.8}\text{Fe}_{0.2}\text{O}_4$	Oxides	800°C	0.0114(1) ^c
	Precursors		0.0137(1)
$\text{Fe}^{3+}(3d^5)$ ^d	-	-	0.0150

a) units: emu/mol.eq.Fe

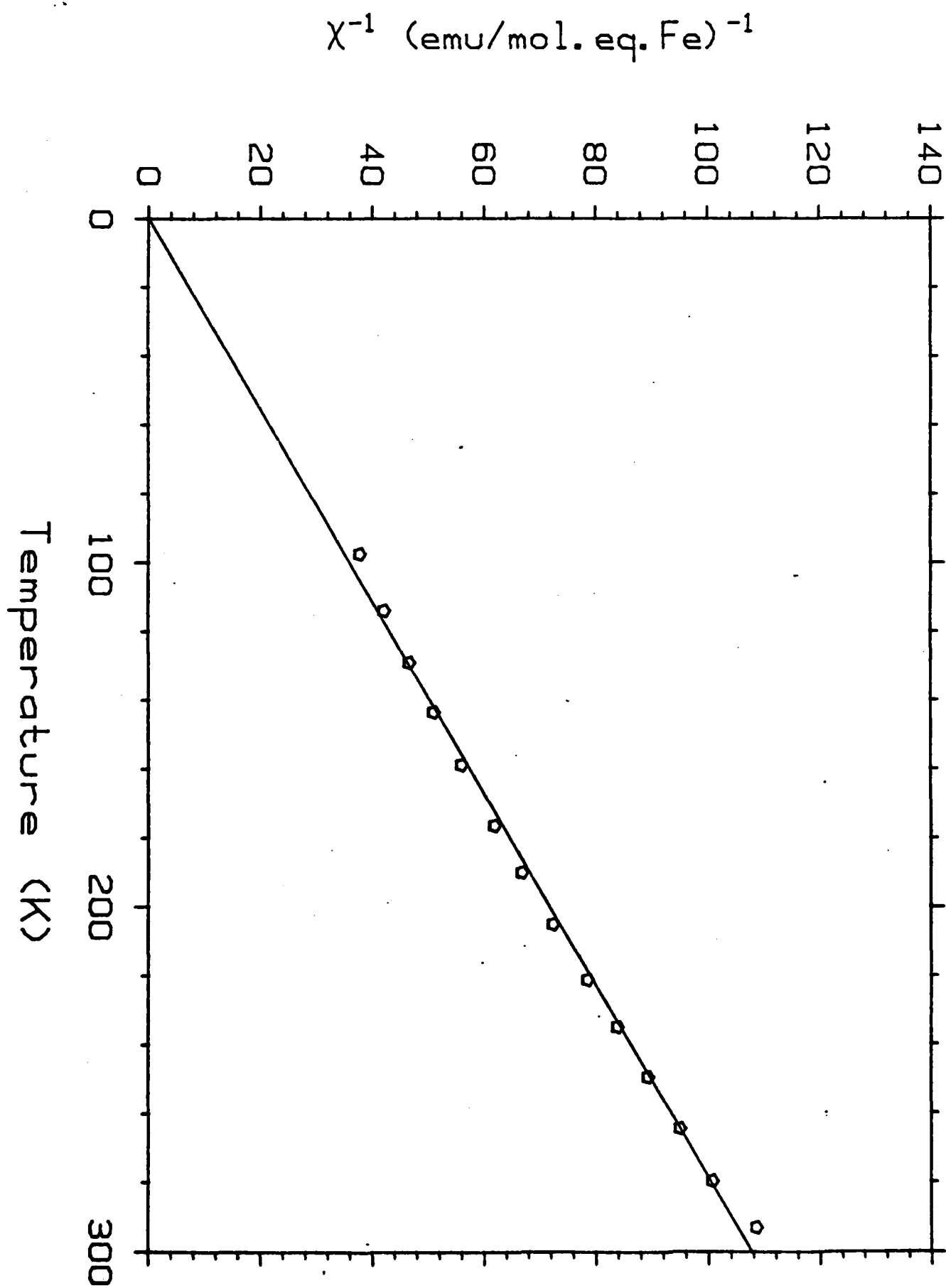
b) corrected for core diamagnetism (11)

c) uncorrected

d) ($\mu_{\text{theo}} = 5.92\mu_B$)

FIGURE CAPTION

Fig. 1 Inverse magnetic susceptibility versus temperature
for CdFe_2O_4 .



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