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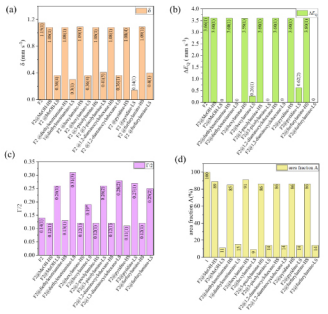
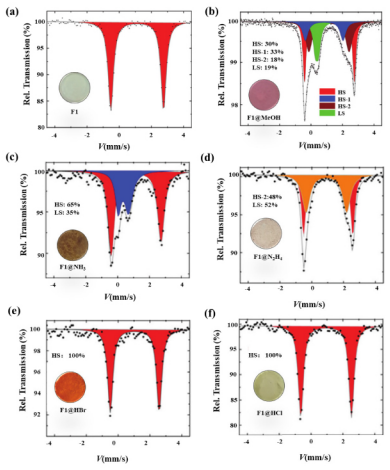
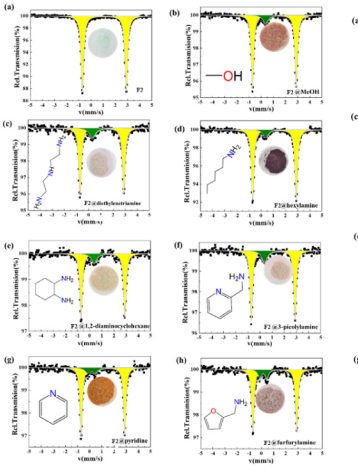
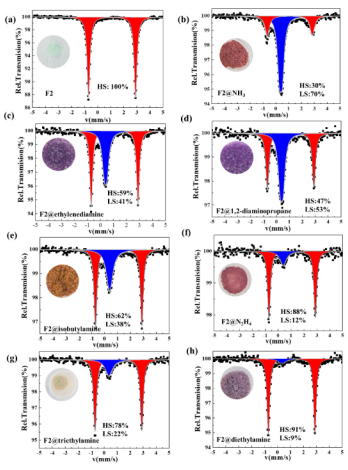
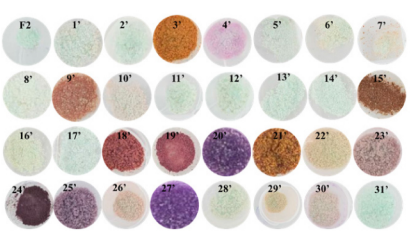
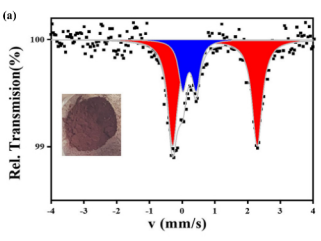
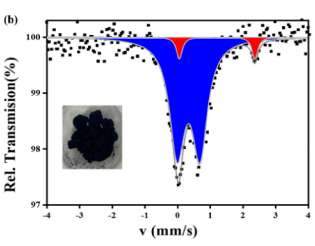
A Groundbreaking Study of ^{57}Fe Mössbauer Spectroscopy as a Tool to Explore Colorimetric Sensors for the Detection of Small Volatile Organics and Toxic Gases



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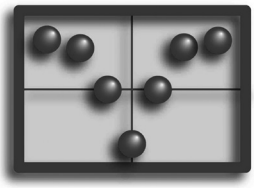

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On the cover:
This issue our front cover features photos is on a new family of iron(II) complexes discovered by Prof. Yann Garciar's group. Two young Mössbauer spectroscopists, Ms. Li Sun and Weiyang Li, both are from IMCN at UCLouvain in Belgium. (See more in Editor's Comments and in the enclosed Mössbauer Spectroscopy Newsletter.)

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~~Guest~~ Editor's Comments

A Groundbreaking Study of ^{57}Fe Mössbauer Spectroscopy as a Tool to Explore Colorimetric Sensors for the Detection of Small Volatile Organics and Toxic Gases

Pursuing efforts to shed light on the work of the next generation of Mössbauer spectroscopists, we discover this month Ms. Li Sun and Weiyang Li, both from IMCN at UCLouvain in Belgium, who have recently discovered a new family of iron(II) complexes able to colorimetric detect a variety of molecules, including toxic industrial chemicals, hazardous gases, etc. These two early stage researchers are both supported by the Chinese Scholarship Council in the frame of their PhD studies. Knowing better their field of investigation is expected to generate future opportunities by focusing on new topics where Mössbauer spectroscopy could be used, and stimulate new ideas and concepts for our readers, which can benefit to our community.

This new family of iron(II) sensors was born in Belgium in 2014, when Dr. Anil D. Naik (a postdoctoral researcher at IMCN at that time, now working for the chemical industry at Gloucester, UK) discovered a material able to switch its spin state in contact to vapor solvents while designing a nitrogen rich bis 1,2,4-triazole-tetrazole ligand (Inorg. Chem. 2014, 53, 1263-1265). Such study attracted considerable interest given that the ligand field of this material was not appropriate to exhibit any spin state crossover behavior, and was pursued by Dr. Yunnan Guo (FNRS postdoc at IMCN Belgium, before being appointed assistant professor at Zhejiang Sci-Tech University, China) on powder and crystal state (J. Mater. Chem. C. 2018, 6, 3895-3900 and Inorg. Chem. Front. 2021, 8, 3532-3546). Later, Li Sun and Weiyang Li discovered that the original material from 2014, actually belong to a new family of sensors which they investigate in the course of their PhD Studies. Their account outline how useful can be ^{57}Fe Mössbauer spectroscopy for probing the spin and oxidation state of these new materials.

Yann Garcia
Associate Editor
Institute of Condensed Matter and Nanosciences
Université Catholique de Louvain, Belgium

MÖSSBAUER SPECTROSCOPY
NEWSLETTER

April 2022

A Groundbreaking Study of ^{57}Fe Mössbauer Spectroscopy as a Tool to Explore Colorimetric Sensors for the Detection of Small Volatile Organics and Toxic Gases

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Li Sun

Li Sun was born in Shandong, China. She received her B.S. degree from Zaozhuang University in 2015. Then she received her M.S. degree in material chemistry working with Prof. Aiguo Wu and Prof. Haining Liu in University of Chinese Academy of Sciences (UCAS) in 2018. She could focus on three-dimensional network of carbon aerogels for boron and iodide adsorption, and oil (Organic solvents)-water separation, in addition, MOFs (ZIF-90) materials for cancer therapeutic exploration applications. She joined Prof. Yann Garcia's group at UCLouvain as a PhD student in 2018. Her research interest focuses on the design and synthesis of colorimetric sensors based on Fe (II) complexes to detect hazardous and toxic gases at room temperature, where Mössbauer spectroscopy play a major role. She has published 13 papers including two ACS and two RSC journals.

Weiyang Li

Weiyang Li completed his undergraduate studies in Geochemistry at the University of Northeast Petroleum University in China. He started his research career as a master student from 2016 to 2019 at Shanghai University of Engineering Science, focusing on metal organic frameworks (MOFs) for application in energy storage. Weiyang have scooped many awards during his master studies, including First Prize Graduate Scholarship and the National Postgraduate Scholarship awarded by Ministry of Education of China for the top final year honours student. Weiyang later joined Prof. Yann Garcia's research group as a PhD student at UCLouvain in Belgium in 2019. Now, he is working on Fe(II) spin crossover supramolecular cages and its sensing application. ^{57}Fe Mössbauer spectroscopy is systematically used to study the sensing mechanism. He has published 12 papers including two ACS and two RSC journals.

Yann Garcia

Yann Garcia was born in Castres (France) in 1972. He studied chemistry and physics at the Université of Bordeaux after being introduced to the field in Bergerac. He obtained his doctorate after a thesis supervised by Acad. Prof. Olivier Kahn at the Institute of Condensed Matter Chemistry of Bordeaux (ICMCB-CNRS). In early 1999, he joined the Institute of Inorganic and Analytical Chemistry of the University of Mainz (Germany) to work with Prof. Dr. Philipp Gütlich as a TMR EU post-doctoral fellow. He was appointed at UCLouvain (Belgium) in 2001 where he is now professor of analytical chemistry. The research interests of Dr. Garcia extend from photoswitchable coordination compounds, spin crossover materials to smart sensors for medicinal and environmental applications, where Mössbauer spectroscopy is applied as a key tool. Dr. Garcia has published more than 270 publications with several cover pages of top chemistry journals, book chapters, etc. Dr. Garcia is associate editor of the *Mössbauer Effect Reference Data Journal and of Chemical Synthesis*, both located in China. As president of the French Speaking Mössbauer Society (GFSM, www.gfsm.fr), he has promoted the technique in Morocco where he joined the honor committee of GFSM 2017 in Béni-Mellal and co-organized ICAMANA 2019 in Oujda. He was elected in 2021 as Vice chair of the international board member of the Applications of the Mössbauer Effect (IBAME, www.ibame.org).

Introduction and context

In recent years, with the continuous improvement of people's quality of life, the demand for a healthy environment has been increasing, thus making the air quality issue a constant concern.[1, 2] Among them, gas pollution not only comes from factories and car exhaust emissions, but also many indoor decorative items, such as home furniture, can also emit a large number of harmful gases.[3] Toxic gases are dangerous to human life and health, leading to skin redness, infection, and, in more serious cases, possible cancer developments.[4] Therefore, in order to live in a more healthy living environment, it is important to accelerate the development of methods to detect and control toxic and harmful gases. Traditional detection methods include gas chromatography, gas chromatography-mass spectrometry, etc.[5, 6] However, these methods rely on expensive and unportable large equipment, so they cannot achieve *in-situ* and *real-time* detection. The sample pretreatment process is more complicated and the detection process is more tedious, which requires higher professional skills. Therefore, they cannot achieve a popular use in daily life. This also apply to the fluorescence detection method,[7, 8] with a relatively high price of the instrument and need of strong professional skills. An alternative has been proposed through electrochemical methods, which are known to be relatively low-cost and portable, less time-consuming, and used for the detection of liquid phase components. Part of electrochemical sensors require however energy input[9] and therefore cannot be considered as green[9]. Therefore, the preparation of sensors for detecting toxic gases with the advantages of easy portability, fast response time, high selectivity and sensitivity, low cost and energy free is an urgent problem to be solved. At present, colorimetric sensors, due to their simple and rapid detection, intuitive and accurate results and excellent detection performance have attracted most attention in recent years.

Generally, a colorimetric sensor is a method of determining the content of a component to be measured by comparing or measuring the color depth of the colored substance based on the color reaction of the generated colored compound.[10] One approach to achieve this kind of material is through the construction of porous coordination polymers (PCPs)[11] (or metal organic frameworks, MOFs)[12], which by their nature contain the voids necessary to absorb guest molecules, thereby changing their properties. However, there are also a few molecular materials without any pores, which can be used as colorimetric sensor. Insights to the color change mechanism have been recently disclosed. This includes: (i) direct coordination of guest molecules to the metal center, leading to

significant changes to the *d*–*d* absorptions;[13-16] (ii) weak interaction of guest molecules with the sensor (such as H-bonding), causing the ligand field strength change of central ions;[17] (iii) modulation of the molecular structure inside the sensor crystal via guest molecules;[18] (iv) guest-exchange of guest molecules in the crystal lattice.[19-21] Regardless of the color change mechanism, the current challenge is to design and synthesize complex molecules that are sensitive to small changes and capable of making color changes. Spin crossover (SCO) materials could be considered as great candidates to act as molecular chemical sensors provided their spin state switching is associated to a color change as signal detection. Among this family of materials, some Fe(II) SCO complexes show stable color sets in ambient conditions corresponding to their high-spin (HS) and low-spin (LS) states.[22, 23] The spin state modification actually depends on subtle changes in their chemical environment,[24] which can be tracked by ^{57}Fe Mössbauer spectroscopy which can quantitatively determine the respective spin state population, i.e. HS and LS states.[25] Recent research progresses in our laboratory have focused on the detection and discrimination among a wide range of toxic and hazardous guest vapor molecules by colorimetric sensors based on Fe(II) complexes, which we describe within this account article.

Colorimetric Gas Sensors based on Fe(II) complexes

Recently, Garcia *et al*. reported on a series of Fe(II) complexes chemically responsive pigment with the ability to differentiate different hazardous gases.[15, 22, 23, 26] Two novel Fe(II) mononuclear complexes, namely $[Fe(trz\text{-}tet)_2(H_2O)_4]\cdot nH_2O$ (**F1**) (*trz-tetH* = 4H-1,2,4-triazolyl)-2H-tetrazole)[22, 23, 26] and $[Fe(H_2btm)_2(H_2O)_2]Cl_2$ (**F2**) (*H$_2$btm* = di(1H-tetrazol-5-yl)methane),[15] were synthesized and characterized as colorimetric sensors for a wide range of Volatile Organic Compounds (VOCs) including toxic gases, such as, methanol, hydrazine, ammonia and various amines, etc. Interestingly, **F1** was found to precisely detect methanol among alcohols and toxic industrial chemicals in the gas phase based on molecular sieving and spin-state change. Neighboring ligands were suspected to coordinate with Fe(II) ions starting from a HS FeN_2O_4 core to form a LS FeN_6 coordination sphere thus provoking a color change, from colorless to red (Figure 1).[22, 23] ^{57}Fe Mössbauer spectroscopy was applied to investigate the driving spin state change of the guest molecules in **F1** (Figure 1). The spectrum of **F1** (Figure 1a) was fitted best with one quadrupole doublet with an isomer shift δ = 1.17(1) mm/s and a quadrupole splitting ΔE_Q = 3.18(3) mm/s, which are typical for HS Fe(II) ions. After $MeOH_{(g)}$ adsorption, a new quadrupole doublet (19%) appears at δ = 0.37(1) mm/s and ΔE_Q = 0.20(2) mm/s, for **F1@MeOH** (Figure 1b), which is characteristic of a LS FeN_6 core with azole ligands. The two remaining contributions correspond to HS Fe(II) sites: HS-1 [δ = 1.14(2) mm/s; ΔE_Q = 2.56(5) mm/s; 33%] and HS-2 [δ = 0.83(3) mm/s; ΔE_Q = 2.40(5) mm/s; 18%]. The situation differs with $NH_{3(g)}$ with a single Fe(II) LS contribution indicating an incomplete switching. For HBr and HCl, no color change is observed, as confirmed by the permanent HS state detected by Mössbauer spectroscopy. This situation exists too for hydrazine monohydrate, but in this case two HS Fe(II) sites are detected (Figure 1). Relevant ^{57}Fe Mössbauer parameters for sensing $MeOH_{(g)}$ and selected toxic industrial chemicals are listed in **Table 1**.

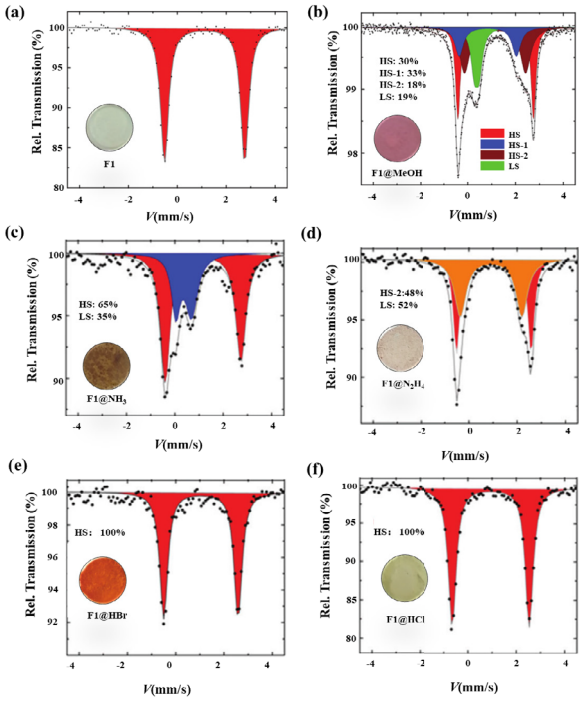

Figure 1. Spin-state tracking by ^{57}Fe Mossbauer spectroscopy of **F1** before and after adsorption toxic gases at room temperature [22].

Table 1. Overview of ^{57}Fe Mössbauer parameters of **F1** for sensing $MeOH_{(g)}$ and industrial harmful gases [22].

	Spin state	Mössbauer parameters			
		δ (mm s^{-1})	ΔE_Q(mm s^{-1})	Γ/2	% of species
F1	HS	1.17(2)	3.18(3)	0.20(1)	100
F1@NH_3	HS	1.17 (1)	3.13(3)	0.25(2)	65
	LS	0.36(3)	0.65(5)	0.30(6)	35
F1@HCl	HS	1.06(1)	3.26(1)	0.20(1)	100
F1@HBr	HS	1.16(1)	3.14(2)	0.20(2)	100
F1@N_2H_4	HS	1.14(1)	3.16(2)	0.16(2)	48
	HS-2	1.03(2)	2.61(8)	0.29(4)	52
F1@MeOH	HS	1.17(1)	3.17(1)	0.12(1)	30
	HS-1	1.14(2)	2.56(5)	0.27(4)	33
	HS-2	0.83(3)	2.40(5)	0.22(4)	18
	LS	0.37(1)	0.20(2)	0.17(2)	19

δ: isomer shift (r.t., vs. α-Fe); ΔE_Q: quadrupole splitting, Γ/2: half width at half maximum.

The investigations were extended to another chemosensor (**F2**) which was found to display different colors after adsorption of a set of various toxic gases (Figure 2). With this system, colors can be distinguished by the naked eye without the need for any cryogenic equipment or temperature pre-treatment at room temperature. Interestingly, **F2** can detect not less than 14 different VOCs and hazardous gases (HGs) at real time, with a high selectivity and ultra-sensitivity. In particular amines, which are detected very quickly (<2 min) with very high sensitivity. In addition, toxic gases were readily identified using a standard chemometric approach (hierarchical clustering analysis) to record and analyze collected data of the color of the picture.[15] In particular, the purple and red colors detection (e.g., after adsorption 1,2-diaminopropane$_{(g)}$ and ammonia$_{(g)}$ molecules, respectively) were systematically associated to the emergence of $Fe^{II}N_6$ LS ions as shown by ^{57}Fe Mössbauer spectroscopy analyses (Figure 3), due to guest molecules replacement of H_2O molecules coordinated to the metal center, leading to significant changes to the *d*−*d* absorptions. More important, the sensor itself has a high sensitivity detection towards toxic gases even at very low gas concentration, especially for ammonia$_{(g)}$. Furthermore, a friendly smartphone-based analytical method was developed to make **F2** accessible for *in-field* applications. ^{57}Fe Mössbauer spectroscopy of **F2** (Figure 3), after adsorption $NH_{3(g)}$ (Figure 3b), and 1,2-diaminopropane (Figure 3d) molecules, a new quadrupole doublet in the center [δ = 0.38(1) mm/s, ΔE_Q = 0.23(1) mm/s] of 70% and [δ = 0.40(1) mm/s, ΔE_Q = 0.22(2) mm/s] of 53%, respectively. For isobutylamine molecules (Figure 3e)), a new quadrupole doublet in the center [δ = 0.38(1) mm/s, ΔE_Q = 0.08(3) mm/s] of 38%, which are characteristic of a LS FeN_6 core with ligands. From Figure 4, after adsorbing hexylamine$_{(g)}$ molecules, a new quadrupole doublet emerges in the center [δ = 0.36(4) mm s^{-1}; ΔE_Q = 0.26(1) mm s^{-1}] of area fraction A = 9%, in the spectrum of **F2@ hexylamine** (Figure 4d), which is characteristic of LS Fe(II) ions. A quadrupole doublet was

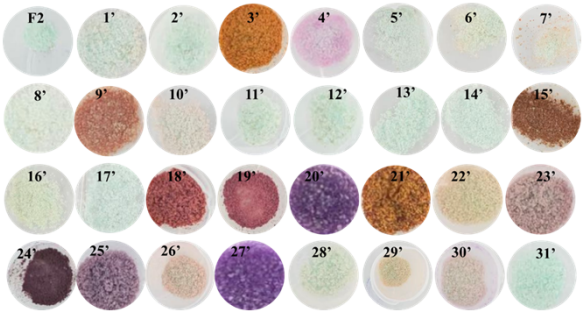

Figure 2. Images depicting vapochromic transformation on the sensing of VOCs by complex **F2** for 120 min at room temperature on ground (**F2**; 1'= water; 2'= acetonitrile; 3'= pyridine; 4'= formaldehyde; 5'= acetone; 6'= chloromethane; 7'= dichloromethane; 8'= tetrahydrofuran; 9'= methanol; 10'= ethanol; 11'=1-pentanol; 12'=1-butanol; 13'= hydrochloric acid; 14'= sulfuric acid; 15'=nitric acid; 16'= bromic acid; 17'= acetic acid; 18'= ammonia; 19'= hydrazine ; 20'= ethylenediamine; 21'= isobutylamine; 22'=1,2-diaminocyclohexane; 23'= furfurylamine; 24'=hexylamine; 25'= diethylamine; 26'= 3-picolylamine; 27'= 2-diaminopropane; 28'= aniline; 29'= triethylamine; 30'= diethylenetriamine; 31'= 2-chloroaniline) [15].

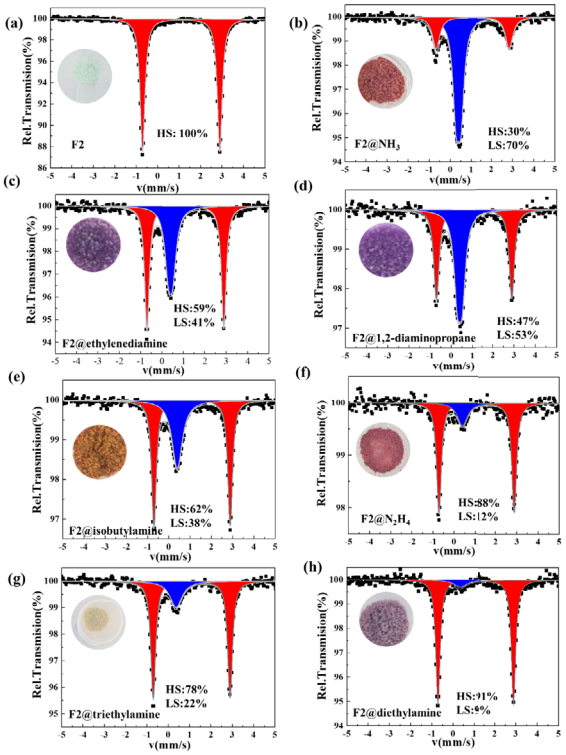

Figure 3. ^{57}Fe Mössbauer spectroscopy analysis of **F2** towards different vapors crystals at room temperature [15].

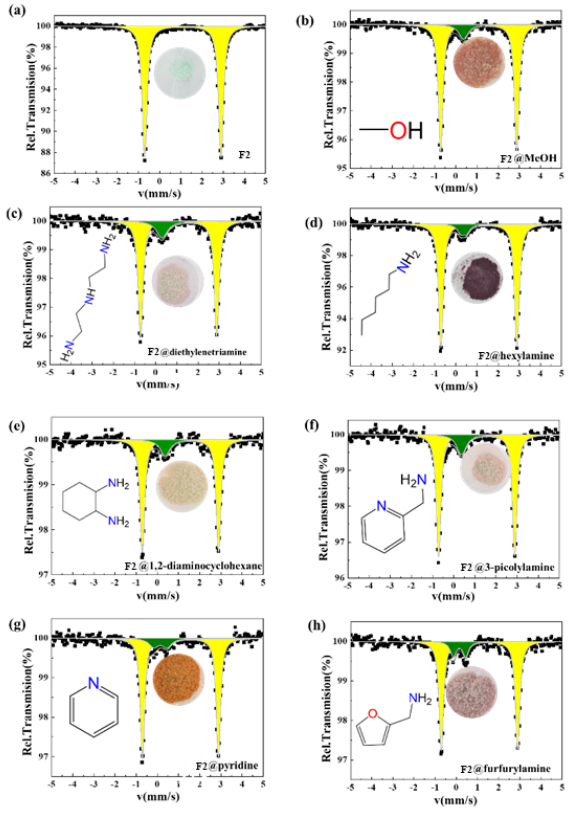

Figure 4. ^{57}Fe Mössbauer spectra at room temperature for (a) **F2**; (b) **F2@MeOH**; (c) **F2@diethylenetriamine**; (d) **F2@hexylamine**; (e) **F2@1,2-diaminocyclohexane**; (f) **F2@3-picolylamine**; (g) **F2@pyridine**; (h) **F2@furfurylamine** [16].

also identified for **F2@pyridine** with $\delta = 0.14(1)$ mm s^{-1} and $\Delta E_Q = 0.62(2)$ mm s^{-1}, whereas a singlet was observed for **F2@MeOH**, **F2@diethylenetriamine**, **F2@3-picolylamine**, **F2@1,2-diaminocyclohexane** and **F2@furfurylamine**, at $\delta \sim 0.38$ mm s^{-1} (Figure 4, Figure 5a and Figure 5b), with area fraction ~14% (Figure 5d).[16] Indeed, replacement of oxygen atoms by nitrogen atoms, which are less electronegative, increases the electronic density of S electrons in the vicinity of the iron nucleus and thereby decreases the isomer shift (the spin states of Fe(II) from HS to HS).[15] Last but not least, our studies have also shown that **F2** has excellent regeneration performance of adsorption and de-adsorption of VOCs. It operates at ambient conditions, is low cost, environmentally friendly, and easy to use, and shows excellent and fast detection performances. Such features offer attractive prospects for **F2**, which could be used for *in-field* detection and food safety control in environmental conditions.

Recently, we have extended our investigations to a new mononuclear Fe(II) complex (**F3**) with formula $[FeL_2](BF_4)_2$ (L = (E)-2-((pyridin-2-ylmethylene)amino)-1Hbenzo[*de*]-isoquinoline-1,3(2H)-dione) for potential $NH_{3(g)}$ sensing applications.[17] The remarkable and quick color

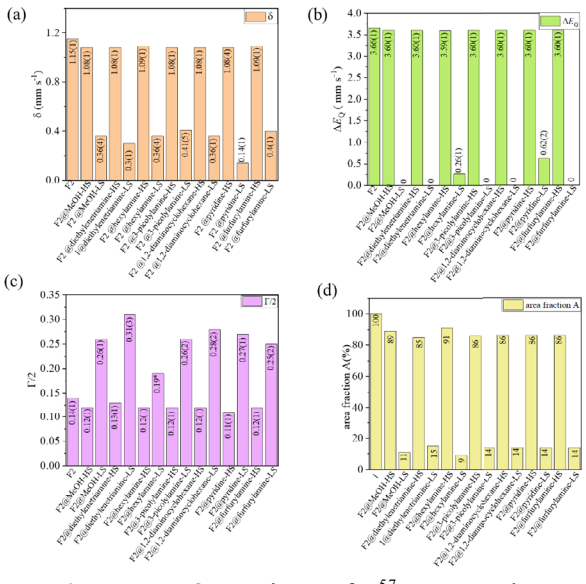

Figure 5. Overview of ^{57}Fe Mössbauer parameters (δ (mm s^{-1}), ΔE_Q (mm s^{-1}), $\Gamma/2$ and % of species) for **F2**, **F2@MeOH**, **F2@1,2-diaminopropane**, **F2@diethylenetriamine**, **F2@hexylamine**, **F2@3-picolylamine**, **F2@1,2-diaminocyclohexane**, **F2@pyridine**, **F2@furfurylamine** at room temperature. (δ: isomer shift (r.t., vs. α-Fe); ΔE_Q: quadrupole splitting, $\Gamma/2$: half width at half maximum) [16].

Table 2. Overview of ^{57}Fe Mössbauer parameters for **F2**, **F2@NH$_3$**, **F2@N$_2$H$_4$**, **F2@isobutylamine**, **F2@1,2-diaminopropane**, **F2@ethylenediamine**, **F2@triethylamine**, **F2@diethylamine** at room temperature [16].

	Spin state	Mössbauer parameters			
		δ (mm s^{-1})	ΔE_Q (mm s^{-1})	Γ/2	% of species
F2	HS	1.15(1)	3.66(1)	0.14(1)	100
F2@NH$_3$	HS	1.08(1)	3.51(3)	0.20(2)	30
	LS	0.38(1)	0.23(1)	0.17(1)	70
F2@N$_2$H$_4$	HS	1.00(1)	2.57(1)	0.17(1)	88
	LS	0.39(3)	0.20(5)	0.14(1)	12
F2@isobutylamine	HS	1.08(3)	3.59(1)	0.12(1)	62
	LS	0.38(1)	0.08(3)	0.28(5)	38
F2@1,2-diaminopro-pane	HS	1.09(1)	3.60(1)	0.14(1)	47
	LS	0.40(1)	0.22(2)	0.19(2)	53
F2@ethylenediamine	HS	1.08(1)	3.59(1)	0.12(1)	59
	LS	0.39(1)	0.20(3)	0.20(1)	41
F2@triethylamine	HS	1.09(3)	3.60(1)	0.13(1)	78
	LS	0.36(1)	0	0.34(1)	22
F2@diethylamine	HS	1.08(1)	3.59(1)	0.12(1)	91
	LS	0.35(1)	0	0.37(6)	9

δ: isomer shift (with respect to α-Fe at 298 K); ΔE_Q : quadrupole splitting; Γ/2: half width at half maximum

change from pink to black after exposure to $NH_{3(g)}$, at room temperature, is shown in Figure 6. After adsorbing NH_3 gas molecules, the ^{57}Fe Mössbauer spectrum is obviously modified (Figure 6b). **F3**@NH_3 exhibits a new quadrupole doublet characteristic of LS Fe(II) ions centered at δ = 0.33(2) mm/s and ΔE_Q = 0.70(4) mm/s with a large area fraction of 93%. Such spin state change could be caused by weak interactions between the oxygen-rich ligands and the guest NH_3 molecules, resulting in a subtle change of the coordination environment of iron centres, and thereby leading to a spin state change observed at room temperature.[17]

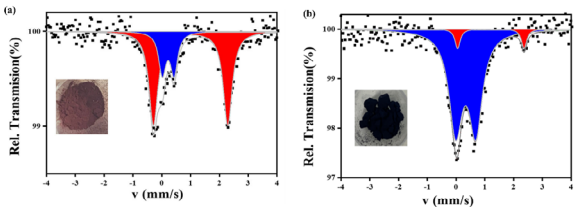

Figure 6. ^{57}Fe Mössbauer spectrum of **F3** (a) before and (b) after adsorption of $NH_{3(g)}$ recorded at 298 K. The red and blue quadrupole doublets correspond to HS and LS Fe(II) ions, respectively. The inset shows the pink color of the powder [17].

Conclusions and perspectives

Colorimetric sensors based on Fe(II) complexes can change their spin state after adsorption of guest vapor molecules at room temperature. New molecules are replaced in the Fe(II) coordination sphere or effect the electronic environment of Fe(II) (change the structure of

Table 3. Overview of selected ^{57}Fe Mössbauer parameters for **F3** and **F3**@NH_3 [17].

	Spin state	Mössbauer parameters			
		δ (mm s^{-1})	ΔE_Q (mm s^{-1})	Γ/2	% of species
F3	HS	1.00(2)	2.58(4)	0.18(3)	74
	LS	0.22(5)	0.42(1)	0.16(1)	26
F3@NH$_3$	HS	1.21(1)	2.31(2)	0.11(1)	7
	LS	0.33(2)	0.70(4)	0.28(3)	93

δ: isomer shift (with respect to α-Fe at 298 K); ΔE_Q: quadrupole splitting; Γ/2: half width at half maximum

the crystal after adsorption gas-phase molecules). Such changes are accompanied with the color change of the sensor and can be distinguished by the naked eye at room temperature. Usually, with a fast response, intuitive and accurate results of a colorimetric sensor, not only achieve the specific detection of toxic gases but also can be used for the *in-field* detection, provided an association to an image database processing method is realized. Broad application prospects in environmental protection and food safety assessment are at this stage foreseen, and **^{57}Fe Mössbauer spectroscopy** is expected to play a key role in this growing area.

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