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Synthesis, Characterization and Antibacterial Properties of Some Thiocyanato Bridged Heteronuclear Complexes of Nickel

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Abstract

A series of heterobinuclear, thiocyanato-bridged complexes of cations $[M(NH2X)_6]^{2+}$, and $[M(en)_3]^{2+}$ (where X = H, Ph; M=Cu, Co, Cd, Zn) with anion $[Ni(NCS)_4]^2$ -have been prepared by 1:1 molar ratio. The complexes have been characterized by FT-IR, ¹³C NMR and UV-vis spectroscopy, metal analysis, magnetic and conductance studies. The complexes have the following composition: $[M(NH_2X)_5][Ni(NCS)_4]$, and $[\{M(en)_2\}\{Ni(NCS)_4\}]$ n. The antibacterial activity of the prepared complexes against the *gram positive, Bacillus cereus* and gram negative *Salmonella typhi, pseudomonas alruginosa, Escherichia coli* pathogens also been investigated. *Key words: Heterobinuclear, Thiocyanato-bridged complexes, Nickel, Antibacterial properties.*

Introduction

Due to its ambient character, the thiocyanate group can serve as a bridging ligand, similarly to the cyanide group. In coordination polymers, a thiocyanate ion must act as a rigid bridged ligand and link a pair of metal centers through 1,1- μ -SCN, 1,1- μ -NCS or 1,3- μ -SCN configuration to satisfy the coordination number of the metal ion [1,2]. Binding mode sensitivity of thiocyanate ion has attained great interest in recent years. The thiocyanate moiety was reported to have an ability to modify the

biochemical behavior when present in mixed ligand coordination complexes. It should be noted that some bimetallic compounds assume different magnetic properties. The literature concerning the structural and/or magnetic studies of thiocyanato-bridged transition metal heterocomplexes is rather limited.

Bimetallic thiocyanate ligand based single crystals of MnHg(SCN)₄ and CdHg(SCN)₄ or the complexes of molecular formula $[MM'(SCN)_4(L)]$ $[M = Co^{2+}, M' = Cd^{2+},$ Hg²⁺; L=N, N'-bis(benzylidene)-1,2,

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phenylenediamine Schiff base] have been synthesized and characterized [1,3]. The heteronuclear $[Hg(SCN)_3Cu(en)_2]NO_3$ complex [en=1,2-ethandiamine and 1,10-phen=1,10phenanthroline], has been synthesized [4]. Based on the bridging function of thiocyanato group two complexes formulated as, [{(Cu(L)_2)_2OH} (Cr(NCS)_6)] •H_2O, where L = 2,2 -bipyridine (bpy) and 1,1,4,7,7pentamethyldiethylenetriamine (PMDT) are reported [5].

Cadmium(II) complexes, [Cd $(\mu$ -SCN)₂(L¹)₂], $[Cd(\mu-SCN)_2(L^2)], [Cd(NCS)(\mu-SCN)]$ $(L^3)]_2$, have been synthesized by reacting cadmium acetate/NH₄SCN with 4-aminopyridine (L^1) , $C_5H_4N-C(CH_3)=NNH_3$ (L^2) , and $C5H_4N-C(CH_3)=N-CH_2-CH_2-N(C_2H_5)_2$ (L^3) , respectively, in methanol [6]. A series of heterobinuclear, thiocyanato-bridged complexes of copper(II) and nickel(II) of macrocyclic ligands having the composition $(ML)_{v}[M'(NCS)_{4/6}]_{v}$ [M=Cu, Ni, M=Ni, Co, Cr, Fe; L=5,12-dimethyl-[14]-1,4,8,11tetraazacyclotetradeca-4,11-diene and 5,7,12,14-tetramethyl-[14]-1,4,8,11tetraazacyclotetradeca -4,11-diene] have been characterized by variable-temperature magnetic susceptibility.2 There are a few heterometallic compounds of the type $[CuL]_{3}[Cr(NCS)_{6}]_{2} \cdot nH_{2}O$ (L=2.2-bipyridine, n=0; L=o-phenanthroline, n = 1; L = diethylenotriamine, n=1); $[CuL_4]_3[Cr(NCS)_6]_2 \bullet nH_2O;$ (L=imidazole, n=2; $[Cu(en)_{2}Mn(NCS)_{4}(H_{2}O)_{2}]n$ (en = ethylendiamine) [7-9]. The literature covering an analogous system, in which the thiocyanate group plays the role of a bridging ligand, it is evident that complexes have tremendous character. The bimetallic complexes of the type [MLx][M'Ly] where, M = Cu, Cd, Ni, Co, Zn and M'= Hg, Lx = ethylenediamine, aniline, NH₃ and Ly = I-, SCN- have been characterized [10]. In continuation of our research herein, the synthesis of thiocyanatobridged heteronu-clear complexes containing [Ni(NCS)₄]²⁻ anionic group are reported. The elemental analyses, FT-IR, NMR, electronic spectra, magnetic and conductivity studies indicate that the reaction between $[Ni(NCS)_{4}]^{2-}$ ion and starting metal(II) complex cations, due to bridging properties of thiocyanato ions. Antibacterial activities of some of the prepared complexes are investigated.

Experimental

Chemicals

Ethylenediamine, aniline, methanol, chloroform and N, N-dimethylformamide (DMF) were obtained from Aldrich Chemical Company Ltd. Ammonium nitrate and Nickel chloride (crystal), Ammonium thiocyanate, Cobalt (II) sulphate were obtained from M|S Merck (Germany). Perchloric acid, nitric acid, sulphuric acid and ammonia were obtained from BDH Chemicals Ltd. All chemicals except solvent were used as received. Solvents were dried by standard methods and distilled under an inert atmosphere.

Analytical methods

The analyses of the metal contents of the prepared complexes were obtained by Atomic Absorption Spectrophotometer (model Thermo Scientific ICE-3000) from Biological Laboratory of Chittagong University, Chittagong. The Infrared spectra of the prepared complexes were obtained by FTIR spectrophotometer (Model- 8900, Shimadzu, Japan) using KBr as the matrix in the range 400-4000 cm⁻¹ from research laboratory of the Department of Chemistry, C.U. Chittagong. Polystyrene was used as the standard to calibrate the spectrophotometer. Electronic absorption spectra were run on Shimadzu UV-Visible Recording Spectrophotometer (Model-1800) using 1 cm cells. An electro thermal melting point apparatus was used for the determination of the melting or decomposition points of the complexes. Using N, N-dimethylformamide (DMF) as the solvent, the solutions of the complexes (of the order of 10-3 M) were used for conductivity measurements. Conductivity measurement was performed on a Philips Conductivity Meter (Model-HI 9255). Determination of conductivity of an electrolytic solution involves measuring of the electrical resistance of that solution at a particular temperature, usually 25°C. Magnetic susceptibility values of some of the prepared complexes were determined using the Magnetic Susceptibility Balance, (Sherwood Scientific) from in the laboratory of Chittagong University, Bangladesh.

Preparation of Bimetallic Complexes [Ni(SCN),]²⁻

2 mmol (0.88 g) nickel chloride crystals were dissolved in about 40 mL of water in a beaker. A deep green color solution of NiCl_2 was formed. Ammonium thiocyanate was added to it when the deep green coloration was fade.

$[M(NH_3)_6]^{2+}$, $[M(anil)_6]^{2+}$ or $[M(en)_3]^{2+}$

2 mmol (0.0425 g) of metal chloride or nitrate was dissolved in about 10 ml of water in another beaker. An excess amount of ammonia, aniline or ethylenediamine was added to it drop wise. The mixture was heated to boil for about 15 minutes.

Heteronuclear complexes

The two solutions were mixed together in their hot states. The mixture was stirred for some time and allowed to cool with frequent stirring. The precipitate was formed four days later and the solid product formed was filtered off and washed with water and dried over calcium chloride. The precipitate formed was preserved in desiccators. The following bimetallic complexes were prepared by the same procedure as stated above.

A possible reaction for the preparation of complex, $\{[Cd(en),][Ni(SCN)_{4}]\}_{n}$

1. $CdCl_2$ + en (excess) = $[Cd(en)_3]Cl_2$ 2. $NiCl_2$ + NH_4SCN = $(NH_4)_2[Ni(SCN)_4]$ Then from 1 and 2,

 $n[Cd(en)_{3}]Cl_{2} + n(NH_{4})_{2}[Ni(SCN)_{4}] = \{[Cd(en)_{2}][Ni(SCN)_{4}]\}_{n} + 2NH_{4}Cl$

The color, yield and melting point of the prepared complexes are given in parentheses. i. [Cu(en)₂][Ni(SCN)₄] (Color: Deep blue; Yield: 95%; m. p.:210°C), ii. [Cd(en),] [Ni(SCN)₄] (Color: Violet; Yield: 95%; m. p.:174°C), iii. $[Co(en)_2][Ni(SCN)_4]$ (Color: Radish brown; Yield: 95%; m. p.: >200°C), $[Cu(NH_3)_5][Ni(SCN)_4]$ (Color: Blue; iv. Yield: 95%; m. p.: >210°C), v. $[Cd(aniline)_5]$ [Ni(SCN)₄] (Color: blue; Yield: 90%; m. p.: $>210^{\circ}C$ (d)), vi. [Cu(aniline)₅][Ni(SCN)₄], (Color: Black; Yield: 95%; m. p. :> 210 C [Zn(aniline)₅][Ni(SCN)₄] (Color: (d), vii. white; Yield: 95%; m. p. :> 210 C (d)), viii. [Co(aniline)₅][Ni(SCN)₄] (Color: black; Yield: 95%; m. p. :> 210 C (d).

Test for of Antimicrobial Evaluation

Some of the synthesized complexes were screened by the agar well diffusion method for their antibacterial activity against different types of bacteria, gram positive- Bacillus cereus and gram negative- *Salmonella typhi*, *pseudomonas alruginosa, Escherichia coli* regarded as pathogen to man. All media and bacteria suspension were prepared using a suitable method. The in-vitro evaluation of antimicrobial activities was performed according to the diffusion technique. The bacteria were grown in nutrient broth at 37°C for 24 hours. The complexes were tested using diffusion on solid media. Sterile (5 mm) diameter sensitivity paper disc were impregnated with concentration of dimethylformamide (DMF) and their bimetallic complexes at concentration of 50µg/ cm³ and placed in the nutrient agar. The plates were then incubated for 24 hours. Additionally antibiotics disc for cephalosporin were tested as positive control. The results were recorded by measuring the growth inhibition (% zones of inhibition) surrounding the disc [10].

Results and Discussion

Bimetallic mixed-ligand complexes were prepared using water as the solvent by the reaction of cationic complex with a anionic complex in the molar ratio 1:1. All the complexes were obtained almost immediately after mixing of the component metal complex solutions. During preparation of { $[Cd(aniline)_5]$ $[Ni(SCN)_4]$ }, the $[Cd(aniline)_5]^{2+}$ complex mixture was heated to dissolve the oily layer formed due to the presence of aniline. All the complexes are insoluble in chloroform. Some of them are moderately soluble in methanol. All complexes are highly soluble in DMSO and DMF.

Qualitative characterization

Primarily, the complexes were qualitatively characterized by the nickel ion identification.

A little amount of the prepared complex was taken in a test tube. Concentrated nitric acid (HNO_3) was added to the sample and gently warmed on a water bath for the decomposition. The solution was made slightly alkaline with NH₄OH. Add a few drops of alcoholic dimethylglyoxime, $(C4H_8O_2N_2)$ was added. A red solution of nickel (II) dimethylglyoxime was appeared as the indication of the presence of nickel.

Infrared spectra

In the mixed metal complexes which are the subject of this project, the main stretching modes are for $v_{\text{N-H}}$, $v_{\text{C-N}}$, $v_{\text{C-C}}$, $v_{\text{C-S}}$, $v_{\text{S-C}} \equiv N$ and $v_{\text{M-N}}$. The infrared spectra for the present compounds taken in the range 400-4000 cm⁻¹ help to indicate regions of absorption due to the above mentioned vibrations. The thiocyanate ion has three fundamental modes of vibration: the C-N stretch, v_1 ; the doubly degenerated v2 and the C-S stretch, v_3 . These vibrational modes occur at different frequency ranges. The N-bonded thiocyanate group exhibits $v_{\text{C-N}}$ in the regions of 2020-2096 cm⁻¹ and the S-bonded thiocyanate group exhibits $v_{\text{C-N}}$ above 2100 cm⁻¹ [2]. Appearance of

strong v_{C-N} bands in the regions 2020-2096 and 2100 cm⁻¹ in the present complexes is a good indication of M-SCN-M coordination of the thiocyanate group. This band gives a clear split, what proves the presence of thiocyanato ions in bridging and terminal positions [5]. The presence of two bands in between 2105-2160 cm⁻¹ clearly indicates the presence of thiocyanate as bridging unit [1]. The weak band corresponding to v(C-S) vibration appears at 771 cm⁻¹, and the vNCS bending mode is observed at 480 cm⁻¹ as a shoulder [2]. The peaks at 3442 and 3255 cm⁻¹ were assigned to the stretching vibrations of amine group [4, 6]. FTIR spectra also show the very broad band at 360 cm⁻¹ with shoulders due to superposition of bands arising from v_{Cr-N} and v_{Cu-N} vibrations. The assignment of v_{Cu-S} vibrations is only tentative because of low intensity of these bands [5]. For all the complexes, the presence of v_{M-N} and v_{M-S} vibration in the same region confirms coordination and N- donor character of the ligands to the metal ion. Furthermore, the presence of bands in $\delta(NCS)$, v(C-S), v(M-N) and v(M'-S) regions are diagnostic for the nature of thiocyanate bonding [1]. A representative IR Spectrum of {[Cd(en),]



Figure 1. IR Spectrum of {[Cd(en)₂][Ni(SCN)₄]}.

¹³C NMR

¹³C nmr of the complex $[Zn(aniline)_5]$ [Ni(NCS)₄] was obtained. A splitting peak at (δ) 123.5 indicates the bridging thiocyanate and that multiplet at (δ) 135 comply with phenyl group of aniline.

UV-Vis spectra

The UV-Vis spectra of all complexes were obtained in DMSO solution. In the UV-Vis spectrum of complex the first electronic absorption band observed at around 260 nm is due to the met¬al to ligand charge transfer (MLCT). The band at around 500 nm assigned

to the *d-d* transition of metal ion indicates some distortion from the perfect symmetry. The broad band at around 400 nm can be assigned to the d-d transition of metal ion and the broadness of the peak reveals the inherent distortion of this type of complexes from perfect symmetry [2,4]. The peaks observed below 350 nm are assumed as due to $\pi \rightarrow \pi^*$, $n \rightarrow \pi^*$ and $n \rightarrow \sigma^*$ transitions. And the peaks observed at 983 may be assumed as due to the presente of metal tetrathiocyanate. A representative electronic spectra of {[Cu(en)₂] [Ni(SCN)₄]} is shown in Figure 2.



Figure 2. Electronic spectra of {[Cu(en)₂][Ni(SCN)₄]}.

Magnetic measurements

From the experimental values of magnetic measurements of some of the prepared complexes are diamagnetic thus indicating the d10 electronic configuration. From the experimental values of magnetic measurements of some of the prepared complexes, it is found that magnetic moments of some of the mixed metal complexes are lower than the expected theoretical spin only values. The possible reason of such lower magnetic moment may be due to weak antiferromagnetic interactions in the mixed metal complex transmitted by the thiocyanato group.2 The exchange interactions seem to be super exchange type and most likely through the thiocyanato or the thio groups. However, weak bonds and longer bond lengths may hide or prevent possibility of exchange interactions [1, 2, 5, 9].

Conductance

The conductance values 0-60 ohm⁻¹cm²mol⁻¹ of the prepared complexes in DMSO solution corresponding to non electrolytes. The low conductance values 60-120 ohm⁻¹cm²mol⁻¹ of the prepared complexes in DMSO solutions corresponding to 1:1 electrolytes strongly support proposed formulation of the prepared complexes This suggests +2 oxidation states of the metal ions present in such complexes. The conductance 150> high values ohm⁻¹cm²mol⁻¹ of the prepared complexes in DMSO solutions indicates their electrolytic nature corresponding to 1: 2 electrolytes strongly support proposed formulation of the prepared complexes [11].

Spectral (IR, UV-vis), magnetic, conductivity data and metal content of the prepared complexes have been given in Table 1.

Code No.	Complexes	υ _{N-H}	v _{c-c}	v _{c-n}	𝔥C≡N (cyanide)	v _{C-S}	υ _{S-C≡N}	υ _{M-N}	% of Ni (Calc.)	electronic spectral data	μ _{eff} B.M.	λ^{o}_{m} ohm ⁻¹ cm ² mol ⁻¹
R 1	[Cu(NH ₃) ₅] [Ni(SCN) ₄]	3280 s 3350ms	970m 1040m	1380w 1468m	2100s 2115ms	750m	630s	690s 522s	13.36 (13.54)	784,384, 309	0.98	68
R ₂	[Cu(en)2] [Ni(CNS)4]	3298m 3261m	1091vs 1274m	1446ms 1465sh	2096vs 2075vs	777ms 796w	505ms 528ms	460w	12.54 (12.49)	370,335, 262	1.83	120
R ₃	[Co(en)2] [Ni(SCN)4]	3190s 3250ms	1106m 1096s	1384s 1363sh	2061vs 2096vs	775sh 790w	513w 650w	472w	11.52 (11.22)	09,328, 261	1.25	91
R4	[Cd(en)2] [Ni(SCN)4]	3167s 3271s	977w 1033m	1367w 1388w	2058vs 2131vs	669ms 752w	518ms 464w	420w	12.38 (17.10)	930,557, 383,266	1.59	165
R5	[Cu(anil)5] [Ni(SCN)4]	3253ms 3300w	1047w 1076w	1448w 1425w	2032vs 2119ms	761w 806w	690w	599w	6.75 (6.53)	549,393, 300	3.51	42
R 6	[Zn(anil)5] [Ni(SCN4)]	3253ms 3325ms	999w 1047ms	1469w 1492s	2121s 2131s	780sh 804w	690ms	520w	6.81 (6.91)	405,296, 264	2.08	49
R ₇	[Cd(anil)5] [Ni(SCN)4]	3253ms 3325ms	999w 1047s	1338w 1419w	2113sh 2131s	759s 770sh	690s	516w	6.71 (6.89)	07,397, 294, 266	2.42	68
R8	[Co(anil)5] [Ni(SCN)4]	3253w 3325w	999w 1047s	1458w 1492s	2088sh 2123s	777w 804ms	599s 690vs	520s	6.54 (6.95)	908,395, 298,265	2.53	53

Table 1. Spectral (IR, UV-vis), Magnetic, Conductivity data and metal content of the complexes.

* vs = very strong; s = strong; ms = medium strong; m = medium; w = weak; vw = very weak; sh = shoulder, λ^{o}_{m}

=Molar Conductivity

Geometry

The crystal structure of [NiL]₃[Fe(NCS)₆]₂ (where L = 5, 6, 12, 13-Me4-[14]-4, 11-dieneN4) suggested the exchange interaction between Cu II and Fe III ions that is transmitted by the thiocyanate groups [12]. The environment of the Fe III ion is octahedral, while the thiocyanate groups are nearly linear. Each Fe III ion is bound to two copper(II) ions when thiocyanate groups coordinate to the copper(II) ions by their sulphur atom [2]. The environment of the NiII ion in [CuL₁] $[Ni(NCS)_6].2H_2O$ complex is squarepyramidal, while the thiocyanate groups are nearly linear [2]. A distorted square-pyramidal structure was proposed for the complex, (CuL) [Co(NCS)₄], (L=N-rac-(5,12-Me2-7,14-Et2-[14]-4,11-diene N4) with four equatorial

nitrogen atoms of macrocyclic ligand and one sulfur atom from the thiocyanato bridging group in an axial position. The cobalt (II) centre posses one bridging and three nonbridging thiocyanate ligands [13]. The structure of $[Cu(en),Mn(NCS),(H_2O),]n$ (en = ethylenediamine) consists of chains in which alternating Cu(en)₂²⁺ and Mn(NCS)₄(H₂O)₂²⁻ entities are bridged by thiocyanate ligands [9]. Compound $[Ni(en)_2]_4 [Fe(CN)_5 NO]_2$ [Fe(CN)₆].5H₂O is obtained by reaction of [Fe(CN)₅NO]²⁻ with [Ni(en)₃]Cl₂ in aqueous solution. The slow dissociation of $[Ni (en)_3]^{2+}$ into [Ni-(en)2]2+ leads to the growth of crystals suitable for X-ray diffraction [4,14]. On the basis of the above discussion and

experimental data it is supposed that heterobinuclear thiocyanato-bridged

complexes have been synthesized by the reaction of cations, [M(NH₃)₆]²⁺, [M(aniline)₆]²⁺ and $[M(en)_3]^{2+}$ with anion $[Ni(NCS)_4]^{2-}$ in 1:1 molar ratio. Complexes having the following compositions. $[M(NH3)_5][Ni(NCS)_4],$ $[M(aniline)_{s}]$ $[Ni(NCS)_{4}]$, and $\{[M(en)_{2}]\}$ $[Ni(NCS)_{4}]$ (where M=Cu, Zn, Cd, Co) are prepared. The coordination geometry around the Ni^{2+} ion in $[Ni(NCS)_4]^{2-}$ anion is squareplanner. The Ni²⁺ centre possesses three nonbridging and one bridging thiocyanate ligands, while the thiocyanate groups are nearly linear.

The environment of the cations in $[M(NH_3)_5]^{2+}$ or $[M(aniline)_{s}]^{2+}$ are octahedral when sixth coordination site is occupied by one bridging thiocyanate ligands from anion. Geometry was also confirmed by the ¹³C nmr of the complex, $[Zn(aniline)_{5}]$ $[Ni(NCS)_{4}]$ when splitting of peak at (δ) 123.5 indicates the bridging thiocyanate and that multiplet at (δ) 135 comply with phenyl group of aniline. The proposed schematic structure of the complex is presented in Figure 3.



Figure 3. Proposed schematic structure of the complex, $[M(NH_2X)_5][Ni(NCS)_4]$.

to two thiocyanate groups from neighbor anions forms a long chain polymeric structure of $[{M(en)_2} {Ni(NCS)_4}]n$. The environment of the cationic metal ion is octahedral, while the thiocyanate groups are nearly linear. The thiocyanate groups coordinate to the

Central metal ion of $[M(en)_2]^{2+}$ being bound metal ions of cations by their sulphur atom. A proposed schematic view of complex $[{M(en)}_{2} {Ni(NCS)}_{4}]n$ is presented in Figure 4. But actual geometry is not possible to be elucidated without crystal structure of the prepared complexes.



Figure 4. Proposed schematic view of complex, [{M(en)₂} {Ni(NCS)₄}]_n.

Antibacterial activity

The inhibition zones of the test organisms for different complexes are presented in Figure 5 (a, b). Results of the inhibition have been graphically shown in Figure 6. It is found that, except R4, all compounds are comparatively effective against Salmonelatyphi and Pseudomonas aeruginosa. The tested complexes except R_1 , R_5 , R_6 , are effective against Bacillus cereus. Only R₄, R₇ of the tested complexes are effective against Escherichia coli. In addition to chelation, some important factor such as nature of the metal ion, metal ion coordinating site,

hydrophilicity, hypophilicity and binuclear structure may have considerable influence on the antibacterial activity. The chelate complexes may deactivate various cellular enzymes which play vital role in various metabolic pathways of these microorganisms. Other factors such as solubility, conductivity and dipole moment affected by the presence of metal ions, may be the reasons for the increased biological activities of the metal complexes. Therefore, the antibacterial activity of the complexes can be ascribed to an intricate blend of all of the above contributions.





Figure 5a. Zone of inhibition against *Bacillus cereus* by complexes R_{4} .

Figure 5b. Zone of inhibition against *Pseudomonas aeruginosa* by complexes R_7 , R_8 ,



Figure 6. The antibacterial activity of the tested metal complexes.

Conclusion

The results of these analyses show that the theoretical and experimental percentages of the metal content are in a close consistence. The prepared complexes have been clearly characterized by FT-IR spectroscopy, UV-Vis spectra, magnetic and conductivity measurements. The data indicate the formation of such complexes. Conductivity

measurements indicate their electrolytic nature consistence to +2 oxidation state of the metal ions present in such complexes. The magnetic measurements indicate some of the complexes to show antiferromagnetic interactions. The high yield of the complexes indicates good possibilities of their analytical uses to determine metal contents of some related compounds. Further works are required to exploit and ascertain such possibilities. From comparative study, the prepared complexes have been found to be quite effective against selected human pathogenic bacteria.

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