

Syntheses, Characterization, and X-ray Crystal Structure of Heteronuclear Zn/Na Assembled with Salen-type Schiff Base

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Abstract: Herein we reported the crystal structure of a dinuclear complex $[\{Zn^{II}Na^I(L)(EtOH)(NO_3)\}]$ (2) obtained by reaction with the metalloligand $[Zn(L)] \cdot (H_2O)$ (1) and sodium nitrate. The metalloligand (1) and the dinuclear (2) complexes are characterized by IR and physical measurement. Spectroscopic evidence indicated that the Schiff base H_2L behave an $N_2O_2O_2$ coordination tetradentate ligand. Suitable crystals of the compound 2 were grown by slow evaporation of ethanol solution for two weeks. The structure of 2 was elucidated by X-ray diffraction analysis. The compound crystallizes in the monoclinic crystal system with a space group of $P2_1/c$. The asymmetric unit of the compound contains one dideprotonated ligand molecule, on tetracoordinated zinc (II) ion, one heptacoordinated sodium(I) ion, one bidentate nitrate group and one coordinated ethanol molecule resulting in a structure formulated as $[Zn(\mu-L)Na(NO_3)(EtOH)]$. In the structure the Zn^{II} ion is tetracoordinated and the environment around the zinc (II) is best described as a planar square geometry. The Na^I ion is seven-coordinated in a severely distorted bipyramid pentagon environment. The interplanar angles between the two phenyl rings with a methoxy substituent is $4.208(4)^\circ$, while the dihedral angle values of these groups with the phenyl carrying amino groups are respectively $7.499(5)^\circ$ and $11.594(5)^\circ$. The Zn–Na distance is $3.4244(9) \text{ \AA}$. In the crystal structure numerous intramolecular and intermolecular hydrogen bonds link the molecules in a three-dimensional network.

Keywords: Zinc, Metalloligand, o-vanillin, 1, 2-diaminobenzene

1. Introduction

Chemists are increasingly interested in the design and synthesis of heteropolynuclear complexes of d and s-block elements owing to original structures with interesting properties in various fields such as magnetism [1, 2], catalysis [3, 4] and luminescence [5]. The major procedure used for synthesizing these kinds of complexes consist to prepare a metal-ligand with Schiff base containing two inners of different sizes [6, 7]. In the metal-ligand, the 3d element occupy the inner with the smaller size [8, 9]. The reactions of

these metal-ligand complexes with metallic centers of different natures can lead polynuclear compounds with very varied structures. Organic ligands capable of maintaining the M^{n+} ($n = 2$ or 3) ion from d-block and the $M'^{n'+}$ ion ($n' = 1$ or 2) from s-block by bridging, are widely used to establish M -(di- μ -phenoxo) $_2$ - M' bridges in order to improve exchanges between metal centers [10–12]. The coordination chemistry of s-block elements has recently developed due to the presence of alkaline elements in biomolecular recognition [13, 14]. One of the most studied ions is Na^+ and some structures containing this element of the block s and an element of the block d show that the element s is located in

the larger site while the transition ion is in the smaller site [6, 7, 15, 16]. The Schiff base bis (2-hydroxy-3-methoxybenzylidene)-1, 2-diaminobenzene, is widely used for synthesizing several 3d/Na heteronuclear complexes whose structures show that the element of s-block is still located in the larger open site, while the d-block element is situated in the smaller closed site [16, 17]. Organic molecules with phenolic oxygen atoms are perspective that the work we report focused on the use of the H₂L Schiff base (H₂L = *N*,

N'-Phenylene-bis-(3-methoxysalicylideneimine)) which has two innerers capable to encapsulate two ions of different natures. It is in this context that we report the synthesis of a complex Zn²⁺/Na⁺ formulated as follows [Zn(μ-L)Na(NO₃)(EtOH)]. The compound is obtained by initially isolating the mononuclear zinc (II) complex before reacting the latter with sodium thiocyanate to obtain a heterotrinnuclear complex (Figure 1). The crystal structure and the physico-chemical are reported herein.

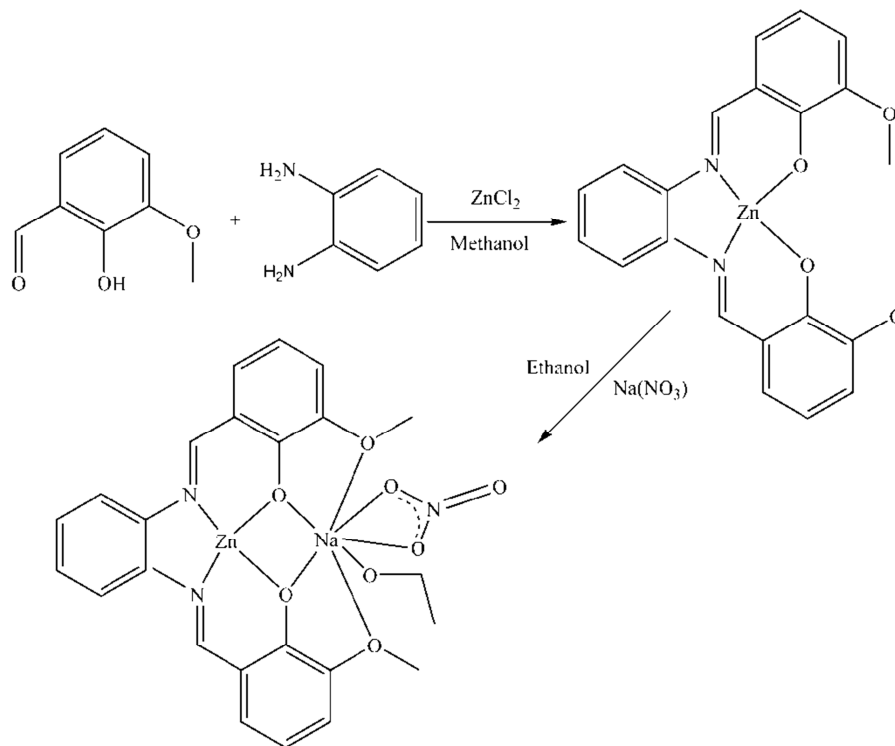


Figure 1. Chemical diagrams for [Zn(L)]·(H₂O) (1) and [{Zn^{II}Na^I(L)(EtOH)(NO₃)}] (2).

2. Materials and Methods

2.1. General Information

o-vanillin and 1, 3-diaminobenzene, zinc chloride tetrahydrate, and sodium nitrate were purchased from Sigma-Aldrich and used as received without further purification. All solvents used were of reagent grade. The ligand (H₂L) was synthesized following the reported procedure [18]. 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 molar conductance of 1×10⁻³ M in DMSO solutions of the metal complexes was measured at 25°C using a WTW LF-330 conductivity meter with a WTW conductivity cell.

2.2. Synthesis and Characterization of (1) and (2)

In a round bottomed flask, the ligand (H₂L) (1 mmol, 0.374 g) was dissolved in 5 mL of acetonitrile. A solution of ZnCl₂ (1 mmol, 0.1363 g) in 5 mL of methanol was added. After two hours under reflux, the yellowish precipitate was

recovered by filtration, washed with ether (2 x 10 mL) and dried in air. The compound was formulated as [Zn(L)]·(H₂O). Yield: 85 %. Elemental Anal. Found (Calcd.) (%): C, 57.72 (57.75); H, 4.40 (4.36); N, 6.12 (6.10). IR (ν, cm⁻¹): [ν(O–H) 3340, δ(OH₂) 851]; 1625 ν(C=N), 1592; ν(C–OPh), 1233; ν(C–OMe), 1201. Λ (S.cm².mol⁻¹): 10.

To a solution of [Zn(L)]·H₂O (1) (1 mmol, 0.4578 g) in 5 mL of DMF was added a solution of NaNO₃ (3 mmol, 0.255 g) in 5 mL of ethanol. The mixture was refluxed for two hours. On cooling yellow solution was filtered. The filtrate was left for slow evaporation. On standing for two weeks, yellow crystals of (2) suitable for X-ray diffraction were isolated. Yield: 78.9 %. Elemental Anal. Found (Calcd.) (%): C, 50.50 (50.48); H, 4.24 (4.22); N, 7.36 (7.38). IR (ν, cm⁻¹): 3429, 3190, 3090, 1617 ν(C=N); 1586; 1533; 1485; 1460; 1241 ν(C–OPh); ν(C–OMe) 1197; [1488 ν₁(NO₃); 1313 ν₃(NO₃); 1041 ν₂(NO₃); Δν 175 cm⁻¹]. Conductance Λ (S.cm².mol⁻¹): 5.

2.3. Crystal Structure Determination

Crystals suitable for single-crystal X-ray diffraction, of the reported compounds, were grown by slow evaporation of MeOH solution of the complexes. Details of the crystal

structure solution and refinement are given in Table 1. Diffraction data were collected using an ENRAF NONIUS Kappa CCD diffractometer with graphite monochromatized MoK α radiation ($\lambda = 0.71073$ Å). All data were corrected for Lorentz and polarization effects. No absorption correction was applied. Complex scattering factors were taken from the program package SHELXTL [19]. The structures were solved by direct methods, which revealed the position of all non-hydrogen atoms. All the structures were refined on F^2 by a full-matrix least-squares procedure using anisotropic displacement parameters for all non-hydrogen atoms [20]. The hydrogen atoms of water molecules and NH groups were located in the Fourier difference maps and refined. Others H atoms (CH and CH₃ groups) were geometrically optimized and refined as riding model by AFIX instructions. Molecular graphics were generated using ORTEP-3 [21]

3. Results and Discussion

3.1. General Study

The ligand H₂L 2-(-(2-(2-hydroxy-3-methoxybenzylideneamino) phenylimino) methyl)-6-methoxyphenol was prepared by mixing under reflux o-vanillin and 1, 2-diamino benzene in molar ratio 2:1 in ethanol, following a reported method [18]. The H₂L react with ZnCl₂ in methanol to yield [Zn(L)]·H₂O (1). This metalloligand react with sodium nitrate in molar ratio 1:3 and the complex formulated as [$\{\text{Zn}^{\text{II}}\text{Na}^{\text{I}}(\text{L})(\text{NO}_3)\}$] (2) was afforded after slow evaporation of the filtrate. The IR spectrum of the metalloligand (1) shows an absorption band at 1625 cm⁻¹ (1632 cm⁻¹ for the free H₂L) which is indicative of a coordinated azomethine (C=N). Additional band $\nu(\text{C}-\text{OPh})$ is pointed at 1233 cm⁻¹ correspond to the deprotonated phenoxy group. The corresponding band in the free ligand appears at 1254 cm⁻¹ band. For the complex (2) the $\nu(\text{C}=\text{N})$ band is pointed at 1617 cm⁻¹ and $\nu(\text{C}-\text{OPh})$ is shifted to high frequency and pointed at 1241 cm⁻¹. The bands at 1488 (ν_1), 1313 (ν_5), 1041 (ν_2) and $\nu_1 - \nu_5 = 175$ cm⁻¹ are indicative of the presence of a bidentate chelating nitrate group. The molar conductance Λ measured in DMF solution are 5 and 7 S cm² mol⁻¹ respectively for (1) and (2) which are indicative of a neutral electrolytes [22].

3.2. Crystal Structure

A novel hetero neutral dinuclear ($\text{Zn}^{\text{II}}\text{Na}^{\text{I}}$) compound has been obtained by step-by-step method and its structure is depicted. The compound crystallizes in the monoclinic crystal system with a space group of $P2_1/c$. The asymmetric unit of the structure of $[\text{Zn}(\mu\text{-L})\text{Na}(\text{NO}_3)(\text{EtOH})]$ contains one dideprotonated L^{2-} ligand molecule $\text{H}_2\text{L} = \text{N}$, N' -Phenylene-bis-(3-methoxysalicylideneimine)), one Zn^{II} cation, one Na^{I} cation, one bidentate nitrate anion and one coordinated ethanol molecule. The molecular structure of the title compound is shown in Figure 2, while selected bond lengths and angles of the coordination environment of the metal centers are listed in Table 2. In the structure of the

dinuclear complex, the Zn atom and the Na atom are bridged by two phenoxo oxygen atoms. The four-coordinated Zn^{II} ion, which occupy the smaller inner of the ligand, is coordinated in a planar square geometry with the basal plane formed by two nitrogen atoms and two phenoxo oxygen atoms. The *cisoid* angles around the zinc atom which are in the range $[84.77(7)^\circ - 95.06(6)^\circ]$, significantly deviates from the ideal values of 90° . The diagonal basal angles are respectively $\text{N1}-\text{Zn1}-\text{O2} = 177.91(7)^\circ$ and $\text{N2}-\text{Zn1}-\text{O1} = 178.73(6)^\circ$ slightly deviates from the ideal values of 180° . These values are comparable to those reported for similar complexes [23]. The zinc atom lies nearly in the plane defined by the N_2O_2 site, the out-of-plane distance being 0.0211 (1) Å. The two Zn–N bond distance values are 1.9404 (15) Å [$\text{Zn1}-\text{N1}$] and 1.9278 (16) Å [$\text{Zn1}-\text{N2}$], and the Zn–O bond distances which lie in the range of 1.8874 (14) Å–1.8937 (13) Å, are in accordance with the reported values for the complex $[\text{Zn}(\mu\text{-L})\text{La}(\text{NO}_3)_3(\text{H}_2\text{O})_2]$ [24]. The Na^{I} ion, which occupy the larger open site O_2O_2 of the ligand, is ligated to two phenoxo oxygen atoms and two methoxy oxygen atoms of the ligand, two oxygen atoms of one bidentate nitrate group and one oxygen atom from a molecule of ethanol. The $\text{Na}-\text{O}_{\text{phenoxo}}$ bond distances are in the range of 2.3953 (16) Å–2.4253 (16) Å (Figure 2, Table 2) and are shorter than the $\text{Na}-\text{O}_{\text{methoxy}}$ 2.6085 (18) Å–2.6350 (18) Å. The distances are comparable to those found in the similar complex $[\text{Cu}(\mu\text{-L})\text{Na}(\text{ClO}_4)(\text{DMF})]$ [12]. The distances of the Na^{I} cation to the two oxygen atoms from the bidentate nitrate group [$\text{Na1}-\text{O5}$ 2.490 (2) Å; $\text{Na1}-\text{O6}$ 2.514 (2) Å] and the oxygen atom of the ethanol molecule [$\text{Na1}-\text{O8}$ 2.364 (2) Å] are in accordance with those found for the complex $[\{\text{Fe}^{\text{II}}\text{Na}^{\text{I}}(\text{L})(\text{EtOH})(\text{HN}(\text{CN})_2)\}(\mu\text{-O})\{\text{Fe}^{\text{III}}\text{Na}^{\text{I}}(\text{L})(\text{NO}_3)\}]_n$ [25]. The sodium cation is situated in the planar cavity defined by the two phenoxo oxygen atoms and the two methoxy oxygen atoms with maximum displacement of 0.131 (1) Å. The interplanar angles between the two phenyl groups with a methoxy substituent is $4.208(4)^\circ$, while the dihedral angle values of these groups with the phenyl carrying amino groups are respectively $7.499(5)^\circ$ and $11.594(5)^\circ$. The Zn–Na distance is 3.4244 (9) Å. The positive charge of the Zn(II) and Na(I) ions are balanced by the ligand L^{2-} and the nitrate anion.

The environment around the 7-fold sodium atom is best described as a severely distorted bipyramid pentagon (Figure 3). The basal angles are in the range $[60.57(5)^\circ - 96.31(7)^\circ]$. These values deviate from the value of the ideal angle for a regular pentagon (72°) and the sum of the equatorial angles around the sodium atom is 365.53° . The apical position for the pentagon pyramid around Na is occupied by O5 and O8 with the angle value of $136.37(7)^\circ$.

The dinuclear units are connected by hydrogen bonds of type $\text{EtO}-\text{H}\cdots\text{O}^{\text{i}}-\text{NO}_2$ and, weak intermolecular hydrogen bonds of type $\text{C}-\text{H}\cdots\text{O}-\text{NO}_2$ ($\text{C13}-\text{H}\cdots\text{O7}^{\text{ii}}$ and $\text{C15}-\text{H}\cdots\text{O5}^{\text{ii}}$) (Figure 4, Table 3). The combined hydrogen bonds links give rise to a three-dimensional network architecture. It should be noted that the methoxy and phenoxo oxygen atoms are by no means engaged in any interaction in the structure.

A survey of the Cambridge Structural Database (CSD) (Version 5.39, last update November 2017; Groom *et al.*, 2016) [26] shows that dinuclear complexes of the ligand bis(2-hydroxy-3-methoxybenzylidene)-1, 2-diaminobenzene where the smaller N_2O_2 cage is occupied by a 3d metal and the larger, open O_2O_2 cage is occupied by one s-block metal are well documented. Complexes formed by transition metal and s-block atom, with the above organic ligand in which the d-block metal atom is situated in the smaller N_2O_2 cage and the s-block metal atom is situated in the larger O_2O_2 cage have been reported: twelve entries corresponding to d-s complexes [1, 10, 11, 16, 27–29] were found in the CSD. One trinuclear Ni–K–Ni complex has been reported recently [30].

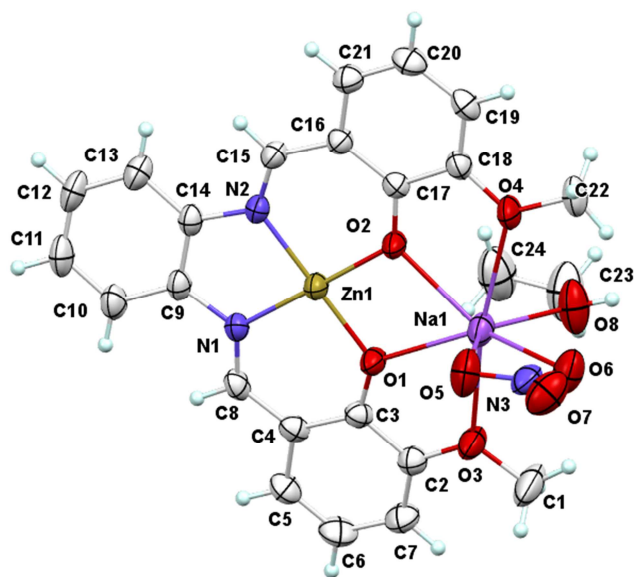


Figure 2. Crystal structure of the tetranuclear complex (2).

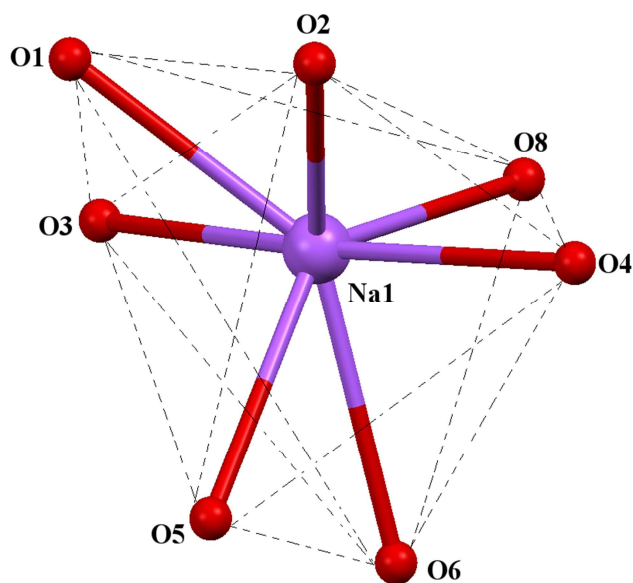


Figure 3. Plot showing the coordination sphere of the two sodium ions in the crystal.

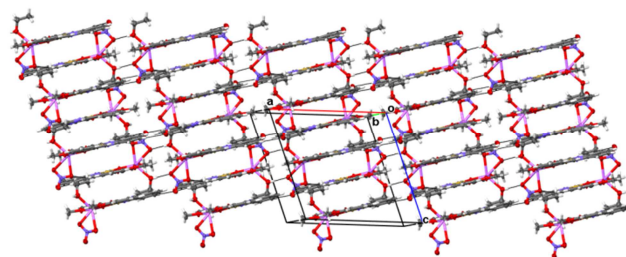


Figure 4. The packing of the complex in the crystal structure.

Table 1. Crystal data, X-ray data collection, data reduction and structure refinement for 2.

Crystal data	
Chemical formula	$C_{24}H_{24}N_3NaO_8Zn$
Mr	570.86
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	292
a, b, c (Å)	13.7247 (5), 13.7805 (4), 13.4006 (6)
β (°)	110.209 (5)
V (Å ³)	2378.47 (17)
Z	4
Calculated density (g. cm ⁻³)	1.5941
F (000)	1178
Radiation type	$\lambda = 0.71073$ Å
μ (mm ⁻¹)	1.11
Crystal size (mm)	0.03 × 0.02 × 0.01
Data collection	
Diffractionmeter	XtaLAB AFC12 (RCD3): Kappa single
Absorption correction	Multi-scan
T_{min}, T_{max}	0.233, 1.000
Limiting indices	
h	-18→17
k	0→18
l	0→18
No. of measured, independent and observed [$I \geq 2\sigma(I)$] reflections	57221, 13219, 11414
R_{int}	0.049
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.039, 0.107, 1.05
No. of reflections	5628
No. of parameters/restraints	340/1
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å ⁻³)	0.38 & -0.56

Table 1. Selected bond lengths [Å] and angles (°) for the complex 2.

Bond	Bond length [Å]	Bond	Bond angles (°)
Na1—O8	2.364 (2)	O2—Na1—O1	64.39 (5)
Na1—O2	2.3953 (16)	O2—Na1—O4	61.35 (5)
Na1—O1	2.4253 (16)	O6—Na1—O4	82.68 (7)
Na1—O5	2.490 (2)	O1—Na1—O3	60.57 (5)
Na1—O6	2.514 (2)	O6—Na1—O3	96.31 (7)
Na1—O4	2.6085 (18)	O1—Zn1—O2	85.58 (6)
Na1—O3	2.6350 (18)	O1—Zn1—N2	178.73 (6)
Zn1—O1	1.8875 (14)	O2—Zn1—N2	95.06 (6)
Zn1—O2	1.8937 (13)	O1—Zn1—N1	94.55 (7)
Zn1—N2	1.9278 (16)	O2—Zn1—N1	177.91 (7)
Zn1—N1	1.9404 (15)	N1—Zn1—N2	84.77 (7)
Na1—Zn1	3.4244 (9)	Zn1—O1—Na1	104.43 (6)
		Zn1—O2—Na1	105.36 (6)

Table 3. Hydrogen-bond geometry (Å, °).

<i>D</i> — <i>H</i> ⋯ <i>A</i>	<i>D</i> — <i>H</i>	<i>H</i> ⋯ <i>A</i>	<i>D</i> ⋯ <i>A</i>	<i>D</i> — <i>H</i> ⋯ <i>A</i>
O8—H8⋯O6 ⁱ	0.798 (19)	2.116 (19)	2.912 (3)	174 (4)
C15—H15⋯O5 ⁱⁱ	0.93	2.51	3.392 (3)	158.7
C13—H13⋯O7 ⁱⁱ	0.93	2.60	3.406 (3)	145.0

Symmetry codes: (i) $-x+2, -y+1, -z+1$; (ii) $-x+1, -y+1, -z+1$.

4. Conclusion

In summary, we have successfully synthesized and studied the hetero dinuclear complex containing one zinc (II) and one sodium (I) ions. A suitable crystal of the complex was obtained by slow evaporation of ethanol solution of the compound for two weeks. Infrared spectra of the compound and the metalloligand were discussed in this paper. Crystal data, X-ray data collection, data reduction and structure refinement of complex 2 are studied. The heteronuclear complex crystallizes in the monoclinic system with a space group $P2_1/c$. The environment around the zinc (II) is square planar while the environment around the 7-fold sodium atom can be described as a severely distorted bipyramidal pentagon.

Supplementary Materials

CCDC-2011039 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.

Conflicts of Interest

The authors declare that they no competing interest.

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