



Intracellular application and logic gate behavior of a ‘turn off-on-off’ type probe for selective detection of Al^{3+} and F^- ions in pure aqueous medium

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ABSTRACT

A new Schiff base tcph, derived from 2-thiophene carboxylic acid hydrazide, has been synthesized and characterized by various spectroscopic techniques. The molecular structure of the compound has also been determined by X-ray crystallography. The tcph acts as a selective Al^{3+} and F^- induced OFF-ON-OFF type of probe in aqueous media. The 1:1 binding stoichiometry between probe and Al^{3+} has been established from Job's plot and further supported by ESI-MS studies. The limit of detection of Al^{3+} ions is determined by 3σ methods, which is found to be 1.35×10^{-9} M. The coordination environment for the tcph- Al^{3+} complex is delineated by NMR titration and DFT calculations. Detailed insights of probe-metal interaction mechanism have been studied by density functional theory (DFT) as well as time dependent-DFT calculations. MTT assay of the probe on live SiHa cells suggests no serious cytotoxicity in cells even at higher concentration. The probe tcph and its tcph- Al^{3+} complex have also been successfully applied to detect Al^{3+} and F^- ions in living cells (SiHa cells), respectively.

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1. Introduction

Fluorescent probes are found to be relevant in the context of sensing of biological and environmentally pertinent metal ions, *in vivo* and *in vitro* to clarify their useful as well as catastrophic effects to living beings [1–3]. Fluorescent imaging of living cells using these probes gave a good insight in understanding the role of metal cations as well as anions in physiological functions and related diseases [4–6]. Widespread use of aluminum in various domains exposes animals to prolonged inhalation of heavy concentration of aluminum dust, causing injuries to the pulmonary system [7], intoxication in hemodialysis [8], amyotrophic lateral sclerosis, osteomalacia, aberrance [9] and contributes to the risk of breast cancer. Aluminum is also responsible for neurofibrillary, enzymatic and neurotransmitter changes in the central nervous system that influence the formation of amyloid fibrils [10,11]. The high concentration of aluminum appears in acidified lakes deadly affects

aquatic lives [12]. Nevertheless, the acid rain increases free Al^{3+} in surface water by leaching from soil and inhibits plant growth by affecting root elongation [13–15]. Aluminum toxicity is the most important factor, being a major constraint for crop production on 67% of the total acid soil area [16]. Among anions, Fluoride ions are undoubtedly important due to their established beneficial role in dental care and clinical treatment for osteoporosis. Overexposure to Fluoride, on the other hand, can lead to acute gastric, fluorosis and urolithiasis [17,18].

Although a number of reports on Al^{3+} ion sensors have been exploited so far, however the interference caused by Fe^{3+} , Cu^{2+} and Zn^{2+} , non-aqueous or mixed aqueous solvents, tedious synthetic methods of preparation, and moderate detection limits are serious concern for most of the chemosensors [19–22]. So, a lot of effort is required to develop new efficient and economic probe with high selectivity and detailed understanding of the binding interactions of metals with receptor. Herein, we present an OFF-ON-OFF type water-soluble fluorescent sensor for the detection of $\text{Al}^{3+}/\text{F}^-$ ions in biological system. In the present work, a Schiff base probe, tcph, has been synthesized by the condensation reaction of thiophene-2-carboxylic acid hydrazide and 2-hydroxy propiophenone, and

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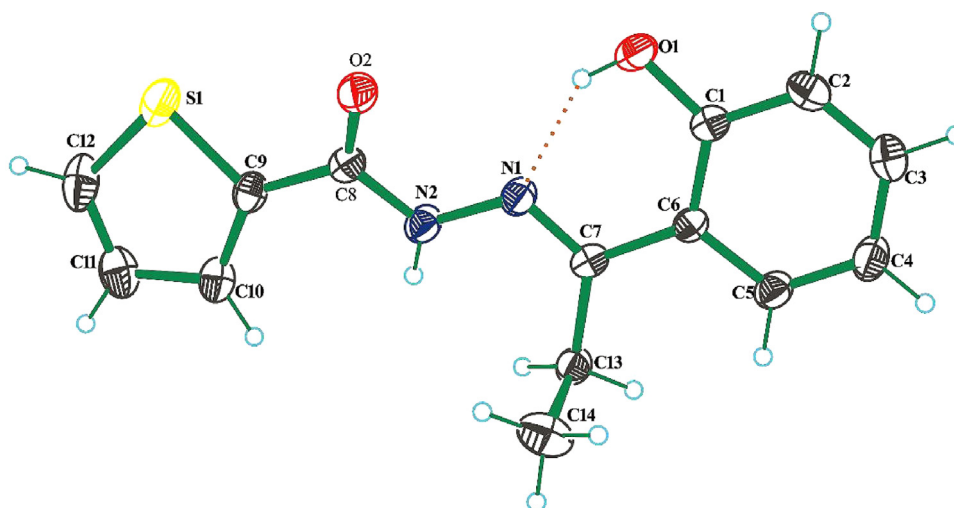


Fig. 1. ORTEP diagram of tcph showing intra-molecular H-bonding with ellipsoid of 30% probability.

Table 1
Crystallographic data for tcph.

Empirical Formula	C ₁₄ H ₁₄ N ₂ O ₂ S
Formula weight	274.33
Temp, K	293(2)
λ (Å)	0.71073
Crystal system	Orthorhombic
Space group	Pbca
a (Å)	13.5699(8)
b (Å)	7.7092(6)
c (Å)	25.576(2)
α (°)	90°
β (°)	90°
γ (°)	90°
V (Å ³)	2675.6(4)
Z	8
D _{calc} (mg/m ³)	1.362
μ (mm ⁻¹)	0.241
F(000)	1152
Crystal size (mm)	0.35 × 0.24 × 0.21
θ range for data collection (°)	3.40–29.21
No. of reflections collected	7873
No. of independent reflections (R _{int})	3075 (0.0490)
Number of data/restraints/parameters	3075/0/173
Goodness-of-fit on F ²	1.007
R ₁ , wR ₂ ^{a,b} [(I > 2 σ (I))]	0.0601, 0.1136
R ₁ , wR ₂ ^{a,b} (all data)	0.1349, 0.1430
Largest difference in peak and hole (e.Å ⁻³)	0.246 and –0.302

Selected bond length (Å) and angle (°) for tcph.

Bond lengths			
O(2)–C(8)	1.227(3)	C(6)–C(7)	1.475(3)
N(2)–C(8)	1.350(3)	C(8)–C(9)	1.477(4)
C(7)–N(1)	1.289(3)	C(1)–C(6)	1.406(4)
N(1)–N(2)	1.392(3)	C(7)–C(13)	1.497(3)
O(1)–C(1) O(1)–H–N(1)	1.354(3) 1.831	C(13)–C(14)	1.512(4)
Bond angles			
O(2)–C(8)–N(2)	122.3(2)	N(2)–N(1)–C(7)	119.0(2)
O(2)–C(8)–C(9)	122.6(2)	N(1)–C(7)–C(6)	115.3(2)
N(2)–C(8)–C(9)	115.1(2)	C(1)–C(6)–C(7)	122.5(2)
C(8)–N(2)–N(1)	117.0(2)	C(6)–C(7)–C(13)	120.9(2)
O(1)–C(1)–C(6)	122.9(2)	C(9)–S(1)–C(12)	91.54(17)

^a $R_1 = \sum ||F_o| - |F_c|| / \sum |F_o|$.

^b $R_2 = [\sum w(|F_o|^2 - |F_c|^2)^2 / \sum w|F_o|^2]^{1/2}$.

characterized by spectral analyses and X-ray crystallography. The small size of the probe, high water solubility and strong binding properties for Al³⁺ lead to high sensitivity and selectivity over the other metal ions. The probe exhibits fluorescence enhancement upon Al³⁺ complexation. In addition, the tcph–Al³⁺ complex could

also be used as cascade sensors for selectively detecting F[–] through a cation displacement approach. A very low detection limit of this probe for Al³⁺ (1.35×10^{-9} M) improves its prospects in environmental and intracellular analyses.

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