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## A colorimetric/luminescent benzene compounds sensor based on bis( $\sigma$ -acetylide) platinum(II) complex: enhancing selectivity and reversibility through dual-recognition sites strategy

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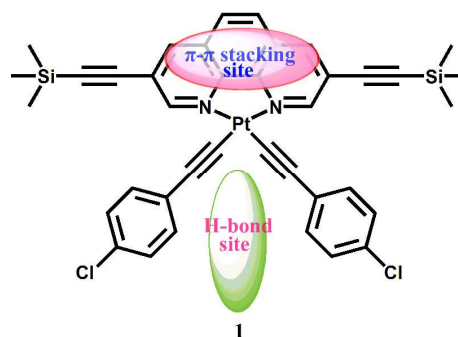
**A square-planar bis( $\sigma$ -acetylide) Pt(II) complex containing dual-recognition sites was designed, synthesized and used as colorimetric/luminescent sensor for detecting vapor of benzene compounds.**

Benzene and its homologues are a class of highly volatile and harmful materials that can cause many human diseases, such as acute and chronic lymphocytic leukaemia, non-Hodgkin's lymphoma and multiple myeloma through inhalation or dermal absorption.<sup>1</sup> The detection of benzene compounds vapors in environment, indoor residential, and workplace is very important. Traditional methods to detect these harmful vapors usually involve the using of expensive and complicated equipments, such as gas chromatography,<sup>2</sup> sensor devices based on fiber-optic devices,<sup>3</sup> diode laser IR absorption spectroscopy,<sup>4</sup> cross-reactive array electronic-nose sensing,<sup>5</sup> and metal oxide semiconductor films.<sup>6</sup> Currently the development of convenient and high performance methods for detecting these compounds is still a great challenge.

Recently vapor sensor based on luminescence materials were received more and more attention.<sup>7</sup> This kind of sensors display concurrent, selective, reversible and naked-eye perceivable luminescence change upon exposure to specific volatile organic compounds and have been proved to be one of the effective, cheap and convenient methods in detection of harmful vapors.<sup>8</sup> Although the research on it has achieved great progress, significant challenge still exists in detection of benzene compounds. So far, only a few examples based on Ru(II),<sup>9</sup> Zn(II),<sup>10</sup> Au(I),<sup>11</sup> Ag(I),<sup>12</sup> Pt(II)<sup>13</sup> and Cu(I)<sup>14</sup> complexes have been reported that could detect vapors of benzene compounds. However, in most cases, benzene compounds molecule just lies in the crystal voids instead of forming effective interaction with the chromophore which results in bad selectivity, low sensitivity and/or reversibility of the sensors. Consequently, the rational design of a chromophore exhibiting stronger interactions

with guest molecules is crucial for the preparation of benzene compounds sensor with better performance.

Since the pioneering work of Che et al about diimine platinum(II) bis( $\sigma$ -acetylide) complexes<sup>15</sup> and their tunable luminescence properties triggered by VOCs, the complexes and their derivatives have been successfully used in detection of various VOC molecules through strong hydrogen bond interaction by their H-bond recognition site (locates between the two acetylene auxiliary ligands), resulting in different packing structures and intermolecular Pt-Pt interaction which eventually lead to dramatic changes in color and/or emission.<sup>8d, 16</sup> However, the solely H-bond recognition site present in almost all of the complexes prevent them to recognize and detect aromatic compounds efficiently owing to the lack of strong  $\pi$ - $\pi$  stacking interactions which usually involve in the recognition of such VOCs. Thus the introduction of  $\pi$ - $\pi$  recognition site into bis( $\sigma$ -acetylide) platinum(II) system is important to improve their selectivity for benzene compounds.



**Scheme 1** Molecular structure and two host-guest recognition sites of complex **1**.

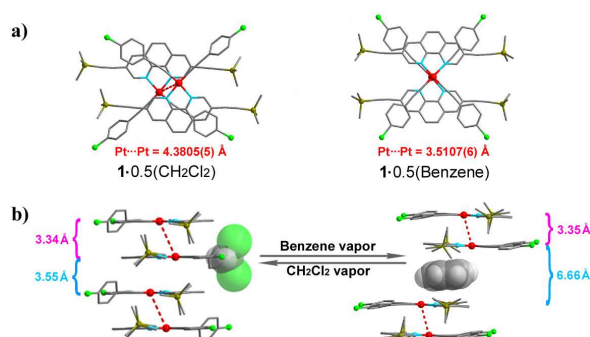
Based on the above considerations, herein we report the design and synthesis of a new bis( $\sigma$ -acetylide) platinum(II) complex [Pt(TMSc $\equiv$ CPhenC $\equiv$ CTMS)(C $\equiv$ CC<sub>6</sub>H<sub>4</sub>-Cl-4)<sub>2</sub>] (**1**) based on 3,8-bis(trimethylsilyl)ethynyl)-1,10-phenanthroline (TMSc $\equiv$ CPhenC $\equiv$ C TMS) bearing bigger  $\pi$ -electronics group (Scheme 1). **1** is characterized by two potential host-guest recognition sites, H-bond site that locates between two phenylacetylene auxiliary ligands and  $\pi$ - $\pi$  stacking site situated above the phenanthroline aromatic rings.

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†Electronic Supplementary Information (ESI) available: Detailed synthetic procedures, TGA diagram, tables and figures giving DFT calculation data, crystal data and additional crystallographic diagrams. CCDC 1055045 [1-0.5(CH<sub>2</sub>Cl<sub>2</sub>)] and 1055046 [1-0.5(Benzene)]. For ESI and crystallographic data in CIF or other electronic format See DOI: 10.1039/x0xx00000x

On one hand, **1** could use the two sites to selectively recognize different guest molecules. On the other hand, the newly imported  $\pi$ - $\pi$  stacking site could provide strong  $\pi$ - $\pi$  interactions for aromatic molecules, which enables **1** to be a potential sensor for detection of benzene compounds vapors. Detailed property study indicates the dual-recognition sites design strategy does work. **1** is an excellent common benzene compounds sensor and exhibits drastic change from yellow to red both in color and luminescence upon exposure to vapor of benzene compounds, and subsequently can be recovered by  $\text{CH}_2\text{Cl}_2$  vapor.

**1** was easily obtained in high yield by the reaction of precursor  $\text{Pt}(\text{TMSC}\equiv\text{CPhen}\equiv\text{CTMS})\text{Cl}_2$  and 4-chlorophenylacetylene according to Sonogashira's method ( $\text{CH}_2\text{Cl}_2/\text{Pr}_2\text{NH}/\text{CuI}$ ) and fully characterized by  $^1\text{H}$  NMR, ESI-MS, IR spectroscopy. **1** can dissolve in common organic solvents and exhibits excellent stability in both solution and solid state. Furthermore, **1** presents good thermal stability up to  $280^\circ\text{C}$  (Fig. S1 $\dagger$ ).

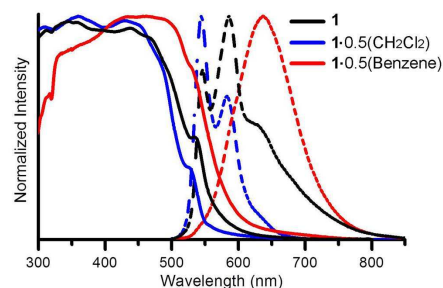


**Fig. 1** a) Vertical view of the dimer structure in  $1-0.5(\text{CH}_2\text{Cl}_2)$  and  $1-0.5(\text{Benzene})$ , showing the antiparallel packing patterns in both structures. b) Side view of the stacking structures in  $1-0.5(\text{CH}_2\text{Cl}_2)$  and  $1-0.5(\text{Benzene})$ , showing the solvate molecules recognized by recognition sites.

Recrystallization of **1** in  $\text{CH}_2\text{Cl}_2$  and benzene lead to yellow  $1-0.5(\text{CH}_2\text{Cl}_2)$  and red  $1-0.5(\text{Benzene})$  crystals respectively. $\ddagger$  Single crystal X-ray analysis reveal that platinum atom exhibits a distorted  $\text{C}_2\text{N}_2$  square-planar configuration in both structures (Fig. 1 and Table S1 $\dagger$ ). In  $1-0.5(\text{CH}_2\text{Cl}_2)$ , solvate  $\text{CH}_2\text{Cl}_2$  molecule is captured by the H-bond site and combines with platinum moiety through multi-hydrogen bonds (Fig. 1b, Fig. S2 and Table S2 $\dagger$ ). Two neighboring platinum(II) moieties display an antiparallel stacking pattern and have a severe slide from one another due to the steric hindrance of  $\text{CH}_2\text{Cl}_2$  molecule (Fig. 1a and Fig. S2 $\dagger$ ). Although the corresponding Pt...Pt separation is 4.3805(5) Å and there is no metallophilic interaction, strong C-H... $\pi$  interactions between the two moieties still connect them into a dimer (Fig. S3 and Table S2 $\dagger$ ). In contrast, in  $1-0.5(\text{Benzene})$  two neighboring platinum(II) moieties are arranged in closer proximity with Pt...Pt distance of 3.5107(6) Å (Fig. 1a). The results of Wiberg bond indices (WBIs) calculation of Pt-Pt contact also reveal a stronger bond order in  $1-0.5(\text{Benzene})$  (0.0968) relative to that in  $1-0.5(\text{CH}_2\text{Cl}_2)$  (0.0169), implying the existence of weak Pt-Pt interaction in  $1-0.5(\text{Benzene})$ . $^{8d, 15a}$  The Pt-Pt interaction and strong  $\pi$ - $\pi$  stacking interactions between alkynyl groups from phenylacetylene and aromatic rings from phenanthroline, connect two neighboring platinum(II) moieties into

a dimer (Fig. S4 and Table S2 $\dagger$ ). Solvate benzene molecule is situated in the  $\pi$ - $\pi$  stacking site between the two phenanthroline groups from two neighboring dimers and contacts with the groups through face-to-face  $\pi$ - $\pi$  stacking interactions (Fig. 1b, Fig. S5 and Table S2 $\dagger$ ). The intra- and inter- dimer separations of  $1-0.5(\text{Benzene})$  are 3.35 and 6.66 Å respectively, compared with 3.34 and 3.55 Å in  $1-0.5(\text{CH}_2\text{Cl}_2)$ . The different packing structures in both crystals demonstrate that in the solid state  $\pi$ - $\pi$  stacking site has a high affinity for benzene, which make **1** a possible vapor sensor for detection of benzene compounds.

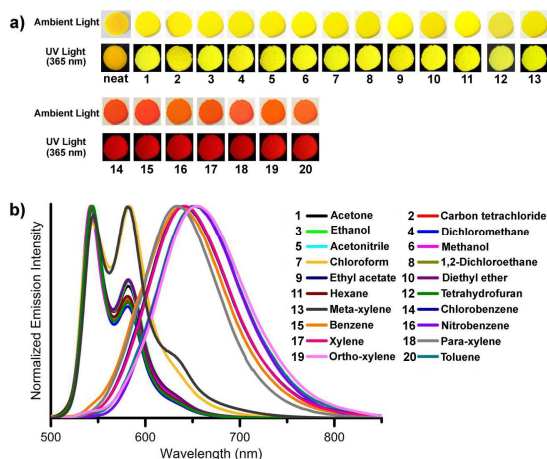
Fig. 2 gives the absorption and emission spectra for solid-state **1**, crystalline  $1-0.5(\text{CH}_2\text{Cl}_2)$  and  $1-0.5(\text{Benzene})$ . **1** exhibits high-energy absorption bands due mainly to intraligand transitions, and broad, low-energy absorption bands at 437-532 nm tailing to 680 nm.  $1-0.5(\text{CH}_2\text{Cl}_2)$  has a similar absorption spectrum. DFT calculation shows its broad, low-energy absorption bands at 429-525 nm tailing to 650 nm can be attributed to a mixture of  $^1\text{LLCT}$  and  $^1\text{MLCT}$  transitions (Fig. S6-S7 and Table S3-S4 $\dagger$ ). In contrast, the low-energy absorption bands of  $1-0.5(\text{Benzene})$  show a distinct redshift to 461-542 nm extending to 780 nm. Such redshift is also correlated with the shortened Pt...Pt distance (the increased Pt-Pt interaction). Under irradiation at  $\lambda_{\text{ex}} = 350-500$  nm, **1** shows a dark-yellow emission peaked at 546 and 585 nm with a shoulder at 629 nm. Crystalline  $1-0.5(\text{CH}_2\text{Cl}_2)$  exhibits a bright yellow luminescence peaks at 544 and 582 nm with a lifetime of 1.152  $\mu\text{s}$  at ambient temperature. Vibronic-structured emission bands in both solids with the vibrational progressional spacing of  $1200\text{ cm}^{-1}$  is typical for the C=C and C=N stretching frequency of the aromatic rings in phenanthroline, suggesting the involvement of  $\text{TMSC}\equiv\text{CPhen}\equiv\text{CTMS}$  in the emission state. Therefore, this yellow luminescence can be assigned to an admixture of  $^3\text{MLCT}$  and  $^3\text{LLCT}$  triplet transitions, as supported by TD-DFT studies (Fig. S7 and Table S3-S4 $\dagger$ ). In contrast, excitation of  $1-0.5(\text{Benzene})$  by near-UV light at room temperature leads to a broad, structureless emission centered at 638 nm with a lifetime of 416 ns. The dramatic red-shift in emission spectra and distinct peak shape strongly suggest different emission mechanism of  $1-0.5(\text{Benzene})$ . Considering the weak Pt-Pt interaction in  $1-0.5(\text{Benzene})$ , the red emission can be attributed to  $^3\text{MMLCT}$  triplet transition which has smaller HOMO-LUMO energy gap than that of the corresponding  $^3\text{MLCT}$  transition, as supported from DFT studies (Fig. S8-S9 and Table S5-S6 $\dagger$ ).



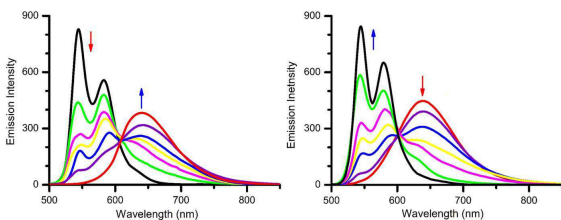
**Fig. 2** UV-Vis absorption spectra (solid) and emission (dash dot) spectra of solid state **1**, crystalline  $1-0.5(\text{CH}_2\text{Cl}_2)$  and  $1-0.5(\text{Benzene})$ .

To examine the color and luminescence responses to VOCs, **1** is deposited on quartz slide and then exposed to various saturated VOC vapors at ambient temperature and the result is shown in Fig.

3a. Remarkably, **1** is only sensitive to benzene compounds, including benzene, toluene, chlorobenzene, nitrobenzene, xylene, para-xylene and ortho-xylene, and presents red color under ambient light and red luminescence under UV light (365 nm) respectively. The corresponding emission spectra have a strong unstructured band centered in the range of 634-654 nm (Fig. 3b). In contrast, other VOCs lead to only yellow color and yellow emission. The corresponding emission spectra have vibronic-structured emission bands at 544 and 582 nm, similar to that of solid **1**·0.5(CH<sub>2</sub>Cl<sub>2</sub>) (Fig. 3b). Such distinct changes in color and luminescence of **1** upon exposure to different VOCs reveal that it has an excellent selectivity in detection of benzene compounds.



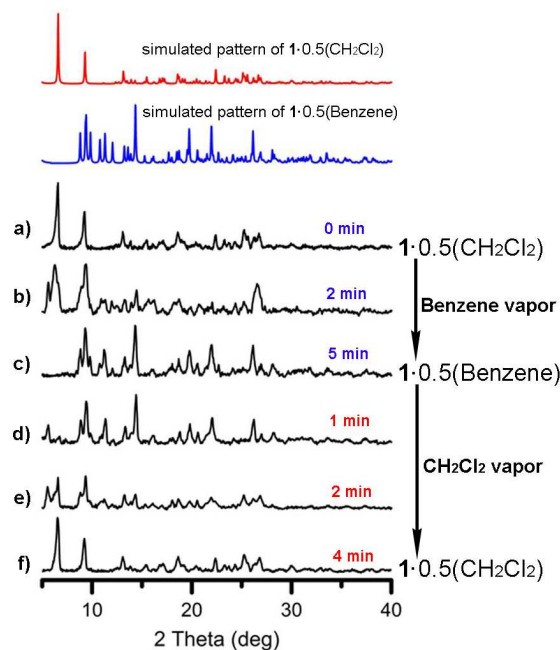
**Fig. 3** a) Photographic images of **1** deposited on quartz slide upon exposure to various VOC vapors under ambient light and UV light irradiation (365 nm). b) Solid-state emission spectra of **1** upon exposure to various VOC vapors at ambient temperature.



**Fig. 4** Emission spectral changes of solid state **1**·0.5(CH<sub>2</sub>Cl<sub>2</sub>) in response to benzene vapor (left) and **1**·0.5(Benzene) in response to CH<sub>2</sub>Cl<sub>2</sub> vapor (right).

In order to explore the reversibility of **1** as a benzene compounds sensor, the dynamic process in a vapor absorbing cycle was monitored by emission spectra. Fig. 4 shows the emission spectral changes of **1** in response to CH<sub>2</sub>Cl<sub>2</sub> or benzene vapor in the reverse process. When the solid sample of **1**·0.5(CH<sub>2</sub>Cl<sub>2</sub>) was exposed to benzene vapor, the vibronic-structured emission bands at 544 and 582 nm were weakened gradually and a broad, unstructured emission band centered at 638 nm was observed and enhanced progressively. Meanwhile, the color of the sample changes gradually from yellow to red. Powder XRD experiments (Fig. 5) indicate during the process the patterns of **1**·0.5(CH<sub>2</sub>Cl<sub>2</sub>) were gradually weakened whereas the patterns of **1**·0.5(Benzene) were progressively enhanced. Within several minutes **1**·0.5(CH<sub>2</sub>Cl<sub>2</sub>) is

completely transformed into **1**·0.5(Benzene). Once the resulted red phase was exposed to CH<sub>2</sub>Cl<sub>2</sub> vapor, the broad unstructured emission band could be reverted to the original vibronic-structured emission bands and the color also changed from red to yellow gradually, corresponding to the transformation of **1**·0.5(Benzene) to **1**·0.5(CH<sub>2</sub>Cl<sub>2</sub>), as revealed by XRD patterns. The response time of a process would take several dozen seconds to a few minutes depending on the thickness of the sample. Therefore, the color and luminescence changes of **1** in response to VOCs is fully reversible.



**Fig. 5** XRD patterns recorded in a vapor absorbing cycle process, **1**·0.5(CH<sub>2</sub>Cl<sub>2</sub>) ⇌ **1**·0.5(Benzene), showing dynamic variations of XRD patterns from a) to c) in the process **1**·0.5(CH<sub>2</sub>Cl<sub>2</sub>) → **1**·0.5(Benzene) by exposure of **1**·0.5(CH<sub>2</sub>Cl<sub>2</sub>) to benzene vapor, and the XRD patterns from c) to f) in the reverse process **1**·0.5(Benzene) → **1**·0.5(CH<sub>2</sub>Cl<sub>2</sub>) by exposing **1**·0.5(Benzene) to CH<sub>2</sub>Cl<sub>2</sub> vapor at ambient temperature.

All the above experiments indicate **1** can use H-bond and π-π stacking recognition sites to selectively and reversibly recognize CH<sub>2</sub>Cl<sub>2</sub> and benzene compounds vapors respectively. The strength of weak interactions of the two recognition sites and their favorite guest molecules should be close. Thus the two phases, **1**·0.5(CH<sub>2</sub>Cl<sub>2</sub>) and **1**·0.5(Benzene), can be conversed reversibly into each other in the presence of excess benzene or CH<sub>2</sub>Cl<sub>2</sub> vapors respectively, accompanying with adjustment of Pt...Pt separation inside the dimmers, and finally dramatic color and luminescence change. Remarkably, no detectable degradation was observed even after twenty vapor absorbing cycles (Fig. S10<sup>†</sup>), indicating that **1** has good reversibility and reproducibility in sensing benzene compounds.

Several platinum(II) bis(σ-acetylide) complexes based on 1,10-phenanthroline and its derivatives had been published, however, their sensing property to benzene compounds were not reported.<sup>16b, 17</sup> In order to check whether dual-recognition sites is a sufficient condition for the Pt(II) complex to sense



benzene compounds, we synthesized one of the such compounds (Pt(phen)C≡CC<sub>6</sub>H<sub>4</sub>) and used it as an example to check the response. Interestingly, no changes in color or luminescence were observed when it was exposed to vapors of different benzene compounds. A detailed comparison of structures of the compound and **1** indicates the former exhibits a layer packing structure with strong and complicated hydrogen bond networks between adjacent platinum moieties. In contrast, **1** adopts a column packing structure with simple and weaker hydrogen bonds between adjacent molecules which allow the π-π interactions between aromatic guest molecule and phen unit of the Pt(II) molecule to play an efficient role to change the packing structure. Thus the two kinds of weak interactions, namely hydrogen bond and π-π interaction, being close to each other in strength is another determining factor to affect the recognition function.

### Conclusions

In conclusion, a new vapochromic and vapoluminescent platinum(II) complex containing dual-recognition sites for detection of benzene compounds vapor was designed and synthesized. Compared with other reported benzene compounds vapor sensors,<sup>9-14</sup> **1** has obvious advantages of concurrent, selective, naked-eye perceivable, sensitive, reversible, reproducible, and easy performance. Thus the dual-recognition sites strategy opens up fresh opportunities to design new benzene compounds vapor sensors with better performance.

### Acknowledgements

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### Notes and references

‡ Crystal data for **1**·0.5(CH<sub>2</sub>Cl<sub>2</sub>): C<sub>38.50</sub>H<sub>33</sub>Cl<sub>3</sub>N<sub>2</sub>PtSi<sub>2</sub>, *M* = 881.29, monoclinic, *a* = 7.1264(4) Å, *b* = 38.160(2) Å, *c* = 14.3796(10) Å, β = 96.337(4)°, *V* = 3886.6(4) Å<sup>3</sup>, *T* = 296(2) K, space group *P*2<sub>1</sub>/*c*, *Z* = 4, 30480 reflections measured, 6824 independent reflections (*R*<sub>int</sub> = 0.037). The final *R*<sub>1</sub> values were 0.0371 (*I* > 2σ(*I*)). The final *wR*(*F*<sup>2</sup>) values were 0.0974 (*I* > 2σ(*I*)). The final *R*<sub>1</sub> values were 0.0484 (all data). The final *wR*(*F*<sup>2</sup>) values were 0.1029 (all data).

Crystal data for **1**·0.5(Benzene): C<sub>41</sub>H<sub>35</sub>Cl<sub>2</sub>N<sub>2</sub>PtSi<sub>2</sub>, *M* = 877.88, monoclinic, *a* = 20.638(3) Å, *b* = 10.9657(16) Å, *c* = 19.312(3) Å, β = 103.687(7)°, *V* = 4246.3(10) Å<sup>3</sup>, *T* = 296(2) K, space group *P*2<sub>1</sub>/*c*, *Z* = 4, 25213 reflections measured, 6879 independent reflections (*R*<sub>int</sub> = 0.082). The final *R*<sub>1</sub> values were 0.0691 (*I* > 2σ(*I*)). The final *wR*(*F*<sup>2</sup>) values were 0.1468 (*I* > 2σ(*I*)). The final *R*<sub>1</sub> values were 0.1326 (all data). The final *wR*(*F*<sup>2</sup>) values were 0.1780 (all data).

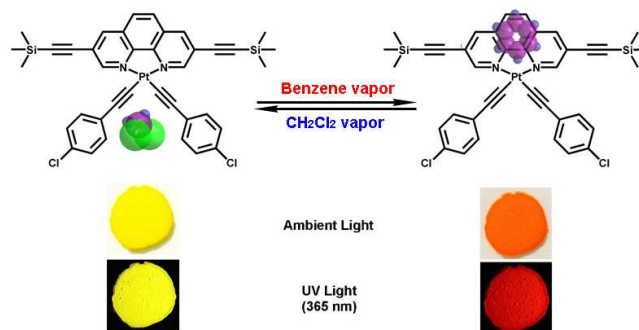
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## Graphical Abstract



A concurrent, selective, naked-eye perceivable, sensitive, reversible, reproducible, and easy performance sensor for detecting vapor of benzene compounds has been developed.