# Luminescent Lanthanide Complexes as Sensors for Organophosphorus Nerve Agents

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Luminescent europium complexes were synthesized and investigated as optical sensors for organophosphorus chemical warfare agents (nerve agents). The objective of this study is to develop a rapid "turn-off" detection method based on fluorescence quenching. Europium thenoyl-trifluoroacetonate complexes with and without co-dopants ( $Tb^{3+}$ ,  $Nd^{3+}$ ,  $Pr^{3+}$ ) were prepared and characterized by infrared spectroscopy, UV-vis absorption and photoluminescence emission. The Eu(III) tris(thenoyltrifluoroacetonate)-1,10-phenanthroline [Eu(TTA)<sub>3</sub>(phen)] complex exhibited intense red luminescence, which was selectively quenched in the presence of organophosphorus nerve agent simulants. In particular, dimethyl methylphosphonate (DMMP) and malathion (an organophosphate pesticide simulant) caused significant fluorescence quenching of Eu(TTA)<sub>3</sub>(phen). Photoluminescence (PL) spectra showed a progressive decrease in Eu<sup>3+</sup> emission intensity (especially the peak at 612 nm) with increasing analyte concentration, with complete quenching at around 1.2  $\mu$ M malathion. The lanthanide-based sensing mechanism is attributed to ligand displacement or energy transfer interactions between the nerve agent analogs and the Eu<sup>3+</sup> complex. These findings demonstrate a simple and fast optical method for detecting phosphorous nerve agent compounds and the results are compared with existing sensor technologies. The Eu<sup>3+</sup> complex sensor demonstrates potential for field-deployable detection of chemical warfare agents, with potential advantages in selectivity and real-time response.

Keywords: Luminescent, Europium complexes, Sensor, Organophosphorus detection.

# INTRODUCTION

Organophosphorus chemical warfare agents (OP CWAs), commonly known as nerve agents (*e.g.* sarin, VX), are potent acetylcholinesterase inhibitors that can cause incapacitation or death within minutes of exposure [1]. Because these agents are often odourless and colourless, fast and sensitive detection is critical for protecting military and civilian populations [2]. Traditional detection methods for nerve agents include chromatographic techniques (GC-MS) [3,4], immunoassays [5,6] and colorimetric tests [7,8], which can be highly sensitive but may require specialized equipment or reagents. In recent years, luminescent sensors have gained attention as a rapid and portable alternative for chemical warfare agent detection [9-11]. These methods often exploit supramolecular interactions or chemical reactivity of the agents to produce an optical signal change, such as fluorescence turn-on or turn-off.

Lanthanide complexes, particularly those of europium (Eu<sup>3+</sup>) and terbium (Tb<sup>3+</sup>), are attractive for luminescent sensing due to their unique photophysical properties [12]. Trivalent

lanthanide ions exhibit narrow line-like emission bands and long luminescence lifetimes originating from f-f electronic transitions [13]. However, direct f-f absorption is weak (Laporteforbidden), so luminescent lanthanide sensors typically employ an "antenna" ligand that strongly absorbs UV light and transfers energy to the lanthanide center [12,14]. For example,  $\beta$ -diketone ligands like thenoyltrifluoroacetone (HTTA) and heterocyclic chelators like 1,10-phenanthroline (phen) are well-known sensitizers for Eu³+ and Tb³+ luminescence [15,16]. The resulting complexes can emit bright fluorescence (e.g., Eu³+ typically emits red at ~612 nm, Tb³+ emits green at ~545 nm) when excited by near-UV light.

Importantly, lanthanide luminescence can be modulated by the chemical environment [17]. If a molecule (such as nerve agent or simulant) interacts with the complex, for instance, by binding to the metal center or displacing a coordinated ligand—it can disrupt the energy transfer process and quench the luminescence. Moreover, lanthanide-based sensors can offer advantages such as large Stokes shifts (minimal background interference) and potential for time-gated detection to eliminate autofluore-

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**Characterization:** Infrared spectra were recorded on a

scence [18]. Recent reviews highlight numerous fluorescent and luminescent probes developed for detecting chemical warfare agents [19,20]. Among these, Eu(III) complexes have shown high selectivity for organophosphates as some designs exhibit a luminescence "turn-on" when the Eu<sup>3+</sup> binds an organophosphate, while others show luminescence quenching upon exposure to the toxin [21,22].

In this work, we focus on europium thenoyltrifluoroacetonate complexes and their response to organophosphorus nerve agent simulants. We report the synthesis of Eu(TTA)<sub>3</sub>(H<sub>2</sub>O)<sub>2</sub> and Eu(TTA)<sub>3</sub>(phen) complexes (where TTA = 2-thenoyltrifluoroacetone, phen = 1,10-phenanthroline), as well as Eu(III) complexes co-doped with other lanthanides (Tb<sup>3+</sup>, Nd<sup>3+</sup>, Pr<sup>3+</sup>). The luminescence properties of these complexes were characterized and their sensing performance for nerve agent analogs (including DMMP-a sarin simulant and malathion-an organophosphate pesticide) was evaluated via fluorescence quenching. The results are compared with literature reports on lanthanidebased chemical warfare agents (CWAs) detection, demonstrating the potential and limitations of this luminescent sensing approach.

## **EXPERIMENTAL**

All chemicals and solvents were purchased from Sigma-Aldrich (USA), unless otherwise stated. Europium(III) chloride hexahydrate (EuCl<sub>3</sub>·6H<sub>2</sub>O), while terbium(III) chloride hexahydrate (TbCl<sub>3</sub>·6H<sub>2</sub>O), neodymium(III) chloride hexahydrate (NdCl<sub>3</sub>·6H<sub>2</sub>O) and praseodymium(III) nitrate (Pr(NO<sub>3</sub>)<sub>3</sub>·6H<sub>2</sub>O) were used for co-doped complex synthesis. Ligands 2-thenoyltrifluoroacetone (HTTA) and 1,10-phenanthroline (phen) were also obtained from Sigma-Aldrich, USA. Analytical-grade solvents, including ethanol, methanol, acetonitrile and acetone, were purchased from TCI (Japan). The organophosphorus simulants used in sensing experiments were dimethyl methylphosphonate (DMMP), a sarin analog and malathion, a VX/soman simulant in liquid form.

Synthesis of europium(III) complexes: The Eu(III)  $\beta$ diketonate complexes were synthesized following modified literature procedures [23].

 $Eu(TTA)_3(H_2O)_2$ : Europium(III) chloride (1 mmol) was dissolved in ~10 mL ethanol and HTTA ligand (3 mmol) was added. A slight molar excess of triethylamine was then introduced to deprotonate HTTA, precipitating the Eu(TTA)<sub>3</sub> complex. The mixture was refluxed for 2 h and then cooled. The resulting Eu(TTA)<sub>3</sub>(H<sub>2</sub>O)<sub>2</sub> complex was collected by filtration, washed with water and ethanol and dried.

**Eu(TTA)<sub>3</sub>(phen):** The above Eu(TTA)<sub>3</sub>( $H_2O$ )<sub>2</sub> (1 mmol) was suspended in ethanol and mixed with 1,10-phenanthroline (1 mmol). The mixture was heated under reflux for 3 h, during which phenanthroline replaced the coordinated water, yielding Eu(TTA)<sub>3</sub>(phen). The product was filtered off and dried, resulting in a light-red powder. Co-doped Eu/Ln complexes of the type  $Eu_xLn_{1-x}(TTA)_3$ (phen) were prepared by simultaneously adding Eu<sup>3+</sup> and another lanthanide (Tb<sup>3+</sup>, Nd<sup>3+</sup> or Pr<sup>3+</sup>) salts in the desired molar ratios (for example, x = 0.5 for Eu<sub>0.5</sub>Tb<sub>0.5</sub>) to the HTTA reaction, followed by the same steps as above. The co-doped complexes were obtained as crystalline powders ranging in colour from yellow to orange. All the complexes were stored in the dark to prevent photodegradation.

FT-IR spectrometer (using KBr pellet). UV-Visible absorption spectra were measured with a UV-Vis spectrophotometer (range 200-800 nm). Photoluminescence (PL) emission spectra were recorded on a fluorescence spectrometer. Excitation was typically set at 365-385 nm (matching the absorption of the ligands). Emission was collected in the 500-750 nm range to capture the characteristic Eu<sup>3+</sup> f-f emissions. The emission intensities were corrected for instrumental response and compared across samples. Elemental analysis by energy-dispersive X-ray spectroscopy (EDX) was performed on selected samples to verify the presence of Eu and other dopant metals in the complexes.

Luminescence quenching experiments: The sensing behaviour of the Eu(III) complexes toward organophosphorus compounds was evaluated in both solution and solid-supported formats. For solution-phase tests, a stock solution of Eu(TTA)<sub>3</sub>-(phen) was prepared (0.01 g of complex dissolved in 10 mL of a 1:1 mixture of MeOH/MeCN). This corresponds to an approximate complex concentration of ~10<sup>-4</sup> M. The stock was further diluted to obtain working solutions (dilution factors 10× and 100×) to study concentration effects on luminescence. Aliquots of these solutions (without analyte) were placed in quartz cuvettes and excited with UV light (366 nm) to observe initial luminescence. Then, small volumes of a DMMP solution or malathion solution were added incrementally to the cuvette and the fluorescence spectra were recorded after each addition. For DMMP (a volatile liquid), an MeCN solution was prepared, whereas for malathion (70% technical grade in acetone), dilutions were prepared in acetone. Typical additions ranged from 150 μL to 300 μL of malathion solution into ~2 mL of Eu(III) complex solution, yielding final analyte concentrations in the low micromolar range. The emission intensity at 612 nm was monitored as a function of added analyte.

**Preparation of paper-based testing:** For solid-supported tests, filter paper strips were impregnated with the Eu(III) complex to simulate a portable test strip. Whatman filter papers were soaked in the Eu(TTA)<sub>3</sub>(phen) solution (0.01 g/10 mL in MeCN), then dried under ambient conditions to leave a thin luminescent film of the complex on paper. These papers showed a bright red fluorescence under a UV lamp (365 nm) in a dark box. To test detection, a small drop of the organophosphate simulant was placed on the luminescent paper and allowed to dry for ~30 sec. The paper was then observed under the UV lamp. Changes in the fluorescence (such as quenching or colour change) at the spot of analyte application were noted. For a more quantitative analysis, paper samples after exposure were extracted with a small amount of MeCN and the extract's fluorescence spectrum was measured to assess the remaining Eu emission.

## RESULTS AND DISCUSSION

To confirm ligand coordination, the key IR bands included the C=O stretch of the β-diketonate in the free HTTA ligand appears around 1625 cm<sup>-1</sup>, which is found to be shifted to ~1590 cm<sup>-1</sup> in the Eu(TTA)<sub>3</sub> complexes due to coordination. The disappearance of the broad O-H band of water in Eu(TTA)<sub>3</sub>(phen) confirmed replacement of H<sub>2</sub>O by phenanthroline. The Eu1348 Vu et al. Asian J. Chem.

(TTA)<sub>3</sub> complexes showed strong absorption in the near-UV (around 340-380 nm) attributed to the  $\pi$ - $\pi$ \* transitions of the aromatic thenoyl ligand and phenanthroline.

Luminescence properties of Eu(III) complexes: The synthesized europium(III) complexes were obtained in good yield as stable powders. The Eu(TTA)<sub>3</sub>(phen) complex, in particular, displayed a strong red luminescence under UV excitation. The emission spectrum of Eu(TTA)<sub>3</sub>(phen) in acetonitrile solution (excited at 385 nm) shows the characteristic Eu<sup>3+</sup> emission peaks at approximately 580 nm, 592 nm, 612 nm, 652 nm and 702 nm, corresponding to transitions from the excited <sup>5</sup>D<sub>0</sub> state to  $^{7}F_{J}$  levels (J = 0, 1, 2, 3, 4, respectively) (Fig. 1). The dominant peak is the electric-dipole  ${}^5D_0 \rightarrow {}^7F_2$  transition at 612 nm (red emission), which is hypersensitive to the ligand field symmetry. The fact that  ${}^5D_0 \rightarrow {}^7F_2$  is much stronger than the  ${}^5D_0 \rightarrow {}^7F_1$  magnetic-dipole transition (592 nm) indicates that the Eu<sup>3+</sup> ion resides in a low-symmetry coordination environment lacking an inversion center – consistent with bidentate TTA and heterocyclic phenanthroline ligands coordinating asymmetrically around Eu. The emission colour was a pure red and no green emission from Tb<sup>3+</sup> was observed in the Eu(TTA)<sub>3</sub>(phen) sample (which contained only Eu).

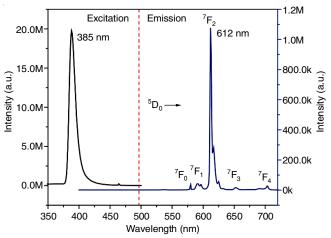


Fig. 1. Excitation spectrum (black line,  $\lambda_{em}=612$  nm) and emission spectrum (blue line,  $\lambda_{ex}=385$  nm) of the Eu(TTA)<sub>3</sub>Phen complex in acetonitrile solution

We explored co-doping Eu with other lanthanides to tune the luminescence properties. Doping with Tb3+ in mixed complexes Eu<sub>x</sub>Tb<sub>1-x</sub>(TTA)<sub>3</sub>(phen) yielded materials that still showed primarily Eu<sup>3+</sup> red emission under 365 nm excitation. For example, a sample with x = 0.5 (Eu<sub>0.5</sub>Tb<sub>0.5</sub>(TTA)<sub>3</sub>phen) exhibited the same Eu emission peaks, though with lower overall intensity than the pure Eu complex. Pure Eu(TTA)<sub>3</sub>(phen) had the highest luminescence intensity, while introducing Tb<sup>3+</sup>, Nd<sup>3+</sup> or Pr<sup>3+</sup> generally decreased the Eu-centered emission intensity (Fig. 2). In particular, heavy doping with Nd<sup>3+</sup> or Pr<sup>3+</sup> led to drastic quenching of Eu emission (e.g. Eu<sub>0.1</sub>Pr<sub>0.9</sub> complex showed only ~0.5% of the Eu(TTA)<sub>3</sub>(phen) intensity), likely because Nd3+ and Pr3+ centers can non-radiatively dissipate the excitation energy. Small amounts of Tb<sup>3+</sup> (e.g., Eu<sub>0.9</sub>Tb<sub>0.1</sub>) had a more moderate effect, but still the brightest emitter was the undoped Eu(TTA)<sub>3</sub>(phen) complex. These results indicate

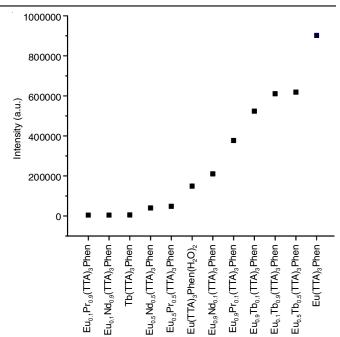


Fig. 2. Fluorescence intensities at 612 nm ( $\lambda_{ex}$  = 385 nm) of various Eu complexes co-doped with other lanthanides

that for maximum sensor brightness, the Eu complex alone is preferable, whereas adding other lanthanides can introduce competitive deactivation pathways or simply dilute the number of Eu<sup>3+</sup> emitters.

Fluorescence quenching by organophosphorus agents: Both solution and paper-based tests showed a clear quenching of Eu<sup>3+</sup> luminescence upon exposure to organophosphorus compounds. Visually, when a drop of DMMP solution was added onto the luminescent Eu-complex coated paper, the red glow was immediately diminished at that spot under UV light. Similarly, a drop of malathion (0.1% in acetone) caused the red emission on the paper to fade or extinguish (Fig. 3). This "turn-off" response is presumably due to the binding of the organophosphate to the Eu center or close interaction that disrupts the energy transfer from the ligands. Malathion, being a larger organophosphate with P=S (thionophosphate), can coordinate or interact with Eu<sup>3+</sup> and may even form a non-luminescent adduct, thereby quenching the fluorescence. DMMP, which is smaller and less strongly coordinating (a neutral phosphonate), also caused quenching but required a higher concentration or did not quench as completely as malathion under similar conditions. This aligns with literature observations that certain simulants (like DMMP) cause only mild dynamic quenching, needing a large excess to significantly reduce luminescence, whereas more strongly binding organophosphates can quench efficiently at lower levels.

Quantitative PL measurements in solution confirmed the quenching effect. The emission spectra of Eu(TTA)<sub>3</sub>(phen) in acetonitrile exhibit a significant decrease in fluorescence intensity with increasing amounts of malathion solution (Fig. 4). The initial Eu<sup>3+</sup> emission (no analyte) is intense; upon adding 0.70  $\mu$ M malathion, a significant decrease in luminescence is observed and with each incremental increase in malathion concentration, the emission peaks (580-702 nm) diminish

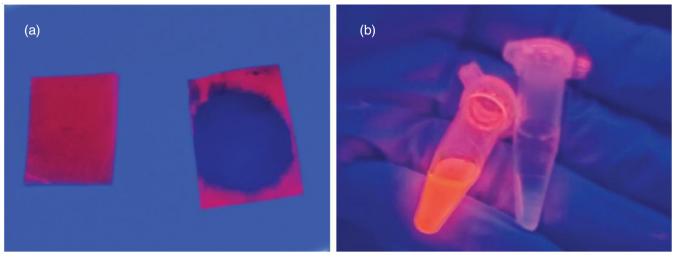


Fig. 3. Quenching effect of malathion (0.1% in acetone) in both (a) paper-based test and (b) solution

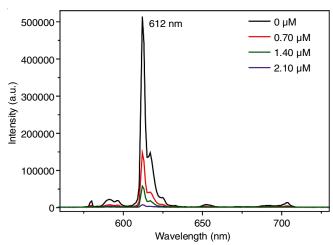


Fig. 4. Emission spectra of Eu(TTA)<sub>3</sub>Phen complex showing fluorescence quenching with increasing concentrations of malathion

further. At the highest tested malathion level (~2.10 µM), the Eu emission was nearly completely quenched. The quenching mechanism is likely a combination of static and dynamic quenching. A static quenching would involve the formation of a Eu-(organophosphate) adduct that is non-emissive, effectively sequestering Eu<sup>3+</sup> from luminescing. A dynamic (collisional) quenching could also occur, especially in solution, where the excited Eu complex transfers energy to the quencher during its lifetime. Prior studies have noted that nerve agents with P=O (oxon) groups, like G-series agents, often cause dynamic quenching unless they have additional binding functionality, whereas P=S agents or those with good ligating ability cause static quenching by directly coordinating to the Eu center [24,25]. In our experiment, malathion (which has a P=S and multiple coordinating sites) likely binds Eu<sup>3+</sup>, displacing the antenna ligand or saturating the coordination sphere, resulting in static quenching (complete loss of Eu emission at high concentration). This reasoning is supported by the observation that quenching by malathion was not reversible upon diluting the solution or waiting (Eu emission did not recover after adding an excess of malathion).

Comparison with existing methods: The detection sensitivity achieved here (micromolar range for malathion in solution, visible quenching of a drop of ~millimolar-level simulant on paper) is comparable to other simple luminescencebased nerve agent sensors in literature. Dennison & Johnston [2] reported that a Eu/Tb-phenanthroline complex could detect V-type nerve agents (VX, VG) at sub-ppb levels with immediate response, but required much higher concentrations (>350 ppm) of the G-agent simulant DMMP for a similar quench, indicating the variability in sensor response to different agents. The Eu(TTA)<sub>3</sub>(phen) system shows a stronger response to malathion (P=S analog) than to DMMP (P=O analog), which is consistent with those findings. Compared to traditional analytical instruments (GC-MS can detect nerve agents at ppb or lower and enzyme-based kits detect down to nM levels), the lanthanide luminescent sensor is less sensitive in absolute terms. However, the advantages of the Eu-complex sensor are its simplicity and immediacy, thus, the presence of an organophosphate can be discerned by the naked eye under a UV lamp, without any reagents or power-intensive devices. Additionally, the sensor can be deployed on paper strips or in transparent matrices, making it suitable for on-site field testing. Recent developments in fluorescent probes for CWAs include sophisticated molecules that undergo reaction with the agent to yield a fluorescent product or polymers and MOF-based materials that signal the presence of nerve agents. In comparison, the present approach uses a non-covalent interaction with a preformed luminescent complex, which means the response is fast (immediate quenching upon contact) and the material can, in principle, be regenerated if the quencher is removed (though complete restoration may require removing bound analyte).

Future improvements to this system could involve incorporating the Eu(TTA)<sub>3</sub>(phen) complex into a solid matrix (such as a polymer film or metal-organic framework) to enhance the stability and allow sensing of vapour-phase agents. In fact, lanthanideorganic frameworks have been studied for gas-phase detection of organophosphorus compounds, taking advantage of porosity and luminescent signal amplification. Another approach is to functionalize the ligands to increase affinity for specific agents – for 1350 Vu et al. Asian J. Chem.

example, attaching an oxime or hydroxyl group that can hydrogenbond with phosphonate esters. Such modifications might yield a "turn-on" sensor, where the Eu(III) complex is initially non-emissive until the target binds and triggers luminescence, providing an unambiguous positive signal. Overall, the current results establish a foundation that Eu(TTA)<sub>3</sub>-based complexes can serve as convenient turn-off probes for nerve agent simulants and they invite further development to improve the sensitivity and selectivity.

#### Conclusion

In summary, the luminescent lanthanide complexes for the detection of organophosphorus nerve agent analogs have been synthesized and evaluated as optical sensors. Europium thenoyltrifluoroacetonate containing phenanthroline ligand [Eu(TTA)<sub>3</sub>(phen)] was synthesized and found to exhibit strong red fluorescence under UV excitation. This emission was quenched upon exposure to representative organophosphates (DMMP and malathion), enabling a turn-off sensing mechanism for nerve agent simulants. Through spectral analysis, the quenching effect of malathion was quantified, revealing complete luminescence suppression at 1 µM concentrations. The quenching is attributed to the interaction of the organophosphate with the Eu(III) complex, likely through combined static binding and dynamic energy transfer processes. Co-doping Eu(III) complexes with Tb<sup>3+</sup>, Nd<sup>3+</sup> or Pr<sup>3+</sup> was explored; however, the highest luminescence intensity (best sensor signal) was obtained with the undiluted Eu(TTA)<sub>3</sub>(phen) complex. The practical implications of this research lie in the development of simple fluorescent test strips or solutions that can quickly indicate the presence of nerve agents without advanced instrumentation. The Eu(III) complex on filter paper showed a clear visual response to microliter droplets of simulant under a handheld UV lamp, demonstrating the feasibility of field-applicable detection. While the sensitivity is currently on the order of micromolar (which is sufficient for high doses but lower than advanced lab detectors), there is potential to improve this by structural optimizations or using time-resolved luminescence techniques to reduce background interference. The use of lanthanide luminescence for chemical warfare agents (CWAs) sensing is still an emerging area and this work contributes to that growing field by validating a particular Eu(III) complex system. Future work should focus on broadening the range of detectable agents (including G- and V-series nerve agents or their surrogates) and enhancing sensitivity. Incorporating the luminescent complex into nanofiber mats or polymer films could allow detection of vapour-phase agents at lower concentrations by pre-concentrating the analyte. Additionally, performing a mechanistic investigation (e.g. via NMR or lifetime measurements) would clarify the quenching interaction, guiding the design of turn-on sensors that respond via specific binding. With further development, lanthanidebased luminescent sensors could become valuable tools for rapid screening of chemical warfare agents, complementing existing detection technologies with a robust and easy-to-use method.

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## **CONFLICT OF INTEREST**

The authors declare that there is no conflict of interests regarding the publication of this article.

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