

X-Ray Crystal Structure and Cation Binding Properties of a New Phthalide-fused Indoline Derivative

(Struktur Hablur Sinar-X dan Sifat Pengikat Kation bagi Terbitan Ftalida-Indolin)

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ABSTRACT

An efficient and a green route to synthesize phthalide-fused indoline, 3-[(5-chloro-1,3,3-trimethylindolin-2-ylidene)methyl]isobenzofuran-1(3H)-one (3) has been developed by the coupling reaction of 5-chloro-2-methylene-1,3,3-trimethylindoline, 1 and 2-formylbenzoic acid, 2 under solvent-free domestic microwave irradiation. The compound was produced in 85% yield. Compound 3 was characterized by analytical and spectral methods and its structure was confirmed by chemical crystallography. Compound 3 was successfully crystallized in triclinic system with space group $P\bar{1}$. The molecular structure consists of fused 1 and 2 groups connected by the enamine carbon. Binding study of 3 towards different types of metal cations was done by colorimetric detection and UV-vis titrations. Compound 3 exhibited good selectivity and sensitivity for Sn^{2+} compared to other metal cations tested. The stoichiometric binding ratio of 3 toward Sn^{2+} is found to be 1:1 and the binding constant (K_a) is $1.07 \times 10^4 \text{ M}^{-1}$ on the basis of Job's plot and Benesi-Hildebrand analysis.

Keywords: Binding study; chemical sensor; green synthesis; Phthalide-indoline; Sn(II) ion

ABSTRAK

Satu kaedah sintesis hijau dan cekap bagi sintesis ftalida-indolin, 3-[(5-kloro-1,3,3-trimetilindolin-2-ylidin)metil]isobenzofuran-1(3H)-on (3), telah dibangunkan melalui tindak balas pengkupelan antara 5-kloro-2-metil-1,3,3-trimetilindolin, 1 dan asid 2-formilbenzoik, 2 menggunakan iradiasi gelombang mikro tanpa pelarut dengan peratusan 85%. Sebatian 3 telah dicirikan melalui kaedah analisis dan spektrum, serta strukturnya disahkan melalui kristalografi kimia. Hablur tunggal sebatian 3 telah berjaya dihasilkan dan mempunyai sistem hablur triklinik dengan kumpulan ruang $P\bar{1}$. Struktur molekul 3 adalah merupakan gabungan kumpulan 1 dan 2 yang berikat pada karbon enamina. Kajian pengikatan antara sebatian 3 dengan pelbagai kation logam telah dijalankan menggunakan kaedah kolorimetri dan kaedah pentitratan ultralembayung boleh nampak (UV-vis). Sebatian 3 menunjukkan kepilihan dan kepekaan yang baik terhadap ion Sn^{2+} berbanding dengan kation logam lain yang diuji. Nisbah stoikiometri pengikatan antara 3 dengan Sn^{2+} dan pemalar pengikatan (K_a) masing-masing adalah 1:1 dan $1.07 \times 10^4 \text{ M}^{-1}$ berdasarkan plot Job dan analisis Benesi-Hildebrand.

Kata kunci: Ftalida-indolin; ion Sn(II); kajian pengikatan; penderia kimia; sintesis hijau

INTRODUCTION

Study on selective and sensitive chemosensors are widely attractive because of their instantaneous response toward a certain analyte, high sensitivity with low limit of detection, accuracy and uncomplicated design as compared to the costly conventional method of detecting metal ions (Li & Xiao 2016; Qu et al. 2017). Heterocyclic molecule with ring opening ability is one of the important aspects in designing a highly selective and sensitive chemosensor (Chatterjee et al. 2016). Colorimetric chemosensor has also attracted much attention whereby a target analyte can be identified by 'naked eye' (Zhu et al. 2015). In a recent study on tin compounds in landfill leachates, Sn^{2+} ionic species exhibited bacterial methylation forming toxic methyltin which may be released into the environment (Peeters et al. 2014). Sn^{2+} and inorganic tin compounds have several unfavourable biological effects on human and moderate

toxicity to aquatic organisms and the environment (Howe & Watts 2005). Meanwhile in another study by Mathews et al. (2015), Sn(II) chloride was successfully used as a treatment system to remove mercury from wastewater, however, possible impacts of tin bioaccumulation toward human or ecological risk may be of concern. Thus, an *in situ* method of detecting and quantifying tin metal ions is essential in monitoring the environmental and biological systems.

Phthalides, also known as isobenzofuranones, are classified under lactones with its five-membered O-heterocyclic ring and is fused with benzene. 3-substituted phthalides are usually found in natural products and are well known for their wide range of biological application (Beck & Chou 2007; Karmakar et al. 2014). It is also one of the main classes in the production of commercial dyes (Bamfield 2010). Indoline, an aromatic N-heterocycle,

possess biological activities like antibacterial (Michael Barbour et al. 2014) and cytotoxicity against cancer cells (Azizian et al. 2012). Besides, it is also a building block of highly conjugated cyanine with good optical properties, great biocompatibility and low toxicity toward biosamples (Sun et al. 2016). These are the desirable properties of a chemosensor.

Much effort is being expended in synthesizing natural products containing the phthalide and N-heterocyclic moiety. Green route of synthesizing 3-substituted phthalides using the microwave irradiation was reported with better yield and efficiency (Landge et al. 2008; Srivastava et al. 2013). The use of microwave irradiation in reaction provides many advantages which include performing solventless reactions, reduction of time taken for a reaction, instantaneous and efficient heating and less waste production (Gangrade et al. 2015). This work will follow the green route of synthesis whereby reaction will be done in a solventless medium using the microwave irradiation method.

MATERIALS AND METHODS

Reaction was performed under microwave irradiation method using a domestic microwave oven (Electrolux, model EMM2017X, PCR). Chemicals and solvents were purchased from Sigma Aldrich and System and used directly without further purification. All UV-vis spectra were recorded on Shimadzu UV-1800 spectrophotometer using a quartz cell with a path length of 1 cm in 95% EtOH; FTIR spectrum was recorded on Perkin-Elmer Spectrum GX spectrophotometer in the range 400-4000 cm⁻¹ using KBr pellet method; ¹H and ¹³C NMR (in CDCl₃) spectra recorded on a Bruker Avance 400 MHz spectrometer at 400.2 MHz and at 100.6 MHz, respectively using TMS as internal standard; mass spectrum recorded on a Bruker micrOTOF-Q spectrometer by ESI-MS in the positive ion mode. Single crystal X-ray experiments were performed on Bruker D8 QUEST diffractometer with MoK α radiation.

MICROWAVE IRRADIATION METHOD

A mixture of 5-chloro-2-methylene-1,3,3-trimethylindoline, **1** (1 mmol) and 2-formylbenzoic acid, **2** (1 mmol) was added into a dry reaction vial. The mixture was then irradiated using a domestic microwave at 100 W for 1 min. The reaction was monitored by TLC (petroleum ether/ethyl acetate, 2:1). After reaction was completed, cold ethanol (2 mL) was added into the reaction mixture. Precipitate was formed upon sonication for 10 s. The reaction mixture was then evaporated to approximately half its original volume and the precipitate was filtered off. The crude product was crystallized by slow evaporation from acetone to obtain pure crystal plates.

3-[(5-Chloro-1,3,3-trimethylindolin-2-ylidene)methyl]isobenzofuran-1(3H)-one(**3**)

The compound was obtained as greenish-yellow crystals in 85% yield (0.29 g). UV (EtOH): λ_{\max} = 226, 290 nm. IR

ν_{\max} (KBr)/cm⁻¹: 3053 (C-H sp²), 2928, 2960 (C-H sp³), 1750 (C=O), 1649 (C=C), 1466, 1493, 1602 (aromatic C=C), 1139, 1287 (C-O), 1062, 1094, 1265 (C-N), 900. ¹H NMR (400 MHz; CDCl₃) δ 1.70 (6H, s, CH₃), 3.00 (3H, s, N-CH₃), 4.21 (1H, d, J = 10.8 Hz, CH-O), 6.51 (1H, d, J = 7.6 Hz, H_{Ar}), 6.60 (1H, d, J = 10.4 Hz, C=CH), 7.12 (2H, m, J = 8.0 Hz, H_{Ar}), 7.45 (1H, d, J = 6.8 Hz, H_{Ar}), 7.56 (1H, t, J = 6.8 Hz, 7.2 Hz, H_{Ar}), 7.70 (1H, t, J = 6.4 Hz, 7.2 Hz, H_{Ar}), 7.91 (1H, d, J = 7.2 Hz, H_{Ar}). ¹³C NMR (100 MHz, CDCl₃) 29.2, 29.9 (CH₃-C-CH₃), 45.2 (N-CH₃), 79.1 (C=CH), 88.7 (CH-O), 106.4 (CH_{Ar}), 122.1 (CH_{Ar}), 122.7 (CH_{Ar}), 124.3 (C_{Ar}-Cl), 125.4 (CH_{Ar}), 126.4 (C_{Ar}), 127.7 (CH_{Ar}), 129.1 (CH_{Ar}), 134.1 (CH_{Ar}), 139.7 (C_{Ar}), 143.8 (C_{Ar}), 150.9 (C_{Ar}), 160.9 (N-C=CH), 170.7 (C=O). ESI-MS m/z calcd for C₂₀H₁₈ClNO₂Na [M + Na]⁺: 362.0924. Found: 362.0921.

SAMPLE PREPARATION FOR BINDING STUDY WITH METAL CATIONS

For colorimetric detection of metal ions, stock solutions (1 \times 10⁻³ M) of **3** and metal ions (Ag⁺, Co²⁺, Cu²⁺, Fe²⁺, Hg²⁺, Ni²⁺, Sn²⁺ and Zn²⁺) were prepared in 95% EtOH, respectively. The color change of 1 mL of **3** (5 \times 10⁻⁴ M) was observed immediately after mixing with 1 mL of stock solution of respective metal ions. Metal ion screening was further analyzed by UV-vis absorption by diluting 50 mL of stock solution of **3** and 100 mL of stock solution of respective metal ions in a 5 mL volumetric flask with EtOH. The final concentrations of **3** and metal ions were 1 \times 10⁻⁵ M and 2 \times 10⁻⁵ M, respectively.

For UV-vis titration, stock solutions of **3** (1 \times 10⁻⁵ M) and SnCl₂ (1 \times 10⁻³ M) were prepared. 3 mL of **3** was taken directly into a quartz cuvette. The spectral measurements were recorded after each aliquot addition (10 mL) of the metal cation solution using a micropipette. All titration experiments were recorded at room temperature.

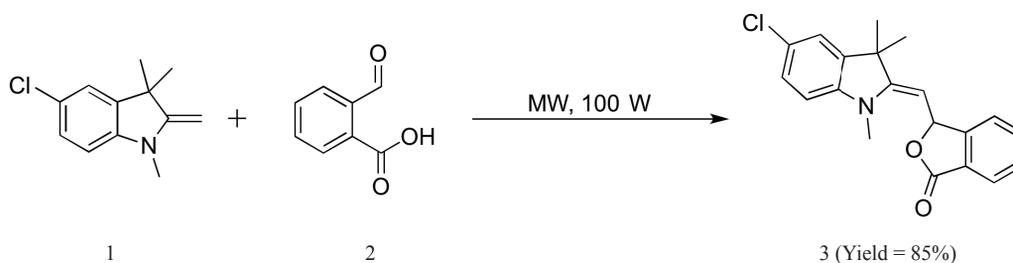
The stoichiometric ratio between **3** and Sn²⁺ ions was studied using Job's plot. Equimolar (1 \times 10⁻³ M) stock solutions of **3** and Sn²⁺ ions were prepared in 95% EtOH. Both the compound **3** and Sn²⁺ ions were mixed at different volumes (from 0 to 200 mL) in 10 mL volumetric flasks and was diluted with EtOH to make a total volume of 10 mL. Each volumetric flask had a total concentration of **3** and Sn²⁺ of 2 \times 10⁻⁵ M. The UV-vis spectra were taken at room temperature.

RESULTS AND DISCUSSION

SYNTHESIS AND CHARACTERIZATION

Compound **3** was synthesized from the reaction of a Fischer base and phthalaldehydic acid. The reaction scheme is shown in Scheme 1. Compound **3** was synthesized in a quantitative yield of 85% by microwave irradiation.

The UV spectrum of **3** in ethanol displayed absorption maximum at around 226 nm and 290 nm indicative of aromatic ring compounds due to $\pi \rightarrow \pi^*$ of benzene chromophores and $n \rightarrow \pi^*$ transition of carbonyl groups.



SCHEME 1. Preparation of a phthalide-fused indoline derivative under microwave irradiation

The FTIR spectrum showed characteristic C=O stretch of lactone ring at 1750 cm^{-1} (Figure 1). Bands between 1062 and 1287 cm^{-1} were assigned as C-O and C-N stretching.

^1H NMR spectrum (Figure 2) of compound **3** displayed 18 hydrogen integrals. According to report by Keum et al. (2011), the indoline group showed regular ^1H NMR resonance pattern for the protons on adjacent methyl

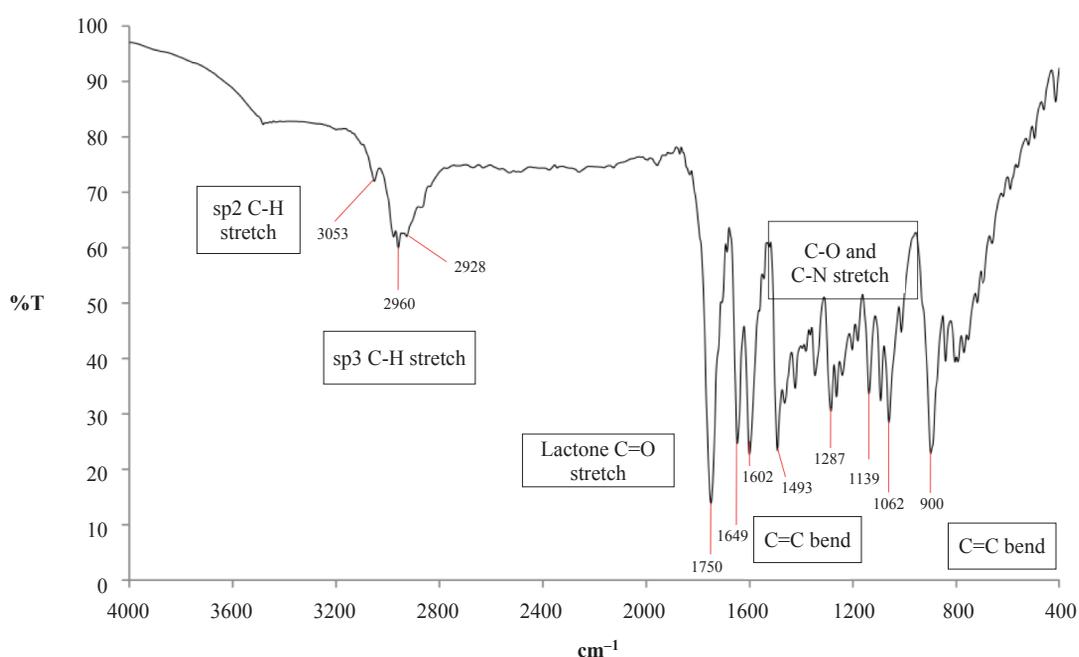


FIGURE 1. FTIR spectrum of **3**

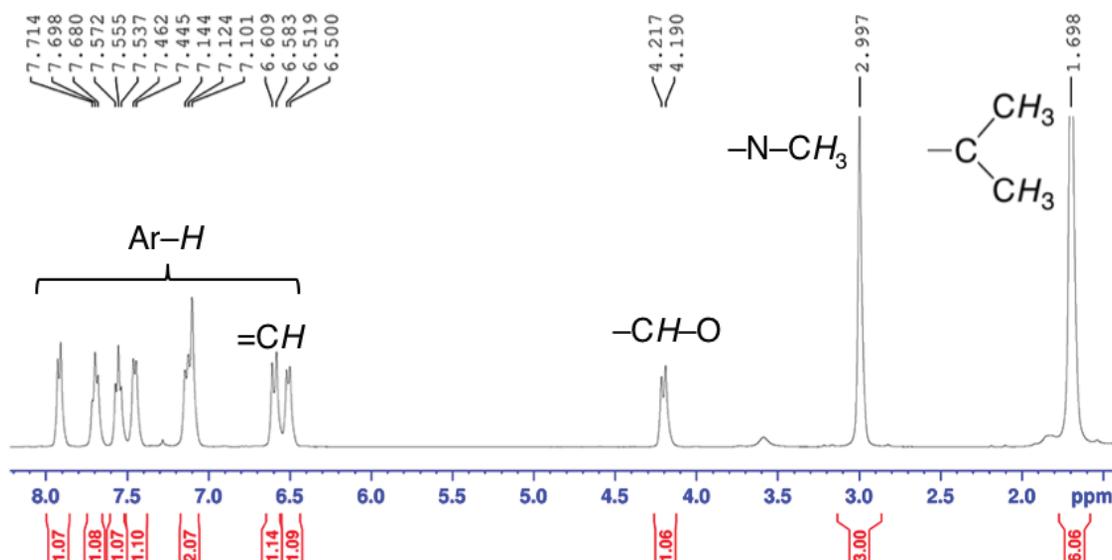


FIGURE 2. ^1H NMR of **3**

groups. The gem-dimethyl protons appeared as a singlet at 1.70 ppm, while N-methyl protons resonated at 3.00 ppm. A doublet at 4.20 ppm belongs to the chiral CH-O proton. The olefinic proton was detected as a multiplet at 6.60 ppm, with a J value of 10.4 Hz. Peaks between 6.5 and 8.0 ppm were resonated by aromatic protons. The ^{13}C NMR spectrum had 19 discrete carbon resonance signals and each peak was assigned by comparing to the spectrum of a phthalide-fused indoline derivative reported by Chunaev et al. (1982).

The molecular weight of $\text{C}_{20}\text{H}_{20}\text{NO}_2$ is 339.8190 g/mol. The ESI-MS spectrum showed an intense pseudomolecular ion peak at m/z 362.0921, $[\text{M} + \text{Na}]^+$.

X-RAY CRYSTALLOGRAPHY

Single crystal of **3** was subjected to X-ray crystallographic analyses to further confirm its chemical structure. The crystal was grown by slow evaporation of acetone solution at room temperature. Compound **3** crystallized in monoclinic system with space group $P1$. The crystal system and refinement parameters are shown in Table 1.

The molecule **3** consists of two fused 5-chloro-2-methylene-1,3,3-trimethylindoline and isobenzofuran-1(3*H*)-one groups connected by the enamine carbon C9 (Figure 3) with C8–C9 and C9–C10 of bond lengths 1.475 (3) and 1.335 (3) Å, respectively.

BINDING STUDY WITH METAL IONS

The colorimetric detection of mixture of compound **3** and metal salts were done qualitatively and observed by naked eyes. Color change was observed when colorless solution of compound **3** (5×10^{-4} M) was mixed with 2 equivalent of Fe^{2+} and Sn^{2+} , respectively (Figure 4). No color change was detected for all the other metal cations tested. The color of the mixture changed from colorless to greenish-yellow showed that compound **3** possessed ionochromic behavior with Fe^{2+} and Sn^{2+} .

The interaction between compound **3** and various metal ions was studied by UV-vis spectroscopy in EtOH solution. Figure 5 shows the results of metal ion screening with **3**. Solution of **3** was prepared at a concentration of 1×10^{-5} M. No absorption band was found in the visible region from the absorption spectrum of EtOH solution of **3** and is, therefore, colorless. The UV-vis spectra of **3** (1×10^{-5} M) was recorded upon addition of $\text{Ag}(\text{OAc})$, CoCl_2 , $\text{Cu}(\text{OAc})_2$, FeSO_4 , HgI_2 , $\text{Ni}(\text{OAc})_2$, SnCl_2 and $\text{Zn}(\text{OAc})_2$ (2 equiv) in 95% EtOH solution, respectively. New absorption band was seen with maximum absorbance at around 380 nm upon addition of Sn^{2+} . The absorption spectrum of **3** upon addition of Sn^{2+} also showed a drop in the absorbance intensity at around 290 nm, which may be an evidence of a host-guest complex interaction or binding. While upon the addition of other metal ions, no obvious change on the maximum absorbance or new band was detected. The absorbance intensity at 380 nm ($A_{380\text{nm}}$) over the absorbance intensity of blank solution of **3** with various metal ions is represented in a chart

TABLE 1. Crystallographic data collection parameters for **3**

Compound	3
Empirical formula	$\text{C}_{20}\text{H}_{18}\text{Cl}\text{N}\text{O}_2$
Formula weight	339.80
Temperature (K)	304 (2)
Wavelength (Å)	0.71073
Crystal system	Triclinic
Space group	
Unit cell dimensions (Å)	a = 8.178 (7) b = 10.163 (8) c = 11.995 (8)
α, β, γ (°)	$\alpha = 83.46$ (4) $\beta = 89.30$ (5) $\gamma = 69.53$ (5)
Volume (Å ³)	850.9 (12)
Z	2
Density (calculated) (Mg/m ³)	1.326
Absorption coefficient (mm ⁻¹)	0.236
F (000)	356
Crystal size (mm)	0.60 × 0.43 × 0.25
θ range (°)	3.009 – 28.288
Index ranges	$-10 \leq h \leq 10$ $-13 \leq k \leq 13$ $-14 \leq l \leq 14$
Reflections collected	39140
Independent reflections	4200 [R(int) = 0.072]
Completeness to theta	99.3%
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	4200 / 0 / 221
Goodness-of-fit on F^2	1.046
Final R indices [$I > 2\sigma(I)$]	R1 = 0.0559, wR2 = 0.1320
R indices (all data)	R1 = 0.0925, wR2 = 0.1577
Largest diff. peak and hole	0.225 and -0.314 e.Å^{-3}
CCDC No.	1574258

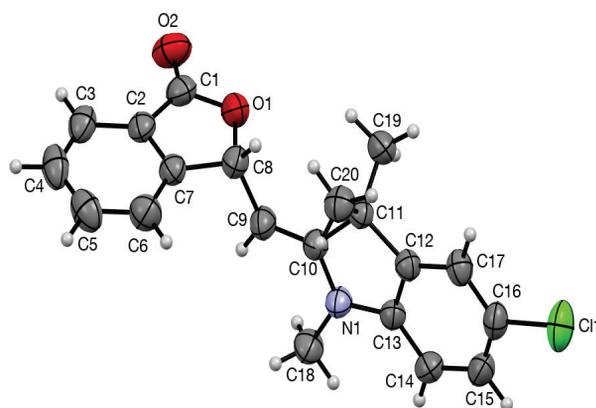


FIGURE 3. ORTEP diagram of **3** drawn at 50% probability ellipsoids

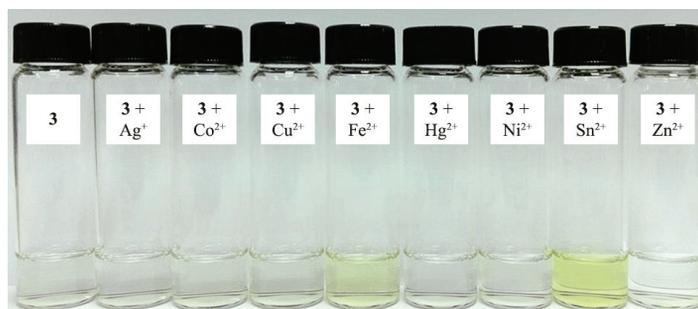


FIGURE 4. Colorimetric detection of **3** with metal salts of Ag(OAc), CoCl₂, Cu(OAc)₂, FeSO₄, HgI₂, Ni(OAc)₂, SnCl₂ and Zn(OAc)₂

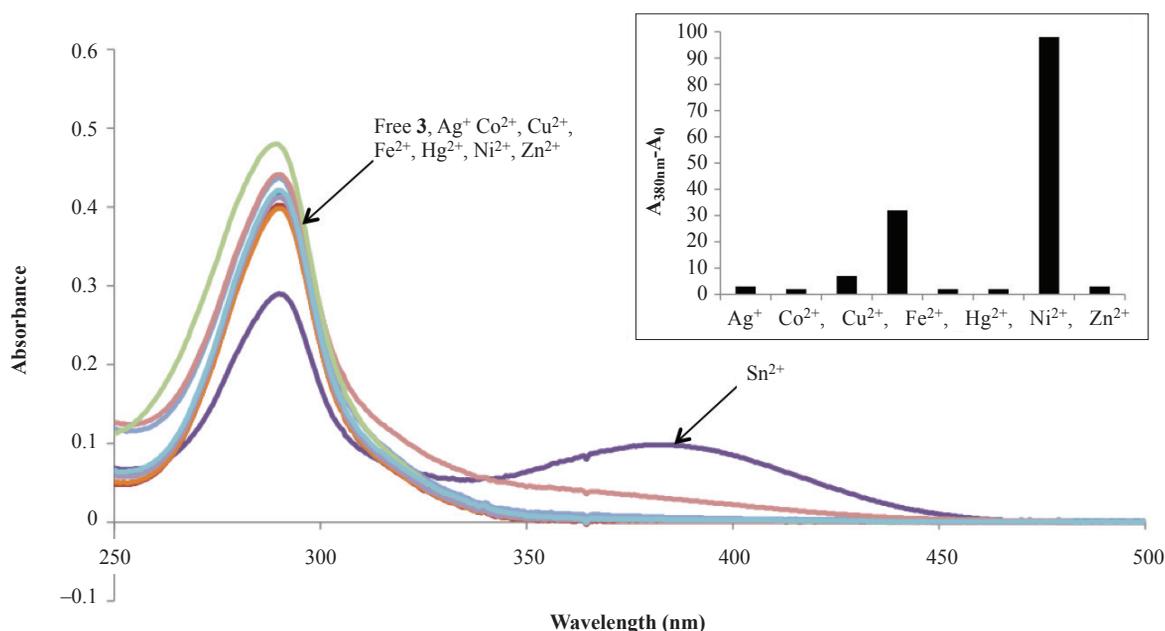


FIGURE 5. The absorption spectra of **3** upon addition of metal salts of Ag(OAc), CoCl₂, Cu(OAc)₂, FeSO₄, HgI₂, Ni(OAc)₂, SnCl₂ and Zn(OAc)₂ (2 equiv) in 95% EtOH solution. Inset: Absorbance intensity at 380 nm, $A_{380\text{nm}} - A_0$ over that of blank solution of **3**, A_0

(Figure 5). With the addition of Sn²⁺ ions, the absorbance of **3** showed a remarkable increase in intensity as compared to the other metal ions.

The absorption spectra of **3** upon titration with Sn²⁺ in 95% EtOH solution were recorded to further investigate the binding of **3** and Sn²⁺ ions. In the absence of Sn²⁺ ions, the free compound **3** remained colorless and did not exhibit any apparent absorption above 380 nm. When the Sn²⁺ ion solution was added into the solution of **3**, an increase in the absorption band at 380 nm was observed and the spectrum passed through two clear isobestic points at 261 and 315 nm (Figure 6). The increase in the absorbance and presence of the isobestic points are confirming the sensitivity of **3** towards Sn²⁺ ions in a solution and the formation of a new **3**-Sn complex in equilibrium with the free compound **3**.

Next, a Job's plot was generated by the continuous variation method to determine the stoichiometric ratio between **3** and Sn²⁺ ions. In this method, each experiment was carried out with different concentrations of both **3**

and Sn²⁺ ions while maintaining the total concentration at 20 mM. The plot was obtained by measuring the absorbance at 380 nm with mole fractions of Sn²⁺ from 0 to 1. The Job's plot analysis showed a maximum at ~0.5 mole fraction of Sn²⁺, indicating that the 1:1 stoichiometry was the possible binding mode of **3** with Sn²⁺ (Figure 7).

The stoichiometric binding was again evaluated using the Benesi-Hildebrand method. The plot of $1/(DA)$ against $1/[\text{Sn}^{2+}]$ from the UV-vis titration data showed a linear relationship implying that **3** associated with Sn²⁺ in a 1:1 stoichiometry (Figure 8). The value of association constant (K_a) of **3** with Sn²⁺ was calculated from the value of intercept/slope to be $1.07 \times 10^4 \text{ M}^{-1}$. This value is within the range 10^3 – 10^5 of those reported for Sn²⁺-binding chemosensors (Adhikari et al. 2016; Bao et al. 2014; Jadhav et al. 2017; Qu et al. 2017; Wang et al. 2015). Hence, we propose the binding mode of **3** with Sn²⁺ via the formation of dianion through the ring opening of lactone according to Bamfield (2010) (Scheme 2).

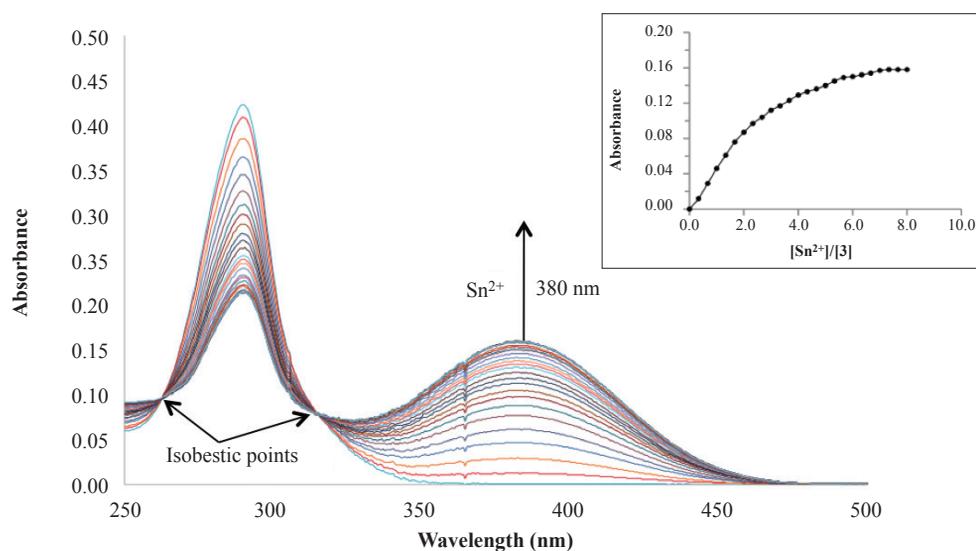


FIGURE 6. Change of absorption spectra of **3** (1×10^{-5} M) upon gradual addition of Sn^{2+} (1×10^{-3} M) ion concentration (10-250 mL) in 95% EtOH. Inset: Plot of UV-vis absorbance at 380 nm as a function of Sn^{2+} concentration

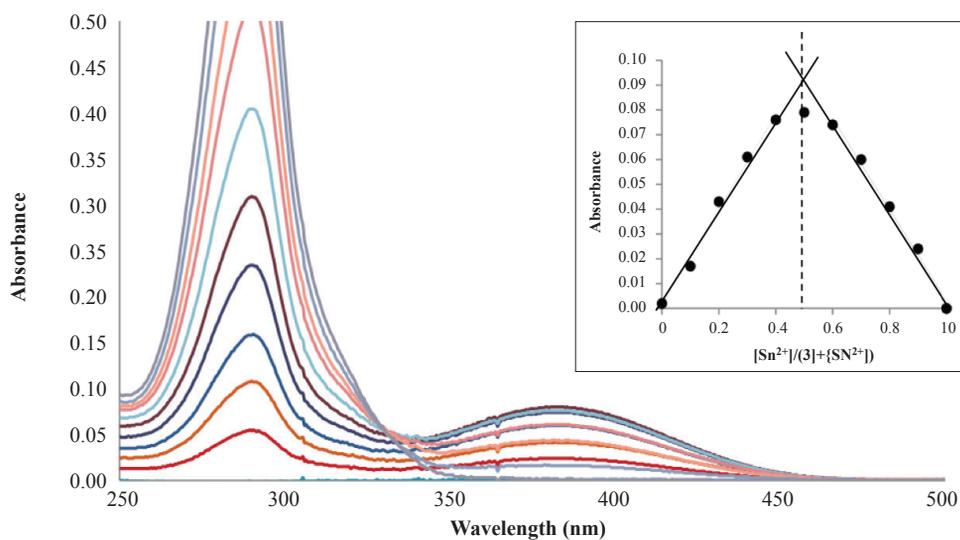


FIGURE 7. Job's plot of **3** and Sn^{2+} in 95% EtOH solution, with a total concentration of 20 mM ($[\mathbf{3}] + [\text{Sn}^{2+}]$) and a detection wavelength of 380 nm

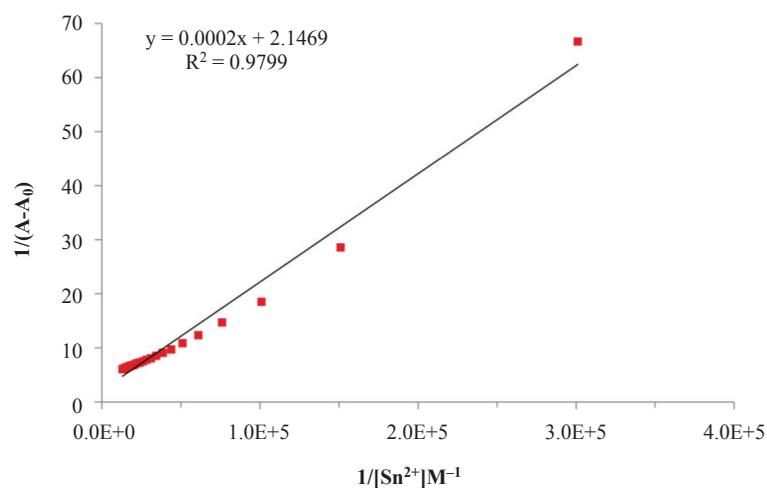
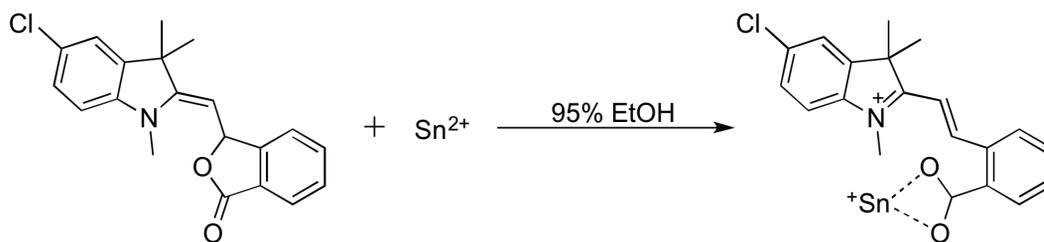


FIGURE 8. Benesi-Hildebrand plot of $1/(DA)$ versus $1/[\text{Sn}^{2+}]$ taken at 380 nm, assuming 1:1 stoichiometry for association between **3** and Sn^{2+}

SCHEME 2. Proposed binding mechanism of **3** with Sn^{2+} ion

CONCLUSION

A phthalide-fused indoline derivative, 3-[(5-chloro-1,3,3-trimethylindolin-2-ylidene)methyl]isobenzofuran-1(3H)-one (**3**), was successfully synthesized through a green route of synthesis using solventless microwave irradiation method which yielded 85% in a short amount of time (1 min). The compound was characterized by analytical and spectral methods and its structure was further confirmed by chemical crystallography. In conclusion, we report compound **3**, which is a selective and sensitive chemosensor for Sn^{2+} ion recognition in 95% EtOH solution by colorimetric detection and UV-vis spectroscopy. The 1:1 binding mode was proposed according to the Job's plot and Benesi-Hildebrand analysis.

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