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Triazole-naphthalene based fluorescent chemosensor for highly selective naked eye detection of carbonate ion and real sample analyses



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ARTICLE INFO	A B S T R A C T		
<i>Keywords:</i> Triazole-naphthalene hybrid Colorimetric sensor Carbonate Real sample analysis Logic gate	Herein, the design and synthesis of triazole-naphthalene hybrid for selective colorimetric and fluorometric sensing of carbonate ion has been reported. The probe has been thoroughly characterized by ¹ H and ¹³ C NMR and HRMS spectroscopy. CK exhibits an excellent selectivity and sensitivity towards carbonate ions over other anions by changes in both UV–Vis absorption spectra and emission spectra. The limit of detection of carbonate ion in absorbance and emission spectra has 7.2 nM and 1.8 μ M respectively. The plausible sensing was confirmed with the help of the Job's plot, FTIR, NMR titration and theoretical studies. Also, naked eye detection method of carbonate from colorless to yellow color is the most conspicuous application of probe CK . The receptor CK providing valuable practical sensor for environmental analyses of carbonate ion.		

gate circuit by using the sensing performance of carbonate ion with probe.

1. Introduction

Recognition of anions by fluorescent and colorimetric chemosensors became more prevalent in environmental monitoring, biological science, drug delivery and medicinal chemistry [1–4]. Amongst the anions, monitoring the occurrence of carbonate ions (CO_3^{2-}) befitted more essential for day-to-day life. Because, CO_3^{2-} ions are the chief constituent in carbon cycle [5] and also it exists predominantly as metal carbonates in the environment [6,7]. The extreme level of carbonate anion also leads to decisive stage [8]. Since there are numerous methods for carbonate sensing, chemosensing are the most sensible and highly selective method for carbonate sensing [9–12]. Hence, designing a chemosensor for the selective detection of $\mathrm{CO_3}^{2-}$ anion without any interferences is an indispensable research area now a days. Furthermore, most of the chemosensors depends upon photo induced electron transfer (PET) mechanism which involves enhancement or quenching of fluorescence in turn produces Off-On or On-Off sensing mechanism [13–16]. Till to date, based on PET mechanism for the carbonate sensing were infrequently described in reports [17]. So, manipulating carbonate sensor via PET mechanism aroused as an outstanding depiction in chemosensor approach. In recent times, Tavallali et al. [18] established novel use of calmagite as colorimetric anion chemosensor and solid-state sensor for carbonate ion in running water. Li et al. [19] developed

inverse opal photonic crystal polymers in aqueous solution for the visual detection of carbonate ions. Velmurugan et al. [20] synthesized dipyrene based fluorescent probe for the dual detection of silver and carbonate ions with its bioimaging applications. Liu et al. [21] proposed the cascade recognition for Fe^{3+} and CO_3^{2-} based on asymmetric squaraine dye with logic gate applications. Singh et al. [22] designed a chemosensor based on hydrazinyl pyridine for selective detection of F⁻ ion in organic media and CO₃²⁻ in aqueous media. Zhang et al. [23] deliberated diarylethene based probe for dual recognition of Cu^{2+} and CO_3^{2-} and its applications. Saleem et al. [24] explained the facile synthesis of an optical sensor for CO_3^{2-} and HCO_3^{-} detection. Apart from these literatures, it is obligatory to construct a chemosensor with simple starting materials aimed at the efficient detection of $\mathrm{CO}_3{}^{2-}$ anion selectively without any competitors. Accordingly, herein we reported newly intended novel naphthalene and triazole hybrid probe from the simple Knoevenagel condensation of 2-Hvdroxy-naphthalene-1-carbaldehvde and naphthalene substituted triazole with good yield for the selective colorimetric sensing of carbonate ion in aqueous medium. The interaction of probe CK with carbonate ion occurs through inhibition of photo induced electron transfer mechanism. The main advantages of our work such as metal free probe, simple detection method compared to potentiometric [25] and electrochemical [26], dual detection method (colorimetric and fluorometric), detection medium is aqueous, naked

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Reagents and condition: (a) N, N-diisopropylethylamine, DMF, $50^{\circ}C$; (b) K₂CO₃, DMF, $50^{\circ}C$; (c) Methanol, Glacial acetic acid, $80^{\circ}C$.

Scheme 1. Schematic representation of the probe synthesis. Reagents and condition: (a) N, N-diisopropylethylamine, DMF, 50 °C; (b) K₂CO₃, DMF, 50 °C; (c) Methanol, Glacial acetic acid, 80 °C.



Fig. 1. (a) Selectivity spectra of CK (10 μ M) with various anions (5 μ M) (b) Sensitivity experiment of CK with difference concentration (0.5 to 5.5 μ M) of carbonate ion.

eye detection, probe is applied to real sample analysis, and we have very low limit of detection range compared to recently published articles. The application of this carbonate sensor in commercial aspect is proceeding.

2. Experimental section

2.1. Materials and methods

All the chemicals and reagents were used in this work are analytical grade. hydrazinecarboximidamide, *N*, *N*-diisopropylethylamine, sodium hydroxide, hydrochloric acid, potassium carbonate, 2-hydroxy-1-naph-thaldehyde purchased from Sigma Aldrich, and all the dry solvent were purchased from Merck. The ¹H and ¹³C NMR spectrum were recorded on Bruker Avance 400 MHz in a DMSO- d^6 solvent using TMS as an internal standard and chemical shifts are given in ppm (δ -scale) and coupling constants are given in Hertz (Hz). Column chromatography was carried out in silica gel (60–120 mesh size) using petroleum ether and dichloromethane as an eluent. UV–Vis spectrum was recorded in a

JASCO (V-630) and emission was recorded in a JASCO (F-8500). In addition, the density functional calculation was performed by using the Gaussian 09 program. The ground-state and excited state geometries were optimized by using the restricted B3LYP/6–31 g (d, p).

2.2. UV-visible and emission spectroscopy

The probe was dissolved in acetonitrile solvent and the concentration of the probe solution is 1 mM. 20 μ L of it was diluted to 2 ml of DD water placed in a 3 ml cuvette and the total concentration of probe were 10 μ M for the selectivity, and competitive spectra in absorbance spectroscopy. UV–Vis spectra were obtained at room temperature. The total concentration of probe was 60 μ M for the selectivity, and competitive spectra in emission spectroscopy. Emission spectroscopy were obtained at room temperature.



Fig. 2. Interference study of 1. CK alone (10 μ M) 2. CK + CO₃²⁻ 3. CK + CO₃²⁻+CH₃COO⁻, 4. CK + CO₃²⁻+SCN⁻, 5. CK + CO₃²⁻+Br⁻, 6. CK + CO₃²⁻+Cl⁻, 7. CK + CO₃²⁻+F⁻, 8. CK + CO₃²⁻+HCO₃⁻, 9. CK + CO₃²⁻+HPO₄²⁻, 10. CK + CO₃²⁻+NO₃⁻, 11. CK + CO₃²⁻+NO₃⁻, 12. CK + CO₃²⁻+S²⁻, 13. CK + CO₃²⁻+I⁻, 14. CK + CO₃²⁻+SO₄²⁻, 15. CK + CO₃²⁻+HO₃⁻, 16. CK + CO₃²⁻+PO₄²⁻, 17. CK + CO₃²⁻+CIO₄²⁻, 18. CK + CO₃²⁻+HSO₃⁻.UV-vis spectrum, concentration of carbonate ion (5 μ M) and competitive ion concentration is (5 μ M).



Fig. 3. Selectivity spectra of the CK (60 $\mu M)$ with various anions (30 $\mu M)$ in fluorescence.

2.3. Synthetic procedure of probe CK

The synthesis of the compound **CK** started with commercially available starting material **1** which is treated with hydrazinecarboximidamide in presence of *N*, *N*-diisopropylethylamine and dimethyl formamide solvent at 50 °C for 12 hrs, 20 ml of 2 N NaOH solution is added for further completion of reaction. Then the mixture is filtered and the filtrate is neutralized with 2 N hydrochloric acid. The obtained precipitate is filtered, dried and recrystallized from ethanol. Compound **2** is obtained as a white solid with 76% yield. To a mixture of compound **2**, K₂CO₃ and DMF, 2-chloromethylacetate is added slowly at 50 °C and the solution is stirred constantly for 12 hrs. Then the reaction mixture is poured into water to get the solid precipitate and the solid is filtered, dried and recrystallized from ethanol [27]. Compound **3** obtained as a white solid with 88% yield. A mixture of compound **3** and 2-hydroxy naphthaldehyde is heated to 80 °C for 12 hrs in presence of catalytic amount of glacial acetic acid and methanol solvent to get the solid



Fig. 4. Interference study of 1. CK alone, 2. CK + CO_3^{2-} 3. CK + $CO_3^{2-}+CH_3COO^-$, 4. CK + $CO_3^{2-}+S^{2-}$, 5. CK + $CO_3^{2-}+HCO_3^-$ 6. CK + $CO_3^{2-}+SCN^-$, 7. CK + $CO_3^{2-}+Br^-$, 8. CK + $CO_3^{2-}+CI^-$, 9. CK + $CO_3^{2-}+F^-$, 10. CK + $CO_3^{2-}+I^-$, 11. CK + $CO_3^{2-}+HPO_4^-$, 12. CK + $CO_3^{2-}+NO_3^-$, 13. CK + $CO_3^{2-}+N_3^-$, 14. CK + $CO_3^{2-}+SO_4^{2-}$, 15. CK + $CO_3^{2-}+NO_2^-$, 16. CK + $CO_3^{2-}+PO_4^{2-}$, 17. CK + $CO_3^{2-}+CIO_4^{2-}$, 18. CK + $CO_3^{2-}+HSO_3^-$.concentration of carbonate ion (30 μ M) and competitive ion concentration is (30 μ M).



Fig. 5. Fluorescence spectra of CK upon the gradual addition (2.5 to 32.5 $\mu M)$ of ${\rm CO_3}^{2-}$ ion.



Scheme 2. Plausible sensing mechanism of probe with receptor.



Fig. 6. Job's plot of carbonate ion in (a) absorbance spectra and (b) emission spectra.



Fig. 7. FTIR spectra of CK alone and CK with carbonate ion.

product **CK**. The product is filtered, washed well with water, dried and weighed. The yield of compound **CK** found to be 88%. ¹H NMR (400 MHz, DMSO) δ 12.65 (S, 1H), 10.13(S, 1H), 8.62 (d, J = 8 Hz, 1H), 8.38 (d, J = 8 Hz, 1H), 8.00 (d, J = 8 Hz, 1H), 7.82 (d, J = 8 Hz, 1H), 7.74 (t, J = 16 Hz, 1H), 7.68(t, J = 16 Hz, 2H), 7.47(d, J = 4 Hz, 1H), 7.38(d, J = 8 Hz, 3H), 7.04 (d, J = 6 Hz, 1H), 4.11 (dd, 2H), 2.96 (s, 3H) 2.68–2.60 (m, 1H), 1.19(q, 2H) 0.86(q, 2H). ¹³C NMR (400 MHz, DMSO) δ 169.02, 163.29, 162.67, 158.44, 150.77, 142.93, 132.48, 129.04, 128.31, 128.10, 127.62, 127.01, 126.88, 125.51, 124.62, 122.96, 122.72, 119.17, 110.18, 52.98, 33.92, 13.38, 8.07, 7.62. HRMS *m/z* calcd 508.1569, found 508.1743.

3. Results and discussion

CK synthesized from the condensation reaction of naphthalene substituted triazole and 2-hydroxy-naphthalene-1-carbaldehyde (Scheme 1). After that, our synthesized compound was confirmed by various analytical techniques. Hereafter, we measured the optical properties of the CK in both absorbance and emission spectroscope in DD water. In absorbance spectrum, CK shows three absorbance band at 282, 338, 402 nm, the 282 and 338 nm band represented as π - π * and n- π * transition respectively. The lower energy band at 402 nm is may be due to the photo induced electron transfer and C-N isomerism. The probe CK also has three absorbance band after the addition of carbonate ion,

probe 402 nm band was disappeared and new band at 454 nm was appeared and remaining two π - π^* and n- π^* was slightly bathochromic shifted and remaining others anions does not have spectral changes (Fig. 1a). Hereafter, we doing the sensitive experiment in absorbance spectra, the probe CK with incremental addition of carbonate ion, we observed the gradually decreased of 402 nm band and formation of new band at 454 nm band. Upon the high concentration of carbonate ion, 402 nm band was disappeared and 454 nm band was obtained, and moreover two isosbestic bands were appeared at 327 and 421 nm, these bands represent our probe CK and carbonate has strong interaction. (Fig. 1b).

In competitive experiments, the probe CK with the presence of carbonate and remaining other analyte shows that 454 nm band was appeared, this studies clearly shows that CK is highly selective carbonate sensor towards other anions (Fig. 2). Hereafter, we calculated the limit of detection is 7.2 nM from the absorbance titration spectra (Fig. S4). Finally, the absorbance response of CK to carbonate was analyzed by the linear plot (Fig. S5) of Benesi-Hildebrand equation: $1/(A - A_0) = 1/\{K (A_{max} - A_0) [CO_3^{2-}]\} + 1/[A_{max} - A_0]$, where A_0 is the absorbance register in the presence of added analyte, A_{max} is absorbance in the presence of added [analyte]_{max}, K is an binding constant. The binding constant of probe with carbonate is 22.8×10^{-2} M from the absorbance titration spectra.

In addition, we performed reversible experiment for our chemosensor **CK**. The notable change in the naked eye (colorless to yellow color) of **CK** has happened after the addition of CO_3^{2-} due the formation of $CK-CO_3^{2-}$ complex. Then, the absorbance wavelength retained to the original basal condition of **CK** when acetic acid was added to the solution of $CK-CO_3^{2-}$ complex, suggesting that the presence of acetic acid brings about the release of free CK and the formation of bicarbonate anion. The reversible interconversion of **CK** was repeated several times with acetic acid, demonstrating that **CK** can be developed as an excellent reversible chemosensor for the detection of carbonate anion (Fig. S6).

3.1. Emission spectra

In Emission spectra, CK shows weak emission at 513 nm in DD water, the 513 nm band represents the photo induced electron transfer from naphthalene imine moiety to the triazole moiety and C—N isomerism also presented, after that we added various anions such as CH₃COO⁻, N₃⁻, HCO₃⁻, SO₄²⁻, I⁻, SCN⁻, NO₃⁻, F⁻, S²⁻, HPO₄⁻, Cl⁻, Br⁻ emission spectra does not change, but after the addition of carbonate ion, the intensity of 513 nm band has increased and strong fluorescence was happened. The increasing intensity after addition of carbonate ion to probe is attributed due to the inhibition of PET process (Fig. 3). Then, interference studies were carried in the probe alone, probe with



Fig. 8. ¹H NMR titration spectra of CK alone and CK with difference concentration of carbonate ion.



Fig. 9. Optimized structure of (a) CK alone and (b) CK with carbonate ion.

carbonate, and probe with carbonate in presence of another analyte. From the competitive experiment results shows a characteristic intensity of 513 nm band does not have drastic changes indicated that CK selectively sense carbonate ion even though presence of other relevant anions (Fig. 4).

To explore the interaction of **CK** with carbonate, emission spectra variation of **CK** was investigated by using the titration of different concentration of carbonate. The probe 513 nm band intensity was gradually increased by the gradual addition of carbonate ion (Fig. 5). This emission titration spectra (Fig. S7) helps to calculate the limit of detection (LOD), we found the LOD of carbonate ion in emission spectra is 1.8 μ M. Benesi-Hildebrand equation are $1/I - I_{min} = 1/I_{max} - I_{min} + (1/K[C]) (1/I_{max} - I_{min})$ where I_{min} is the emission intensity of CK without carbonate, I is the emission intensity of **CK** at the concentration of carbonate, I_{max} is the emission intensity of **CK** at the concentration of complete saturation point, K is the binding constant, [C] is the concentration of **CK**. The binding constant value has been

determined from the slope of the plot $(I_{max}-I_{min}) \ / \ (I-I_{min}) \ vs \ 1/[C]$ for CK-CO₃^{2–} (Fig. S8). The binding constant of carbonate in CK is 8.3×10^{-2} M.

3.2. Mechanism of carbonate in CK

The sensing mechanism (Scheme 2) of CK with carbonate was confirmed by jobs plot, FTIR, NMR and DFT studies. Initially, we know the binding stoichiometric of probe with analyte by using the Job's plot analysis. Job's plot experiment has been accomplished using UV–visible and emission spectroscopy (Fig. 6). The Job's plot results shows that binding efficiency of receptor CK and carbonate is 2:1 binding stoichiometric. Our probe has hydroxyl group, so that two molecules of hydroxyl group in CK is interacted with one molecule of the carbonate ion, this is reason for 2:1 binding ratio of carbonate with probe CK

In FTIR spectra (Fig. 7) shows that **CK** has stretching frequencies 3434, 2932, 1735, 1608 and 1465 cm^{-1} has naphthalene substituted



Fig. 10. Frontier orbitals of (a) CK alone and (b) CK with carbonate ion.



Fig. 11. Photograph of colorimetric response of CK alone and CK with various anions, CH_3COO^- , SCN^- , Br^- , Cl^- , F^- , CO_3^{2-} , HCO_3^{--} , HPO_4^{2-} , NO_3^{--} , N_3^{--} , S^{2-} , I^- and SO_4^{2--} . (From left to right).



Fig. 12. Photograph of fluorometric response of **CK** alone and **CK** with various anions CH_3COO^- , SCN^- , Br^- , Cl^- , F^- , CO_3^{-2} , HCO_3^- , HPO_4^{-2} , NO_3^- , N_3^- , S^{-2} , I^- and SO_4^{-2} (From left to right).

O—H stretching, aromatic C-H stretching frequency of naphthalene ring, ester carbonyl C=O stretching frequency, imine C=N stretching frequency, aliphatic C-H stretching frequency respectively. After that interaction between the **CK** and sodium carbonate, shows that the probe **CK** stretching frequencies were present but in addition to that new broad stretching frequency from 2975 to 3458 cm⁻¹ is appeared due to the carbonate anion interact with hydroxyl proton of naphthalene ring so

that stretching frequency behaves like an acidic proton frequency and also carbonate carbonyl C=O stretching frequency was formed in the region of 1467 cm⁻¹, further more confirmation of new stretching frequency at 848 cm⁻¹ represented C=O stretching frequency of carbonate. The band 588 cm⁻¹ is due to the OH bending of acidic proton. These studies clearly shows that our naphthalene substituted hydroxyl proton acts as binding site for carbonate ion.

Table 1

Truth table for logic circuit.

S. No.	CK alone	${\rm CO_3}^{2-}$	$\begin{array}{l} Output \\ \lambda_{max} = 454 \ nm \end{array}$
1.	0	0	0
2.	0	1	0
3.	1	0	0
4.	1	1	1



Fig. 13. Molecular logic circuit.

In addition to that, the interaction between the probe and analyte was determined by proton NMR titration method. In the Fig. 8. shows that two singlet peaks in the aromatic region, highly downfield singlet represented as hydroxyl proton, another one singlet represented as imine proton, remaining other aromatic protons represents as the two moiety of naphthalene ring in the probe CK, after that aliphatic peaks in the highly upfield protons represented the cyclopropane ring protons, the aliphatic peak at 4.11 ppm represented the methylene proton and the 2.96 ppm peaks represented the methoxy proton. After that the addition of the 0.25 equiv. of carbonate ion in to CK probe, the probe hydroxyl proton peak intensity was decreased and at the same time remaining other aromatic and aliphatic peaks are upfield and furthermore, the addition of 0.25 equiv. of carbonate ion in to probe-analyte solution, in this case the hydroxyl proton peak was vanished and remaining other peak are slightly upfielded. NMR titration studies clearly shows that our probe hydroxyl proton interacts more with carbonate ions when compared to other protons of our probe. Thus, the binding stoichiometric 2:1 was confirmed by NMR titration studies.

Furthermore, our probe alone and probe with analyte were investigated by Gaussian 09 program with the help of the density functional theory B3LYP/6.31g (d, p) as basis set. The optimized structure of probe **CK** alone and **CK** with carbonate ion in the Fig. 9. For the probe **CK**, the electron density is mainly present in the cyclopropane substituted naphthalene moiety in the ground state but in the excited state electron density are transfer from cyclopropane substituted naphthalene to hydroxyl substituted naphthalene moiety, it's due to PET process. Hereafter, probe with analyte shows that the electron density present in the triazole and hydroxyl substituted naphthalene moiety in the HOMO state, but in the LUMO state electron density are move to the carbonate moiety, this transition clearly shows that carbonate ion inhibit the PET process from the triazole to hydroxyl naphthalene substituted moiety (Fig. 10).

3.3. Application

In naked eye study, the probe **CK** dissolved in aqueous medium, at that same time various anions such as CH_3COO^- , F^- , CI^- , Br^- , I^- , SCN^- , NO_3^- , N_3^- , HCO_3^- , SO_4^{2-} , S^{2-} , HPO_4^- and CO_3^{2-} are dissolved in water solvent. The addition of the various mono and divalent anions are added to the probe **CK**, no color changes was observed except carbonate anion addition. The probe **CK** has colorless solution when the addition of carbonate anion, color changes was observed from colorless to yellow color in the naked eye system (Fig. 11). The formation of 454 nm band is

the reason for colorless to yellow color appearance in the naked eye system. At the same time, probe with carbonate has enhance the emission in green color, but remaining others anions does not have emission under 365 nm UV chamber (Fig. 12).

3.4. Real sample analysis

More important study of the practical applicability of probe CK, real sample analyses were achieved to determine the concentration of carbonate ion in the four different water samples. The sample 1 is reverse osmosis treated water, sample 2 is without treatment reverse osmosis water (tap water) from the Madurai Kamaraj University, Madurai district, Tamil Nadu, India. The sample 3 is collected from Vaigai river, Madurai district, Tamilnadu, India. The sample 4 is collected from Thamirabarani at Thoothukudi district, Tamilnadu, India. The water samples were spiked with standard carbonate at various concentrations and determined by a standard addition method using the probe CK. We prepared the stock solution of carbonate ion as different concentration levels of 20 to 50 μ M. All the characteristics data are tabulated in the Table S1. The detection of carbonate ion concentration is very close to the concentration of added carbonate ions in the sample 1 and sample 2. but the sample 3 and 4 are slightly difference is due to the interfering of real ions. Even though, the average recovery of the spiked samples was found to be 100, 98, 112, 118 for RO, tap, and Vaigai, Thamirabarani water samples respectively. These results show that the probe CK has excellent selective and high sensitivity detection of carbonate ion in real samples with high accuracy.

3.5. Logic gate

The sensing performance of **CK** was also examined by logic circuit model. The presence of probe in the solution denoted as 1, suppose the absence of probe denoted as 0, like as the same for analyte in the inputs. Hence the output has 454 nm absorbance band, in the UV–Visible spectrum shows if probe alone does not form the 454 nm band, if analyte alone does not form the 454 nm band, the 454 nm will be appeared when the solution has both probe and carbonate ions so it is denoted as 1 and other remaining as 0 (Table 1). After that we make the molecular logic circuit is integrated with the help of various logic gates such as NOT, OR, EXOR, AND gates. Based on the developing truth table, **CK** found to entirely mimic the new logic gate circuit (Fig. 13) for the detection of carbonate ion.

4. Conclusion

In summary, we have established a new triazole and naphthalene hybrid-based sensor for naked eye detection of carbonate ion. The chemosensor **CK** exhibits an excellent selectivity and sensitivity towards carbonate ions over other anions by changes in both UV–Vis absorption spectra and emission spectra. The detection limit of carbonate ions is 7.2 nM from UV–Visible spectroscope and 1.8 μ M from emission spectroscopy, this is the lowest ever reported in the literature. Moreover, the plausible sensing mechanism were confirmed by various methods such Job's plot, FTIR, proton NMR titration, and theoretical studies. We can also apply our probe for the naked eye detection of carbonate ion in solution phase. Furthermore, the probe **CK** is efficaciously applied in the analysis of the environment samples for the rapid detection of carbonate ions. Atlast, we hope that the new probe **CK** may be beneficial biological sensor for carbonate ions.

CRediT authorship contribution statement

Harikrishnan Muniyasamy: Data curation, Validation. Chithiraikumar Chinnadurai: Conceptualization, Data curation. Malini Nelson: Validation, Writing – original draft. Muniyappan Chinnamadhaiyan: Software, Resources, Validation. Siva Ayyanar: Supervision, review and editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary material

Supplementary data to this article can be found online at https://doi.org/10.1016/j.inoche.2021.108883.

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