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A new Zn(II) complex of *N'*-(1-(thiophen-2-yl)ethylidene)isonicotinohydrazide: synthesis, spectral characterization, X-ray structure

Dame Gadiaga

LSAO-MED, Gaston Berger University, Saint-Louis, Senegal
LCCOB Department of Chemistry, University Cheikh Anta DIOP, Dakar, Sénégal

Adama Sy*

LSAO-MED, Gaston Berger University, Saint-Louis, Senegal
LCCOB Department of Chemistry, University Cheikh Anta DIOP, Dakar, Sénégal
e-mail: adama.sy@ugb.edu.sn

Andrea Daolio

Department of Chemical Sciences, Life Science and Environmental Sustainability, University of Parma, Parco Area delle Scienze 17/A, 43124 Parma, Italy

Ibrahima Elhadji Thiam

LCCOB Department of Chemistry, University Cheikh Anta DIOP, Dakar, Sénégal

Paolo Pio Mazzeo

Department of Chemical Sciences, Life Science and Environmental Sustainability, University of Parma, Parco Area delle Scienze 17/A, 43124 Parma, Italy
Biopharmanet-TEC, University of Parma, Parco Area delle Scienze 27/A, Parma 43124, Italy

Alessia Bacchi

Department of Chemical Sciences, Life Science and Environmental Sustainability, University of Parma, Parco Area delle Scienze 17/A, 43124 Parma, Italy
Biopharmanet-TEC, University of Parma, Parco Area delle Scienze 27/A, Parma 43124, Italy

Mohamed Gaye

LCCOB Department of Chemistry, University Cheikh Anta DIOP, Dakar, Sénégal

Abstract

The hydrazone ligand E-*N'*-(1-(thiophen-2-yl)ethylidene)isonicotinohydrazide (HL) was synthesized by a one-step condensation reaction and characterized by elemental analysis, infrared spectroscopy, and ^{1}H and ^{13}C NMR spectroscopies. The Zn(II) complex derived from ligand (HL) was synthesized and characterized by elemental analysis, conductivity measurements in DMF solutions, FT-IR and electronic spectroscopies, and single-crystal X-ray diffraction. Elemental analysis showed that the Zn(II) complex is composed of a single metal, organic ligand, and chloride ions in a 1:1:2 molar ratio. This Zn(II) complex is a neutral electrolyte in solution in DMF. The mononuclear complex **1** crystallizes in the triclinic space group $P\bar{1}$ with the following unit cell parameters: a = 5.831 (3) Å, b = 9.337 (4) Å, c = 13.738 (4) Å, V = 731.6 (5) Å^3, Z = 2. The asymmetric unit of complex **1**

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*Corresponding author

contains one Zn(II) ion, one ligand molecule, and two coordinated chloride ions. In this complex, the ligand exists in its overall neutral bidentate form. The hydrazine group is deprotonated while the pyridine nitrogen atom is protonated. The ligand is coordinated to the Zn(II) through the carbonyl oxygen and the azomethine nitrogen. The metal cation is also coordinated with two chloride ions, resulting in a tetra-coordinated Zn(II). The environment around the cation Zn(II) is best described as a tetrahedral geometry. Numerous hydrogen bonds and an intramolecular chalcogen bond consolidate the structure into a three-dimensional network.

Introduction

Schiff base hydrazones are a class of organic compounds synthesized by the condensation of a primary amine or hydrazide with a ketone or aldehyde [1, 2]. These two precursors can be functionalized to provide multiple coordination sites [3, 4]. The resulting condensation compounds may exhibit donor sites such as O, N, or S, which are very useful for preparing complexes in coordination chemistry [5, 7]. Depending on the number of sites and their relative positions in space, these ligands can generate mononuclear or polynuclear complexes [8, 10]. Depending on the chemical functionalities present on the precursors, these ligands can exist in neutral or deprotonated forms. Consequently, keto-iminol tautomerization can occur in both solution and solid states [11, 14]. Nicotinic hydrazide-derived Schiff bases are widely studied in coordination chemistry for their ability to bind to metal ions via the azomethine nitrogen atom, the carbonyl oxygen atom, and the pyridine nitrogen atom [15, 18]. Combined with acetylthiophene or a keto precursor possessing one or more sulfur atoms, nicotinic hydrazides allow the synthesis of ligands exhibiting both hard and soft sites, thus offering interesting coordination properties [18, 20]. Research articles on transition metal complexes derived from ligands formed from nicotinic hydrazides have described compounds with interesting properties, such as fluorescence [21, 22], magnetism [22, 23], antibacterial [24, 25], catalytic [26, 27], and antitumor [28, 29] properties. In this context, and as part of our study on hydrazone complexes [16, 30, 33], we prepared the hydrazone and characterized the ligand (E)-*N*'-(thiophen-2-ylmethylene)isonicotinohydrazide (HL) and its Zn(II) complex. We also characterized these compounds by spectroscopic analysis, and the structure of the Zn(II) complex was determined by XRD.

Experimental

Materials and instrumentation

Isonicotinic hydrazide, 4-acetylthiophene, zinc chloride heptahydrate were purchased from Sigma-Aldrich and used as received without further purification. All solvents used were of reagent grade. Melting points were determined on a Büchi 570 melting-point apparatus and were uncorrected. Elemental analyses of C, H and N were recorded on a VxRio EL Instrument. Infrared spectra were obtained on an FTIR Spectrum Two of Perkin Elmer spectrometer in the 4000-400 cm^{-1} region. The UV-Visible spectra were recorded on a Perkin Elmer Lambda UV-Vis spectrophotometer. The molar conductance of 1×10^{-3} M in DMF solution of the metal complexes was measured at 25 °C using a WTW LF-330 conductivity meter with a WTW conductivity cell.

Synthesis and characterization of *N*'-(1-(thiophen-2-yl)ethylidene)isonicotinohydrazide (HL)

To a solution of isonicotinic hydrazide (3.0 g, 21.89 mmol) in 30 mL of ethanol was added a solution of 2-acetylthiophene (2.76 g, 21.89 mmol) in 5 mL of ethanol and two drops of glacial acetic acid. The mixture was heated under reflux for three hours. On cooling a yellow precipitate appears. The solid was recovered by filtration and washed thoroughly with cold ethanol, before being dried in a desiccator. Yield: 85 %. Mp. > 260 °C. Anal. calcd. for $C_{12}H_{11}N_3OS$: C, 58.76; H, 4.52; N, 17.13; S, 13.07. Found: C, 58.74; H, 4.50; N, 17.09; S, 13.03 %. FT-IR (KBr; cm^{-1}): 3236 [ν(N-H)], 1661 [ν(C=O) amide]; 1596 [ν(C=N)], 1548 [(ν(C=N) + ν(C=C))], 1236 [ν(C-N)], 1049 [ν(N-N)]. ^{1}H NMR [DMSO; δ (ppm)]: 11.017 (S, 1H, $OH_{iminole}$); 7.10 - 7.78 (mult, 7H, H_{Ar}), 2.52 (S, 3H, $-CH_3$). ^{13}C NMR [DMSO; δ (ppm)]: 162.58 (C-OH); 141.65 (C =N); 122.27 - 154.08 (C_{Ar}); 15.56 ($-CH_3$).

Synthesis of the complex

To a solution of *N′*-(1-(thiophen-2-yl)ethylidene)isonicotinohydrazide (HL) (0.2451 g, 1 mmol) in 10 mL of ethanol was added a solution of $ZnCl_2 \cdot 7H_2O$ (0.2624 g, 1 mmol) in 10 mL of ethanol. The mixture was stirred at room temperature for two hours. The resulting clear solution was filtered, and the filtrate was left for slow evaporation. On standing for five days, yellow prismatic crystals suitable for X-ray single-crystal diffraction analysis were formed and collected.

X-ray data collection, structure determination, and refinement

Single crystals of **1** were grown by slow evaporation of methanol solution of the complex. The crystals were kept at 200(2) K during data collection. Lorentz polarization and absorption corrections were applied for all the experiments. Data reduction was carried out using APEX v4 software [34]. The structures were solved by direct methods using SHELXT [35] and refined by full-matrix least-squares on F^2 with anisotropic displacement parameters for the non-H atoms using SHELXL2016/6 [36] as implemented in Olex2 [37]. Absorption correction was performed on the basis of a multiscan procedure using SADABS. Structural analysis was aided by use of the program PLATON [38]. The hydrogen atoms were calculated in ideal positions with isotropic displacement parameters set to 1.2 Ueq of the attached atom. Molecular graphics were generated using ORTEP-3 [39]. The crystallographic details of compound **1** are summarized in Table 1, and the bond lengths and bond angles are listed in Tables 2 and 3, respectively.

Table 1. Crystal data and structure refinement for compound **1**

Chemical formula	$C_{12}H_{11}Cl_2N_3OSZn$
M_r	381.57
Crystal system	Triclinic
space group	$P\bar{1}$
Temperature (K)	200
***a* (Å)**	5.831 (3)
***b* (Å)**	9.337 (4)
***c* (Å)**	13.738 (4)
α (°)	93.138 (11)
β (°)	101.097 (12)
γ (°)	92.70 (2)
***V* (Å^3)**	731.6 (5)
Z	2
Radiation type	Mo *K*α

μ (mm^{-1})	2.18
Crystal size (mm)	0.4 × 0.3 × 0.2
Absorption correction	Multi-scan
T_{min}, T_{max}	0.659, 0.745
Index ranges	$-7 \le h \le 7$, $-11 \le k \le 11$, $-17 \le l \le 16$
No. of measured reflections	27971
No. of independent reflections	2990
No. of observed [$I > 2\sigma(I)$] reflections	2655
R_{int}	0.059
$R[F^2 > 2\sigma(F^2)]$	0.028
$wR(F^2)$	0.070
Goodness-of-fit on F^2	1.04
No. of reflections	2990
No. of parameters	182
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.40, -0.30

Results and Discussion

General study

The ligand *N*′-(1-(thiophen-2-yl)ethylidene)isonicotinohydrazide (HL) was prepared by a facile condensation of 2-acetylthiophene and isonicotinohydrazide in ethanol. The complex was obtained by adding to solution of the ligand an equimolecular methanolic solution of $ZnCl_2.7H_2O$. The yield was quantitative and the product is soluble in polar organic solvents such as methanol or acetonitrile. The elemental analyses of the ligand molecule and its zinc (II) complex are in accordance with the chemical formulae obtained from spectroscopic characterization and X-ray diffraction study. The FTIR spectrum of the ligand reveals an intense band at 1660 cm^{-1} attributed to the $\nu_{C=O}$ vibrations of the amide group [40, 41]. The band observed at 1596 cm^{-1} is attributed to the $\nu_{C=N}$ vibrations of the imine group [42, 43], and those appearing in the 1548–1407 cm^{-1} region are due to the $\nu_{C=C}$ and $\nu_{C=N}$ vibrations of the aromatic rings [43, 45]. The absorption bands observed in 1236 cm^{-1} region are assigned to the $\nu_{C–N}$ vibrations of the amide group, and the band observed at 1049 cm^{-1} is due to the $\nu_{N–N}$ azomethine vibration. The presence of the C–N bond vibration indicates the existence of the ligand in its amide form in the solid state. This is confirmed by the presence of the $\nu_{N–H}$ vibration at 3236 cm^{-1}. The ^{1}H NMR spectrum of the ligand reveals the presence of signals corresponding to the eleven protons of the ligand. The singlet observed at 2.52 ppm is assigned to the methyl protons. The signals appearing in the 7.10 - 7.78 ppm regions, integrating for seven protons, are attributed to the protons of the aromatic rings. The signal appearing at 11.017 ppm as a singlet is due to the iminol proton [-N=C(OH)], suggesting the iminolisation of the amide group in DMSO solution.

The ^{13}C NMR spectrum of the ligand recorded in DMSO. The observed signal at 144.57 ppm is attributed to the C=N carbon atom of the imine function. The signals due to the aromatic carbon atoms appear in the range 122.27 - 154.08 ppm. The signal at 162.58 ppm is attributed to carbon atom of the iminol moiety [-N=C(OH)] [46]. This observation corroborates the transformation of the amide group in iminol group in solution as observed in the ^{1}H NMR spectrum. Comparison of the FTIR data of the ligand and those of the complex obtained upon coordination with Zn(II) ion shows that the ligand reacts in its amide form during the complexation. In the FTIR spectrum, the bands due to $\nu_{C=O}$ and $\nu_{C=N}$, shift to lower frequencies, appearing at 1634 cm^{-1} and 1572 cm^{-1}, respectively. These facts are indicative of the involvement of the carbonyl oxygen atom and the azomethine nitrogen atom to the coordination of the Zn(II) ion. Molar conductivity measurements of the complex taken from a freshly prepared millimolar DMF (24 $S.cm^2.mol^{-1}$) solution and after two weeks of storage (25 $S.cm^2.mol^{-1}$) indicate that the complex is a neutral electrolyte according to Geary [47]. The small variation in values allows us to conclude that the complex is stable in DMF solutions. The band at 225 - 231 nm corresponds to the $\pi \rightarrow \pi^*$ transition of the aromatic rings, while the absorptions at 289 - 298 nm and 310 - 334 nm were assigned, respectively, to the $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ transitions of the azomethine group [48, 49]. The electronic absorption band of 362 nm was attributed to $n \rightarrow \pi^*$ internal ligand metal charge transfer (LMCT) of zinc complex [50].

X-ray structure of the complex

Compound **1** crystallizes in the triclinic system in the space group $P\bar{1}$. The asymmetric unit contains one neutral ligand, one Zn(II) ion and two coordinated chloride ions resulting in the formulation of $[Zn(HL)(Cl)_2]$ (Figure 1) as a mononuclear complex. Zn1 is coordinated by two chloride ions, one azomethine nitrogen atom and one carbonyl oxygen atom from the ligand and forming a five membered ring ZnOCNN. The geometry around Zn1, which is tetracoordinate, is determined by the distortion index or tetragonality parameter τ4 = (360° - α - β)/141° where α and β are the two largest angles around the latter [51] . The expected values for τ4 are zero for a perfect square plane and one for a perfect tetrahedron. The value of τ4, which is 0.8902, suggests a distorted tetrahedral geometry. In fact, the values of the angles subtended by the atoms coordinated to Zn1 deviates significantly from the ideal values of 109°28' for a perfect tetrahedron: 115.43 (3)° [Cl1 - Zn1 - Cl2], 107.01 (6)° [Cl1 - Zn1 - O1], 117.63 (6)° [Cl2 - Zn1 - O1], 80.571 (7)° [N1 - Zn1 - O1], 114.32 (6)° [Cl1 - Zn1 - N1] and 116.85 (6)° [N1 - Zn1 - Cl2] (Table 2). The dihedral angle between the pyridyl and the thiophenyl rings of the ligand is 3.07°, likely aided in its planarity by the intramolecular chalcogen bond between N2 and S1. Notably, this moiety and interaction is present in many classes of compounds renowned for their antibacterial activity, hinting at a possible application for this complex. These two rings form dihedral angles with five membered ring ZnOCNN of 6.29° and 9.15°, respectively. The bond lengths of Zn1 - Cl1 = 2.2551 (9) Å and Zn1 - Cl2 = 2.1961 (9) Å are comparable to the bond length values reported for complex Zn(HL)2Cl2 (HL = 1-((2-ethyl-4-methyl-1H-imidazol-1-yl)methyl)-1H-benzotriazole) [52] in which Zn - Cl bond lengths are 2.2468(14) Å and 2.2799 (14) Å. The distance Zn1 - N1(imine) [2.0405 (19) Å] is in good agreement with corresponding bond lengths in tetrahedral zinc complexes [53, 54]. The Zn1 - O1 (carbonyl) distance [1.9916 (16) Å] is shorter than the value reported for the similar complex [Zn(HL)Cl2] (HL = N, N'-cyclohexane-1,2-diylidene-bis(4-fluorobenzoylhydrazide)) in which the Zn - O(carbonyl) bond length is 2.030 (3) Å. The structure of the complex is stabilized by numerous intermolecular hydrogen bonds. The molecules are connected by intermolecular N - H···Cl hydrogen bonds involving protonated pyridine nitrogen atom as donor and chloride anions as acceptor [N3 - H3···Cl1^{i}, i = x-1, y−1, z] and [N3 - H3···Cl1^{ii}, ii = −x, −y+1, −z+2]. Only one intermolecular hydrogen bond involving a sulfur atom as acceptor and C - H as donor [C9 - H9···S1] is present. Numerous hydrogen bonds involving C - H as donor reinforce the structure: C10 - H10···Cl1^{i} [i = x−1, y−1, z], C11 - H11···Cl1^{ii} [ii = −x, −y+1, −z+2], C9 - H9···Cl2^{iii} and C10 - H10···Cl2^{iii} [iii = x, y−1, z]. The combined hydrogen bond network gives rise to a three-dimensional architecture viewed along the b axis (Figure 2, Table 3).

Table 2. Selected geometric parameters (Å, °)

Zn1 - Cl1	2.2551 (9)	Zn1 - O1	1.9916 (16)
Zn1 - Cl2	2.1961 (9)	Zn1 - N1	2.0405 (19)
Cl2 - Zn1 - Cl1	115.43 (3)	O1 - Zn1 - N1	80.57 (7)
O1 - Zn1 - Cl1	107.01 (6)	N1 - Zn1 - Cl1	114.32 (6)
O1 - Zn1 - Cl2	117.63 (6)	N1 - Zn1 - Cl2	116.85 (6)
Zn1 - O1 - C7 - N2	−2.5 (3)	Zn1 - N1 - C5 - C4	174.29 (15)
Zn1 - O1 - C7 - C8	177.12 (14)	Zn1 - N1 - C5 - C6	-4.7 (3)

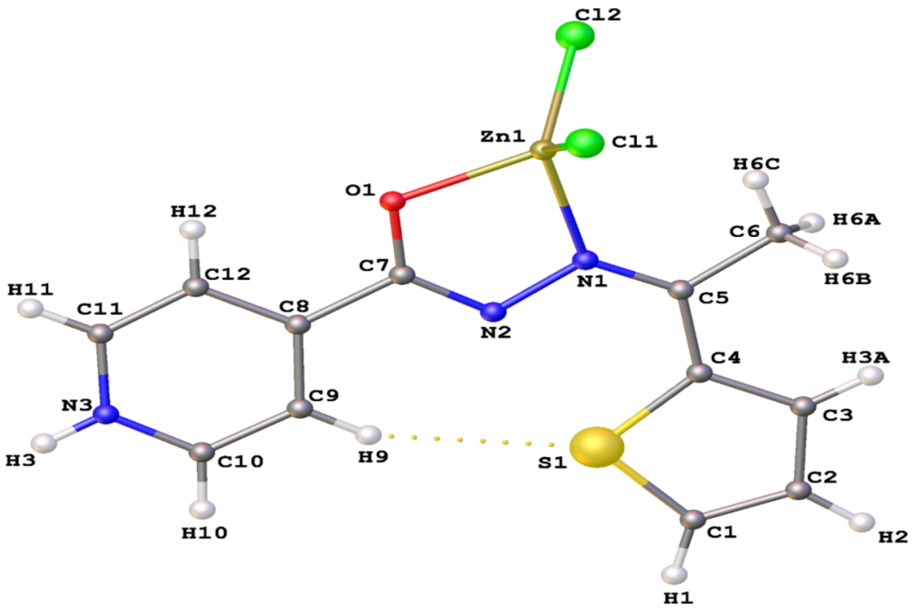

Figure 1. Asymmetric unit of the single crystal structure of complex 1.

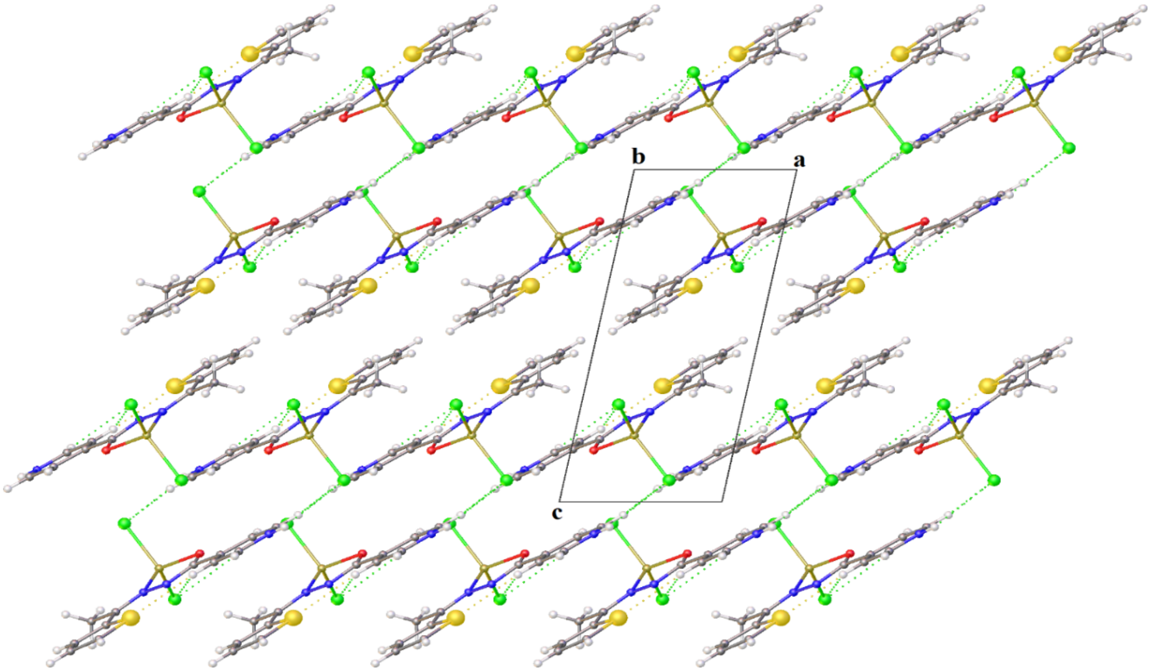

Figure 2. Plot along the b-axis showing the intramolecular hydrogens bonds in the crystal.

Table 3. Hydrogen-bond geometry (Å, °). (i) x−1, y−1, z; (ii) −x, −y+1, −z+2; (iii) x, y−1, z.

D - H···*A*	*D* - H	H···*A*	*D*···*A*	*D* - H···*A*
N3 - H3···Cl1^{i}	0.88	2.57	3.276 (2)	138.3
N3 - H3···Cl1^{ii}	0.88	2.87	3.476 (2)	127.2
C9 - H9···S1	0.95	2.85	3.740 (3)	157.3
C9 - H9···Cl2^{iii}	0.95	2.83	3.495 (3)	127.9
C10 - H10···Cl1^{i}	0.95	2.97	3.488 (2)	115.3
C10 - H10···Cl2^{iii}	0.95	2.93	3.546 (3)	123.5
C11 - H11···Cl1^{ii}	0.95	2.82	3.463 (2)	125.7

Symmetry codes: (i) *x*−1, *y*−1, *z*; (ii) −*x*, −*y*+1, −*z*+2; (iii) *x*, *y*−1, *z*.

Conclusion

In this study, we successfully synthesized and characterized a new hydrazone ligand *N*'-(1-(thiophen-2-yl) ethylidene)isonicotinohydrazide (HL) and its corresponding Zn(II) complex. The ligand was characterized using elemental analysis, FT-IR and NMR spectroscopy, revealing its ability to exist in both amide and iminol forms depending on the medium. The Zn(II) complex was characterized by spectroscopic techniques (FT-IR, UV), conductivity measurement and Single-Crystal X-ray diffraction. Structural analysis revealed that the ligand coordinates to the Zn(II) ion in its neutral bidentate form through carbonyl oxygen and azomethine nitrogen atoms, forming a five-membered chelate ring. Two chloride ions complete the coordination sphere. The environment around the metal center adopts a distorted tetrahedral geometry. Notably, the hydrazine group is deprotonated while the pyridine nitrogen atom is protonated in the solid state. Conductivity measurements demonstrate that the complex behaves as a neutral electrolyte in DMF solution, confirming its stability in polar organic solvents. The crystal structure is stabilized by an extensive network of chalcogen and hydrogen bonds, including N—H···Cl and C—H···Cl interactions, resulting in a three-dimensional supramolecular architecture.

Supplementary Materials

CCDC-2535574 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via https://www.ccdc.cam.ac.uk/structures/, or by e-mailing data_request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44(0)1223-336033.

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