Inorg. Chem. **2000,** *39,* 6142−6144

Synthesis, Crystal Structure, and Magnetic Properties of a One-Dimensional Polymeric Copper(II) Complex Containing an Unusual 1,1′-Bicoordinated Oxalato Bridge

Oscar Castillo,[†] Antonio Luque,*[,†] Jon Sertucha,[†] Pascual Román,[†] and Francesc Lloret[‡]

Departamento de Química Inorgánica, Universidad del País Vasco, Apartado 644, E-48080 Bilbao, Spain, and Departament de Química Inorgànica, Facultat de Química, Universitat de València, Dr. Moliner 50, E-46100 Burjassot, València, Spain

Received May 23, 2000

Introduction

Polynuclear metal complexes containing a bridging oxalato ligand (hereafter noted ox) have played a key role in the development of new concepts in molecular magnetism during the past two decades.[1−3] The remarkable ability of this old but evergreen ligand to mediate electronic effects between paramagnetic metal ions separated by more than 5 Å is at the origin of this.[4−12] A plethora of oxalato-bridged dinuclear complexes has been well-characterized[4] and intensive magneto-structural studies have analyzed, from both experimental[5] and theoretical[6] viewpoints, the strucutural and electronic factors (orbital topology, coordination polyhedra, donor atoms, nature, and substituents of the periphedral ligands) that govern the value of the magnetic coupling through oxalate. A handful of one-dimensional oxalato-bridged systems $[\mathrm{Cu}(\mu\text{-ox})(\mathrm{L})_x]$ (L = nitrogen-donor ligand or water molecule) have been published showing from mediate[7,8] to weak[9] antiferromagnetic and even ferromagnetic couplings.[10−12] Recently, the oxalate anion has allowed the construction of two-[13] and three-dimensional[14] homo- and hetero- transition metal networks which have applications as molecular-based magnetic materials and they exhibit a great diversity of magnetic behaviors, ferro-, ferri-, or antiferromagnetic long-range ordering being observed.

Despite the above-described richness of the polymeric oxalato complex chemistry, the adoption of the bis(chelating) coordination mode of the oxalato bridge (i.e., bis-bidentate) to form five-membered rings with the metal centers is extensively dominant, and up to date the number of compounds structurally characterized in which one[12,15] or two oxygen atoms of the oxalate are noncoordinated is very scarce. In the last case, as far as we are aware, only salts of the $[\mathrm{M}_2(\mu\text{-1,2-ox})(\mathrm{ox})_4]^{6-}$ dimeric anions ($\mathrm{M} = \mathrm{VO}^{2+}$ and $\mathrm{UO}_2{}^{2+}$)[16] and anionic chains of triphenylstannate(IV) ions[17] have been reported in which the oxalato act as 1,1′-*trans*-bidentate ligand between two metallic centers.

Hence, we report herein on the synthesis, crystal structure and the magnetic behavior of the one-dimensional $[\mathrm{Cu}(\mu\text{-ox})(\mathrm{H}_2\mathrm{O})(4\text{-apy})_2]_n$ (**1**) complex (4-apy = 4-aminopyridine), the first example of a transition metal polymeric structure in which the oxalate dianion bridges the metal ions through one oxygen

---

* Corresponding author: Tel.: +34-946 012 701. FAX: +34-944 648 500. e-mail: qipluara@lg.ehu.es

[†] Universidad del País Vasco.

[‡] Universitat de València.

(1) Khan, O. *Angew. Chem., Int. Ed. Engl.* **1985**, *24*, 834.

(2) Khan, O. *Molecular Magnetism*; VCH: Weinheim, Germany, 1993.

(3) Decurtins, S.; Schamalle, H. W.; Pellaux, R.; Fischer, P.; Hauser, A. *Mol. Cryst. Liq. Cryst.* **1997**, *305*, 227.

(4) Felthouse, T. R.; Laskowski, E. J.; Hendrickson, D. N. *Inorg. Chem.* **1977**, *16*, 1077. (b) Julve, M.; Verdaguer, M.; Kahn, O.; Gleizes, A.; Philoche-Levisalles, O. *Inorg. Chem.* **1983**, *22*, 368. (c) Julve, M.; Faus, J.; Verdaguer, M.; Gleizes, A. *J. Am. Chem. Soc.* **1984**, *106*, 8306. (d) Castro, I.; Faus, J.; Julve, M.; Mollar, M.; Monge, A.; Gutiérrez-Puebla, E. *Inorg. Chim. Acta* **1989**, *161*, 97. (e) Castro, I.; Faus, J.; Julve, M.; Gleizes, A. *J. Chem. Soc., Dalton Trans.* **1991**, 1937. (f) Soto-Tuero, L.; García-Lozano, J.; Escriva-Monto, E.; Beneto-Borja, M.; Dahan, F.; Tuchagues, J.; Legros, J. P. *J. Chem. Soc., Dalton Trans.* **1991**, 2619. (g) Gleizes, A.; Julve, M.; Verdaguer, M.; Real, J. A.; Faus, J.; Solans, X. *J. Chem. Soc., Dalton Trans.* **1992**, 3209. (h) Vicente, R.; Escuer, Ferretjans, J.; Stoeckli-Evans, H.; Solans, X.; Font-Bardía, M. *J. Chem. Soc., Dalton Trans.* **1997**, 167. (i) Castro, I.; Calatayud, L.; Sletten, J.; Lloret, F.; Julve, M. *J. Chem. Soc., Dalton Trans.* **1997**, 811. (j) Smekal, Z.; Thornton, P.; Sindelar, Z.; Klicka, R. *Polyhedron*, **1998**, *17*, 10. (k) Castillo, O.; Muga, I.; Luque, A.; Gutiérrez-Zorrilla, J. M.; Sertucha, J.; Vitoria, P.; Román, P. *Polyhedron* **1999**, *18*, 1237.

(5) Julve, M.; Verdaguer, M.; Kahn, O.; Gleizes, A.; Philoche-Levisalles, O. *Inorg. Chem.* **1984**, *23*, 3808. (b) Glerup, J.; Goodson, P. A.; Hodgson, D. J.; Michelsen, K. *Inorg. Chem.* **1995**, *34*, 6255.

(6) Hay, P. J.; Thibeault, J. C.; Hoffmann, R. *J. Am. Chem. Soc.* **1975**, *97*, 4884. (b) Alvarez, S.; Julve, M.; Verdaguer, M. *Inorg. Chem.* **1990**, *29*, 4500. (c) Escuer, A.; Vicente, R.; Ribas, J.; Jaud, J.; Raynaud, B. *Inorg. Chim. Acta* **1994**, *216*, 139. (d) Román, P.; Guzmán-Miralles, C.; Luque, A.; Beitia, J. I.; Cano, J.; Loret, F.; Julve, M.; Alvarez, S. *Inorg. Chem.* **1996**, *35*, 3741. (e) Cano, J.; Alemany, P.; Alvarez, S.; Verdaguer, M.; Ruiz, E. *Chem. Eur. J.* **1998**, *4*, 476.

(7) Michalowicz, A.; Gired, J. J.; Goulson, J. *Inorg. Chem.* **1979**, *18*, 3004.

(8) Cavalca, L.; Chiesi-Villa, A.; Manfredotti, A.; Mangia, A.; Tomlinson, A. A. G. *J. Chem. Soc., Dalton Trans.* **1972**, 391.

(9) Girerd, J. J.; Khan, O.; Verdaguer, M. *Inorg. Chem.* **1980**, *19*, 274. (b) Kitagawa, S.; Okubo, T.; Kawata, S.; Kondo, M.; Katada, M.; Kobayashi, H. *Inorg. Chem.* **1995**, *34*, 4790.

(10) Fitzgerald, W.; Foley, J.; McSweeney, D.; Ray, N.; Sheahan, D.; Tyagi, S. *J. Chem. Soc., Dalton Trans.* **1982**, 1117. Oshio, H.; Nagashima, U. *Inorg. Chem.* **1992**, *31*, 3295.

(11) Suárez-Varela, J.; Domínguez-Vera, J. M., Colacio, E.; Avila-Rosón, J. C., Hidalgo, M. A.; Martín-Ramos, D. *J. Chem. Soc., Dalton Trans.* **1995**, 2143.

(12) Geiser, U.; Ramakrishna, B. L.; Willett, R. D.; Hulsbergen, F. B.; Reedijk, J. *Inorg. Chem.* **1987**, *26*, 3750.

(13) Decurtins, S.; Schmalle, H. W.; Oswald, H. R.; Linden, A.; Ensling, J.; Gutlich, P.; Hauser, A. *Inorg. Chim. Acta* **1994**, *216*, 65. (b) Reiff, W. M.; Meda, L.; Kirss, R. U. *Mol. Cryst. Liq. Cryst.* **1995**, *273*, 181. (c) Farrell, R. P.; Hambley, T. W.; Lay, P. A. *Inorg. Chem.* **1995**, *34*, 757. (d) Mathoniere, C.; Nuttall, C. J.; Carling, S. G.; Day, P. *Inorg. Chem.* **1996**, *35*, 1201. (e) Carling, S. G.; Mathoniere, C.; Day, P.; Malik, K. M. A.; Coles, S. J.; Hursthouse, M. B. *J. Chem. Soc., Dalton Trans.* **1996**, 1839. (f) Bhattacharjee, A.; Iijima, S.; Mitzutani, F. *J. Magn. Magn. Mater.* **1996**, *235*, 153. (g) Pellaux, R.; Schmalle, H. W.; Huber, R.; Fischer, P.; Hauss, T.; Ouladdiaf, B.; Decurtins, S. *Inorg. Chem.* **1997**, *36*, 2301. (h) Andrés, R.; Gruselle, M.; Malézieux, B.; Verdaguer, M.; Vaissermann, J. *Inorg. Chem.* **1999**, *38*, 4637.

(14) Decurtins, S.; Schmalle, H. W.; Schneuwly, P.; Oswald, H. R. *Inorg. Chem.* **1993**, *32*, 1888. (b) Decurtins, S.; Schmalle, H. W.; Schneuwly, P.; Ensling, J.; Gütlich, P. *J. Am. Chem. Soc.* **1994**, *116*, 9521. (c) Coronado, E., Galán-Mascarós, J. R., Giménez-Saiz, C., Gómez-García, Ruiz-Pérez, C.; Triki, S., *Adv. Mater.* **1996**, *6*, 737. (d) Decurtins, S.; Schmalle, H. W.; Pellaux, R.; Schneuwly, P.; Hauser, A. *Inorg. Chem.* **1996**, *35*, 1451. (e) Decurtins, S.; Schmalle, H. W.; Pellaux, R.; Huber, R.; Fisher, P.; Ouladdiaf, B. *Adv. Mater.* **1998**, *8*, 647. (f) Hernández-Molina, M.; Lloret, F.; Ruiz-Pérez, C.; Julve, M. *Inorg. Chem.* **1998**, *37*, 4131.

(15) Garaj, J. *Chem. Commun.* **1968**, 904. (b) Kansikas, J.; Pajunen, A. *Acta Crystallogr.* **1980**, *B36*, 2423.

(16) Kangjing, J.; Jingling, H.; Jiaxi, L. *J. Struct. Chem.* **1983**, *2*, 269. (b) Legros, J. P.; Jeannin, Y. *Acta Crystallogr.* **1976**, *B32*, 2497. (c) Govindarajan, S.; Patil, K. C.; Poojary, M. D.; Manohar, H. *Inorg. Chim. Acta* **1986**, *120*, 103.

(17) Kruger, G. J.; Breet, E. L. J.; Nan Eldik, R. *Inorg. Chim. Acta* **1976**, *19*, 151. (b) Weng-Ng S.; Kumar-Das, V. G.; Hossain, M. B.; Goerlitz, F.; Van der Helm, D. *J. Organomet. Chem.* **1990**, *390*, 19. (c) Weng-Ng, S.; Kumar-Das, V. G. *J. Organomet. Chem.* **1993**, *456*, 175. (d) Weng-Ng, S.; Kumar-Das, V. G.; Luo, B. S.; Mak, T. C. W. *Z. Kristallogr.* **1994**, *209*, 882.

10.1021/ic000543+ CCC: $19.00

Table 1. Crystallographic Data for $[Cu(\mu\text{-ox})(H_2O)(4\text{-apy})_2]_n$ (**1**)

formula	$C_{12}H_{14}CuN_4O_5$
fw	357.82
space group	$C2$ (No. 5)
a, Å	16.644(2)
b, Å	6.357(1)
c, Å	6.752(1)
β, deg	108.12(1)
V, Å^3	679.0(2)
Z	2
T, K	293(2)
λ, Å	0.71069
ρ_{calcd}, g cm^{-3}	1.750
ρ_{obs}, g cm^{-3}	1.76(1)[a]
(Mo Kα), mm^{-1}	1.64
R^b	0.047
$R_w{}^c$	0.054

[a] Flotation $CCl_4/CHBr_3$. [b] $R = \Sigma(||F_o| - |F_c||)/\Sigma|F_o|$. [c] $R_w = [\Sigma w(||F_o| - |F_c||)^2/\Sigma w|F_o|^2]^{1/2}$.

atom only of its two carboxylate groups while the second one remains free (act as a 1,1′-*cis*-bidentate ligand).

Experimental Section

Materials and Methods. All chemicals were of reagent grade and were used as commercially obtained. Standard literature procedures[18] were used to prepare the starting $K_2[Cu(ox)_2]\cdot 2H_2O$. Elemental analyses (C, H, N) were performed on a Perkin-Elmer 2400 microanalytical analyzer. Metal content was determined by absorption spectrometry. IR spectra were recorded on a Nicolet 740 FT-IR spectrometer as KBr pellets in the spectral region 4000−400 cm^{-1}. Magnetic susceptibility measurements were performed on polycrystalline samples of compound **1** with a Quantum Design SQUID susceptometer covering the temperature range 2−300 K and using an applied magnetic field $H = 1000$ G. The susceptometer was calibrated with $(NH_4)_2Mn(SO_4)_2\cdot 12H_2O$. The experimental susceptibility data were corrected for the diamagnetism estimated from Pascal's Tables[19] [-167×10^{-6} cm^3 mol^{-1}], the temperature-independent paramagnetism [60×10^{-6} cm^3 mol^{-1} per Cu^{II}], and magnetization of the sample holder. A prismatic blue crystal of approximate dimensions 0.20 × 0.10 × 0.06 mm was mounted on an Enraf-Nonius CAD4 automatic four-circle diffractometer and used for data collection. Diffraction data were collected at 293 K with graphite-monocromated Mo Kα radiation ($\lambda = 0.71069$ Å) and using the $\omega - 2\theta$ scan technique. Relevant crystallographic data and structure determination parameters for compound **1** are given in Table 1. Unit cell parameters and orientation matrix were determined by least-squares treatment of the setting angles of 25 reflections in the $5 \leq \theta \leq 14°$ range. Examination of two standard reflections, monitored every 2 h, showed no sign of crystal deterioration. The index ranges of data collection were $0 \leq h \leq 23$, $0 \leq k \leq 8$, $-9 \leq l \leq 9$. Intensity data were corrected for Lorentz-polarization and absorption.[20] The maximum and minimum normalized transmission factors were 0.720 and 0.906. The structures were solved by direct methods[21] and refined (on F) by full-matrix least-squares using the X-RAY76 program package[22] on an Alpha 3000−800S computer. Of the 1075 measured independent reflections in the θ range 1−30°, 985 having $I \geq 2\sigma(I)$ were considered as observed and used in the analysis. Non-hydrogen atoms were refined anisotropically. The hydrogen atoms were located from difference Fourier map and isotropically refined. The final Fourier-difference map showed maximum and minimum height peaks of 1.03 and −0.97 e Å^{-3}, respectively. The largest and mean Δ/σ were 0.15 and 0.02 for 128 refined parameters. The goodness-of-fit was 1.49. The final geometrical calculations and the graphical manipulations, were carried out with the PARST95[23] and PLATON[24] programs. Main interatomic bond distances and angles are listed in Table 2.

Table 2. Selected Bond Lengths (Å) and Angles (deg)[a,b] and Hydrogen Contact for Compound **1**

Cu(II) environment			
Cu1−O1	2.004(3)	Cu1−O1^i	2.004(3)
Cu1−N1	1.983(5)	Cu1−N1^i	1.983(5)
Cu1−O3	2.315(4)		
O1−Cu1−O3	88.0(1)	O1^i-Cu1−O3	88.0(1)
O1−Cu1−N1	90.7(2)	O3−Cu1−N1^i	96.4(1)
O1−Cu1−O1^i	176.1(2)	O1^i-Cu1−N1	89.7(2)
O1−Cu1−N1^i	89.7(2)	N1−Cu1−N1^i	167.2(2)
O3−Cu1−N1	96.4(1)	O1^i-Cu1−N1^i	90.7(2)
Oxalato ligand			
O1−C1	1.272(6)	C1−C1^{ii}	1.558(7)
O2−C1	1.235(7)		
O1−C1−O2	125.6(4)	O2−C1−C1^{ii}	117.0(4)
O1−C1−C1^{ii}	117.4(4)		
Hydrogen-Bonding Contacts			
D−H···A	D···A, Å	H···A, Å	D−H···A, deg
O3−H31···O2^{iii}	2.857(5)	1.85(5)	166(5)
O3−H31···O2^{iv}	2.857(5)	1.85(5)	166(5)
N7−H71···O1^v	2.991(6)	2.08(7)	167(7)
N7−H72···O2^{vi}	2.944(6)	2.04(4)	166(4)

[a] Estimated standard deviations in the last significant digits are given in parentheses. [b] Symmetry codes: (i) $-x, y, -z$; (ii) $-x, y, -z + 1$; (iii) $x, 1 + y, z$; (iv) $-x, 1 + y, -z$; (v) $1/2+x, -1/2 + y, z$;(vi) $1/2 - x, 1/2 + y, 1- z$.

Preparation of $[Cu(\mu\text{-ox})(H_2O)(4\text{-apy})_2]_n$ (1). Deep blue single crystals were prepared by slow diffusion techniques in a H-tube containing an aqueous solution of 4-aminopyridine (0.075 g, 0.8 mmol) in one arm and an aqueous solution of $K_2[Cu(ox)_2]\cdot 2H_2O$ (0.071 g, 0.2 mmol) in the other arm. The crystals appeared after two weeks and they filtered off, washed with cold water, ethanol, and diethyl ether, and dried in air. Anal. Calcd for $C_{12}H_{14}CuN_4O_5$ (**1**): C, 40.28; H, 3.94; N, 15.66; Cu, 17.76. Found: C, 39.94; H, 3.86; N, 15.69; Cu, 17.67%. 60−70% yield (based on copper). Thermogravimetric analysis of **1**, under synthetic air with a heating rate of 5 °C min^{-1}, showed a first weight loss of 5.13% at 160 °C attributable to the release of one water molecule (calculated value 5.03%). Thermal degradation finished above 450 °C with the formation of CuO as final product and a total weight loss of 77.88% (calculated value 77.77%). Main IR features (cm^{-1}, KBr pellet): 3470 m for [ν(O−H)]; 3370 s, 3210 s for [ν(N−H)]; 1655 s for [$\nu_{as}(CO_2)$]; 1630 vs for [ν_{as}(C=N)], 1600 for [ν_{as}(C=C)]; 1520 m, 1460 w, 1440 m for [ν(C−N)]; 1360 w, 1330 m, 1300 s [$\nu_s(CO_2)$]; 1210 s for [δ(C−H)]; 1060 w, 1025 m for [ν_s(CO)]; 835 s, 780 m for [$\delta(CO_2)$].

Results and Discussion

The crystal structure of the title compound consists of polymeric neutral $[Cu(\mu\text{-ox})(H_2O)(4\text{-apy})_2]$ chains in which the metal atoms are bridged sequentally by *cis*-bi-monodentate oxalate anions (Figure 1). The copper(II) ion, placed on a 2-fold axis, is in a distorted square-pyramidal environment. The basal plane is formed by two symmetry-related oxygen atoms of two oxalato ligands [Cu−O: 2.004(3) Å] and the endo nitrogen atoms of two 4-apy ligands [Cu−N: 1.983(5) Å] in a trans arrangement. The 4 + 1 coordination sphere of copper atom is completed by an apical oxygen atom from a water molecule with the Cu−O bond (2.315 Å) lying on a 2-fold axis. The

---

(18) Kirschner, S. In *Inorganic Synthesis*; Rochow, E. G., Ed.; McGraw-Hill Book Co.: New York, 1960; Vol. VI, p 1.

(19) Earnshaw, A., *Introduction to Magnetochemistry*; Academic Press: London, 1968.

(20) Walker, N.; Stuart, D. *Acta Crystallogr.* **1983**, *A39*, 158.

(21) Beurskens, P. T.; Admiraal, G.; Beurskens, G.; Bosman, W. P.; García-Granda, S.; Gould, R. O.; Smith, J. M. M.; Smykalla, C. *The DIRDIF program system*; Technical Report of the Crystallography Laboratory; University of Nijmegen: Nijmegen, The Netherlands, 1992.

(22) Stewart, J. M.; Machin, P. A.; Dickinson, C. W.; Ammon, H. L.; Heck H.; Flack, H. *The X-RAY76 system*; Technical Report TR-446; Computer Science Center, University of Maryland: College Park, MD, 1976.

(23) Nardelli, M. *PARST95*. *J. Appl. Crystallogr.* **1995**, *28*, 659.

(24) Spek, A. L., *Acta Crystallogr.* **1990**, *A46*, C34.

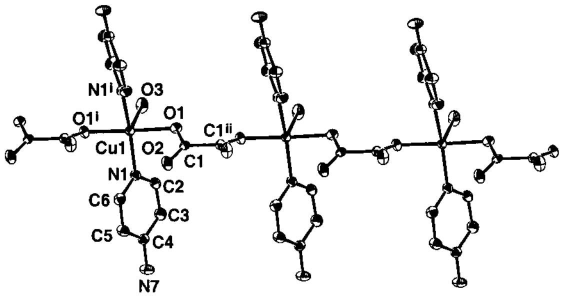

Figure 1. Diagram of the polymeric [Cu(μ-ox)(H_2O)(4-apy)$_2$]$_n$ chain (ellipsoid at 50% probability level).

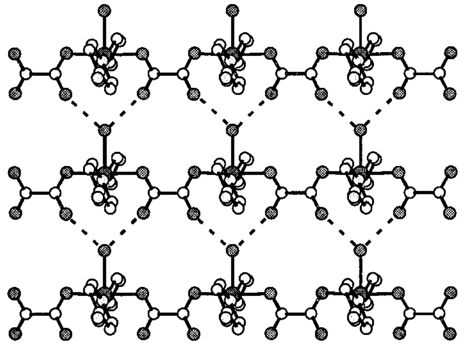

Figure 2. Sheets of chains formed by hydrogen-bonds (dashed lines) involving the coordinated water molecules.

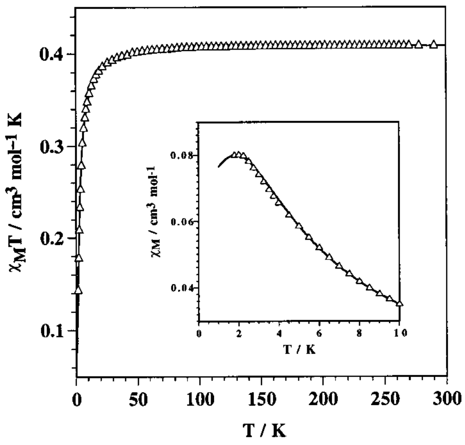

Figure 3. Plots of the thermal dependence of $\chi_M T$ and χ_M for [Cu(μ-ox)(H_2O)(4-apy)$_2$]$_n$: (Δ) experimental data; (–) best fit data (see text).

copper atom is displaced from the mean basal plane toward the apical site by 0.08 Å. The polymeric chains run along the *c* axis of the unit cell, and the basal plane is parallel to the *ac* plane. The mean planes of the aromatic ligand and the oxalate are perpendicular each to other and respect to the basal plane. The intrachain Cu···Cu distance across the oxalato ligand is 6.752(1) Å, which is significantly longer than those published for dimeric and polynuclear complexes with bis-bidentatate oxalato bridges [<6.0 Å],[4,7−11] but within the range [6.5−8.1 Å][16,17] found in polymeric compounds in which the oxalato bridge is bounded to the two metal centers by two oxygen atoms (act as a 1,1′-bidentate ligand). The oxalato ligand, with a two-axis fold passing through the middle of the C−C bond, is not planar and the two $-CO_2$ entities are twisted 28° with respect to each other around the C−C bond. The bond C−O(coordinated) distance [1.272(6) Å], is only slightly longer than the C−O(free) one [1.235(7) Å] owing to the involvement of the free oxygen atoms in an extensive network of hydrogen bonds. Each water molecule forms two hydrogen bonds, related by a 2-fold axis, with two free oxygen atoms from oxalato bridge belonging to a neighboring chain (Figure 2). These O_w−H···O interactions leading to the formation of hydrogen-bonded sheets parallel to (100) plane and placed at two *x* levels ($x = 0$ and $x = 1/2$ levels). The shortest interchain Cu···Cu distance is 6.357(1) Å. The aromatic rings are perpendicular to the sheets and the exo amino groups stablished N−H···O hydrogen bonds with oxygen atoms belonging to two different chains placed in the same *x* level. No face-to-face $\pi-\pi$ interactions between the aromatic rings have been found in the crystal structure.

The polycrystalline X-band spectrum of title compound at room temperature shows an axial-type signal with *g*-tensor values of $g_{\|} = 2.31$, $g_{\perp} = 2.07$ ($\langle g \rangle = 2.16$) which indicates a d($x^2 - y^2$) ground state and it is in accordance with the square-pyramidal geometry of the copper(II) chromophore found in the structural work.

The temperature dependence of the magnetic susceptibility per copper atom of the title compound in the form $\chi_M T$ vs T is shown in Figure 3. At 300 K, $\chi_M T$ is equal 0.41 cm^3 mol^{-1} K. Upon cooling from room temperature, $\chi_M T$ remains nearly constant up to 30 K, whereupon it decrease down to a value of 0.14 cm^3 mol^{-1} K at 2.0 K. An incipient maximum of magnetic susceptibility is observed around 3 K. This magnetic behavior indicates the existence of weak but definitive antiferromagnetic exchange interactions between the copper ions. Since the crystal structure indicates a one-dimensional polymeric structure, the experimental data were least-squares fitted with a numerical expression proposed by Fisher and Bonner[25] for an antiferromagnetic copper(II) uniform chain [the Hamiltonian being $\mathbf{H} = -J\Sigma_i S_i \cdot S_{i+1}$] which leads to $J = -2.2$ cm^{-1}, $g = 2.16$, $\rho = 1.1\%$ and $R = 1.8 \times 10^{-5}$. The percentage of monomeric impurities per mol of copper atom (assumming that the molecular weight of the impurity is the same as that the investigated compound) is given by ρ and R is the agreement factor defined as $\Sigma_i[(\chi_M T)_{obs}(i) - (\chi_M T)_{calc}(i)]^2/\Sigma_i[(\chi_M T)_{obs}(i)]^2$. Attempts to evaluate the possible interchain interactions by means of a J' parameter treated in the molecular field approximation lead to extremely low values without a significant improvement of the fit.

Acknowledgment. This work was supported by the Universidad del País Vasco/Euskal Herriko Unibertsitatea (UPV/EHU) (Project 169.310-EA162/98), the Spanish Dirección General de Investigación Científica y Técnica (DGICYT) (Project 97-1397) and a predoctoral fellowship (UPV/EHU 169.310-035/97).

Supporting Information Available: X-ray crystallographic file in CIF format. This material is available free of charge via the Internet at http://pubs.acs.org.

IC000543+

---

(25) Bonner, J. C.; Fisher, M. E. *Phys. Rev.* **1964**, *A135*, 640.