

# Inorganics 2022

Exploring bacterial growth effects on *Mycobacterium Smegmatis* with the anti-carcinogenic vanadium-compounds

## Supplemental Material

### Authors

Zeyad Arhouma<sup>1,2</sup>, Heide A. Murakami<sup>2</sup>, Jordan T. Koehn<sup>2</sup>, Xiaorong Li<sup>2,3</sup>, Deborah A. Roess<sup>1,4</sup>, Dean C. Crick<sup>1,5</sup> and Debbie C. Crans,<sup>1,2\*</sup>

### Affiliations

<sup>1</sup>Cellular and Molecular Biology, Colorado State University, 80523, Fort Collins, CO, USA

<sup>2</sup>Department of Chemistry, Colorado State University, 80523, Fort Collins, CO, USA

<sup>3</sup>College of Pharmaceutical Sciences, Southwest University, 400715, Chongqing, China

<sup>4</sup>Departments of Biomedical Sciences, Colorado State University, 80523, Fort Collins, CO, USA

<sup>5</sup>Mycobacteria Research Laboratories, Department of Microbiology, Immunology and Pathology, Colorado State University, 80523, Fort Collins, CO, USA

Correspondence email: [debbie.crans@colostate.edu](mailto:debbie.crans@colostate.edu)

## Table of Contents

<b>I. Biological Data</b>	<b>Page</b>
<b>Figure S1.</b> The histograms of the IC <sub>50</sub> values for the effects of the V-complexes and their free ligands on the bacterial growth	4
<b>Figure S2.</b> The growth inhibition curves and IC <sub>50</sub> values for the effects of the V-complexes and their free ligands on the bacterial growth	5
<b>Figure S3.</b> The growth of <i>M. smegmatis</i> in the presence of DMSO	6
<b>II. New vanadium complexes characterization</b>	<b>Page</b>
<b>Figure S4.</b> The <sup>51</sup> V NMR spectrum of [VO(Hshed)(4NO <sub>2</sub> )]	7
<b>Figure S5.</b> The <sup>51</sup> V NMR spectrum of [VO(Hshed)(CN)]	8
<b>Figure S6.</b> The <sup>51</sup> V NMR spectrum of [VO(Hshed)(3OMet)]	9
<b>Figure S7.</b> The <sup>51</sup> V NMR spectrum of [VO(Hshed)(Coum)]	10
<b>Figure S8.</b> The <sup>1</sup> H NMR spectrum of [VO(Hshed)(4NO <sub>2</sub> )]	11
<b>Figure S9.</b> The <sup>1</sup> H NMR spectrum of [VO(Hshed)(CN)]	12
<b>Figure S10.</b> The <sup>1</sup> H NMR spectrum of [VO(Hshed)(3OMet)]	13
<b>Figure S11.</b> The <sup>1</sup> H NMR spectrum of [VO(Hshed)(Coum)]	14
<b>Figure S12.</b> The IR spectrum of [VO(Hshed)(4NO <sub>2</sub> )]	15
<b>Figure S13.</b> The IR spectrum of [VO(Hshed)(CN)]	16
<b>Figure S14.</b> The IR spectrum of [VO(Hshed)(3OMet)]	17
<b>Figure S15.</b> The IR spectrum of [VO(Hshed)(Coum)]	18
<b>Figure S16.</b> The UV-vis spectrum of [VO(Hshed)(4NO <sub>2</sub> )]	19

**Figure S17.** The UV-vis spectrum of [VO(Hshed)(CN)] 20

**Figure S18.** The UV-vis spectrum of [VO(Hshed)(3OMet)] 21

**Figure S19.** The UV-vis spectrum of [VO(Hshed)(Coum)] 22

### III. Speciation Data for the vanadium complexes and free ligands Page

**Figure S20.** The UV-vis spectra are recorded of all the complexes at 0.250 mM in ddH<sub>2</sub>O as a function of time (0, 1, 5, 24 h). 23

**Figure S21.** The UV-vis spectra are recorded of all the complexes at 0.250 mM in supernatant fraction as a function of time (0, 1, 5, 24 h). 24

**Figure S22.** The UV-vis spectra are recorded of all the free ligands at 0.250 mM in ddH<sub>2</sub>O and 7H9 medium at time zero (0 h). 25

**Figure S23.** The <sup>51</sup>V NMR spectra of 10mM [VO(Hshed)(3OMet)] at 0, 1, 5, and 24 h time points in **a)** 50:50 H<sub>2</sub>O:DMSO and in **b)** 50:50 7H9 growth medium: DMSO. 26

**Figure S24.** The <sup>51</sup>V NMR spectra of 10mM [VO(Hshed)(tbc)] at 0, 1, 5, and 24 h time points in **a)** 50:50 H<sub>2</sub>O:DMSO and in **b)** 50:50 7H9 growth medium: DMSO. 27

**Figure S25.** The <sup>51</sup>V NMR spectra of 10mM [VO(Hshed)(4NO<sub>2</sub>)] at 0, 1, 5, and 24 h time points in **a)** 50:50 H<sub>2</sub>O:DMSO and in **b)** 50:50 7H9 growth medium: DMSO. 28

**Figure S26.** The <sup>51</sup>V NMR spectra of 10mM [VO(Hshed)(CN)] at 0, 1, 5, and 24 h time points in **a)** 50:50 H<sub>2</sub>O:DMSO and in **b)** 50:50 7H9 growth medium: DMSO. 29

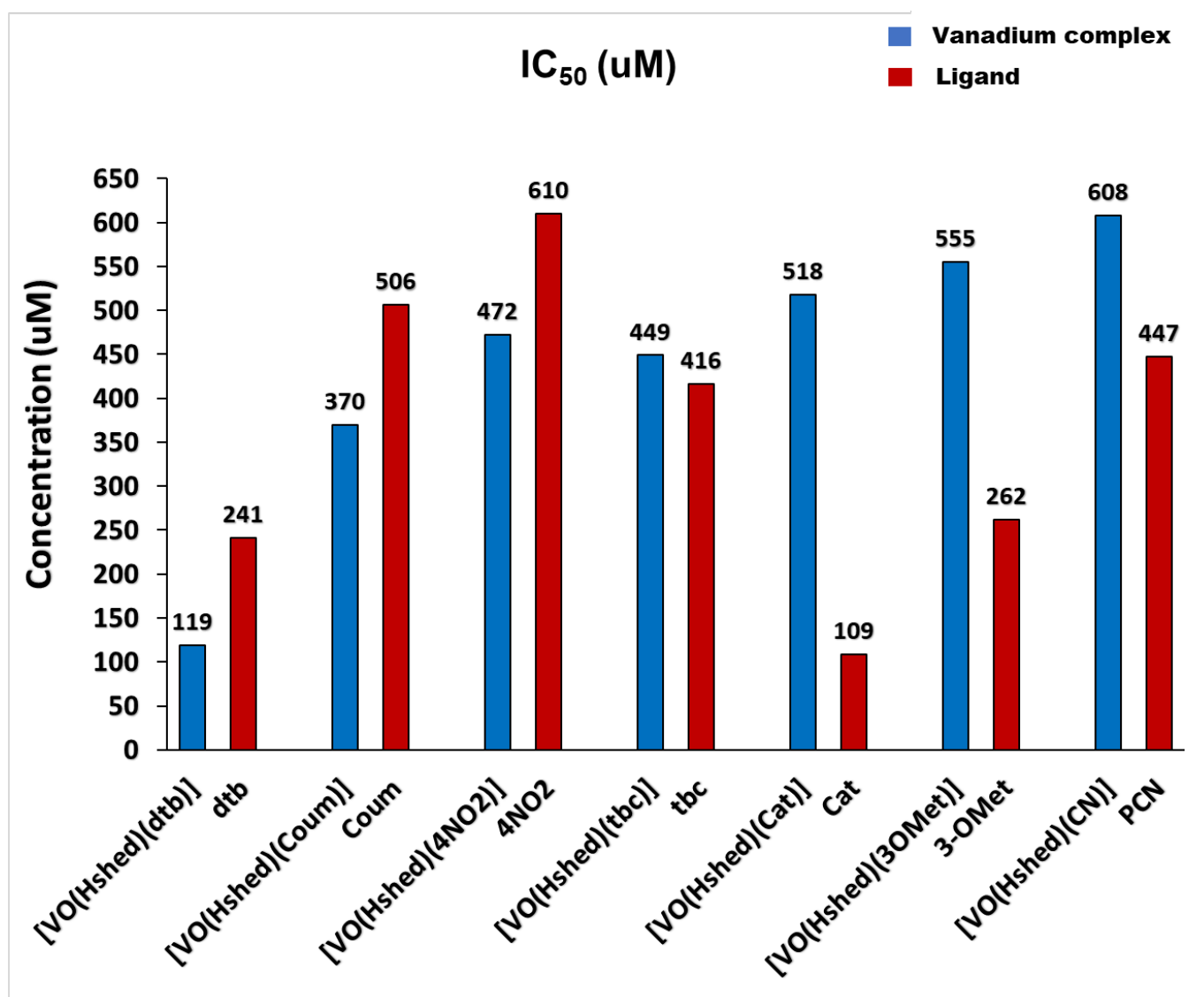
**Figure S27.** The <sup>51</sup>V NMR spectra of 10mM [VO(Hshed)(Coum)] at 0, 1, 5, and 24 h time points in **a)** 50:50 H<sub>2</sub>O:DMSO and in **b)** 50:50 7H9 growth medium: DMSO. 30

**Figure S28.** The <sup>51</sup>V NMR spectra of 10mM [VO(Hshed)(dtb)] at 0, 1, 5, and 24 h time points in supernatant fraction. 31

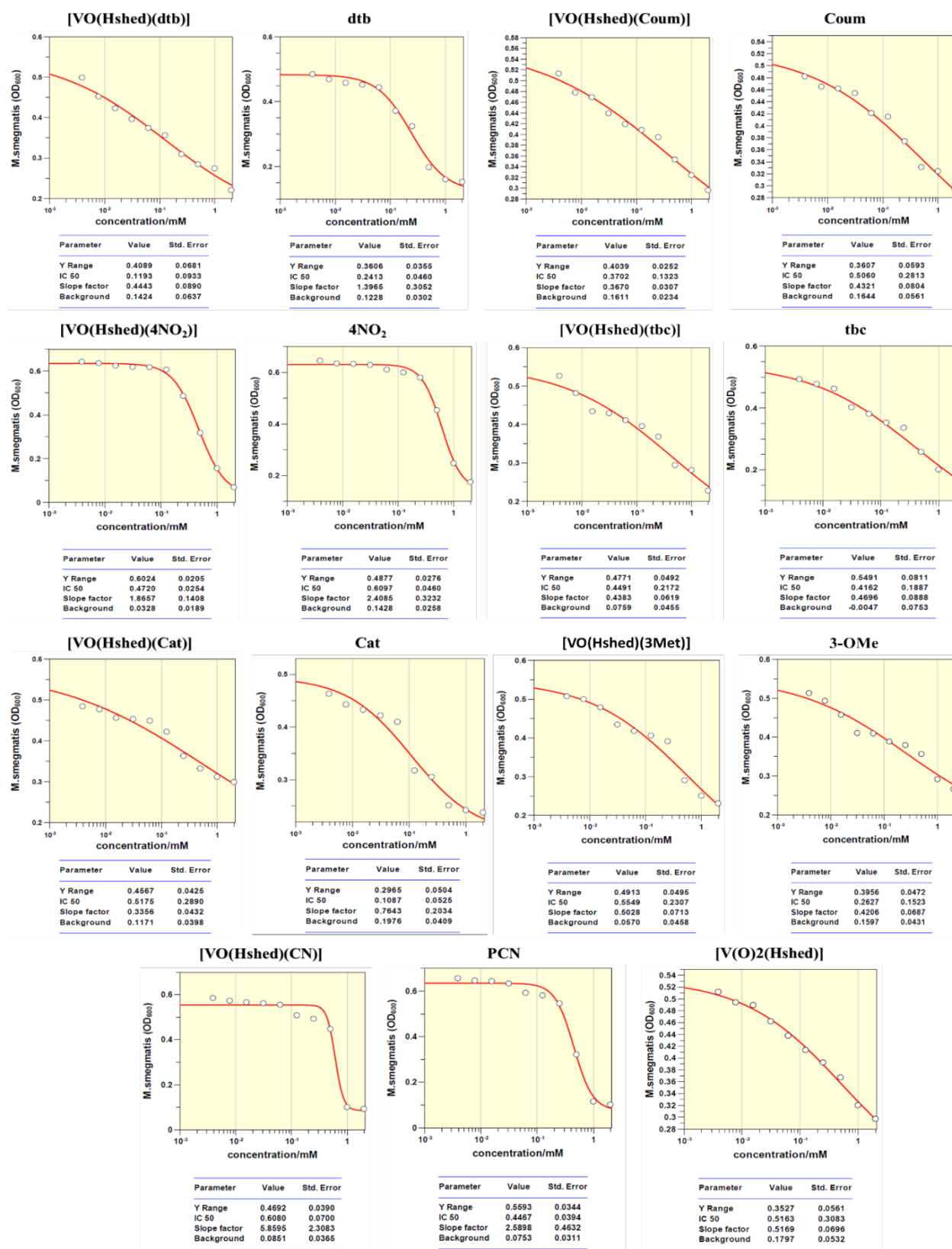
**Figure S29.** The <sup>51</sup>V NMR spectra of 10mM [VO(Hshed)(cat)] at 0, 1, 5, and 24 h time points in supernatant fraction. 32

**Figure S30.** The <sup>51</sup>V NMR spectra of 10mM [V(O)<sub>2</sub>(Hshed)] at 0, 1, 5, and 24 h time points in supernatant fraction. 33

**Figure S31.** The <sup>51</sup>V NMR spectra of 10mM [VO(Hshed)(3OMet)] at 0, 1, 5, and 24 h time points in supernatant fraction. 34



**Figure S1.** The histograms of the IC<sub>50</sub> values for the effects of the V-complexes and their free ligands on the bacterial growth.



**Figure S2.** The growth inhibition curves and IC<sub>50</sub> values for the effects of the V-complexes and their free ligands on the bacterial growth.

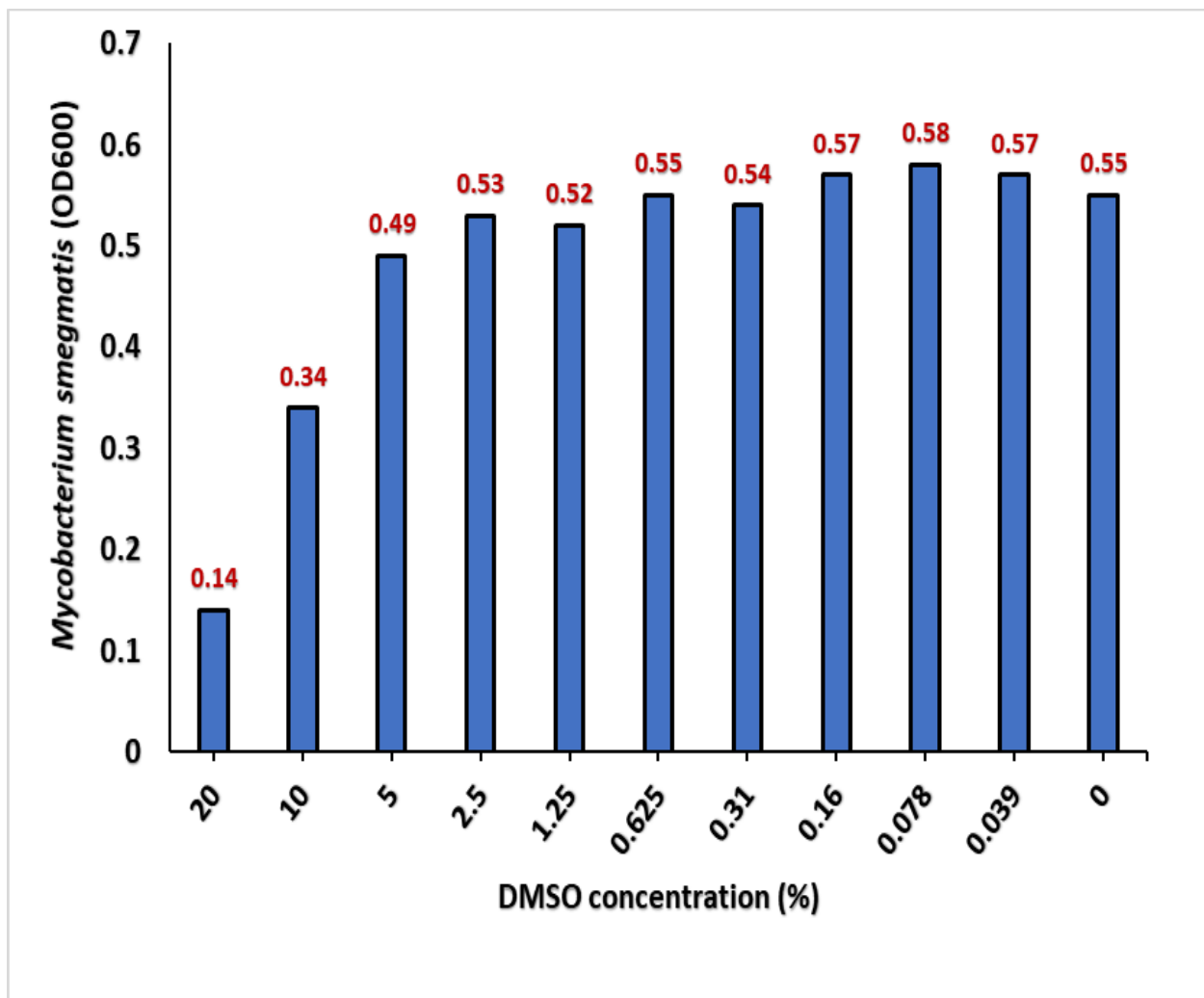
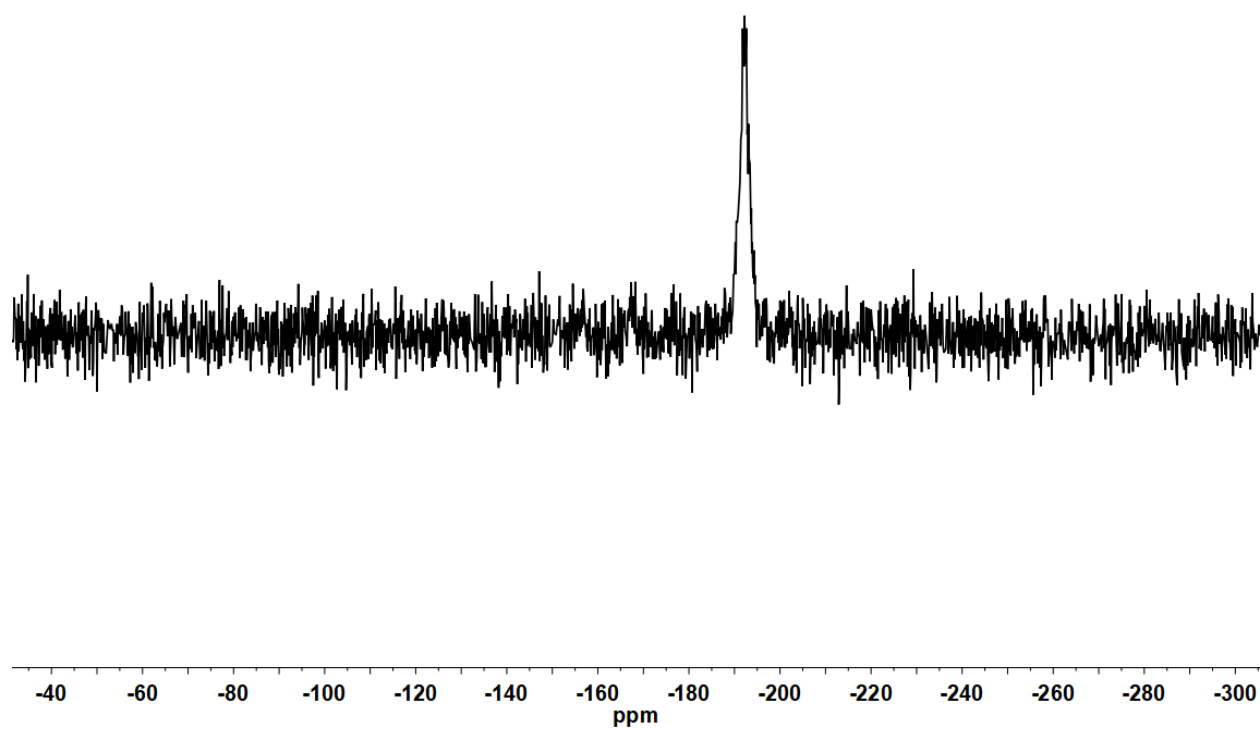
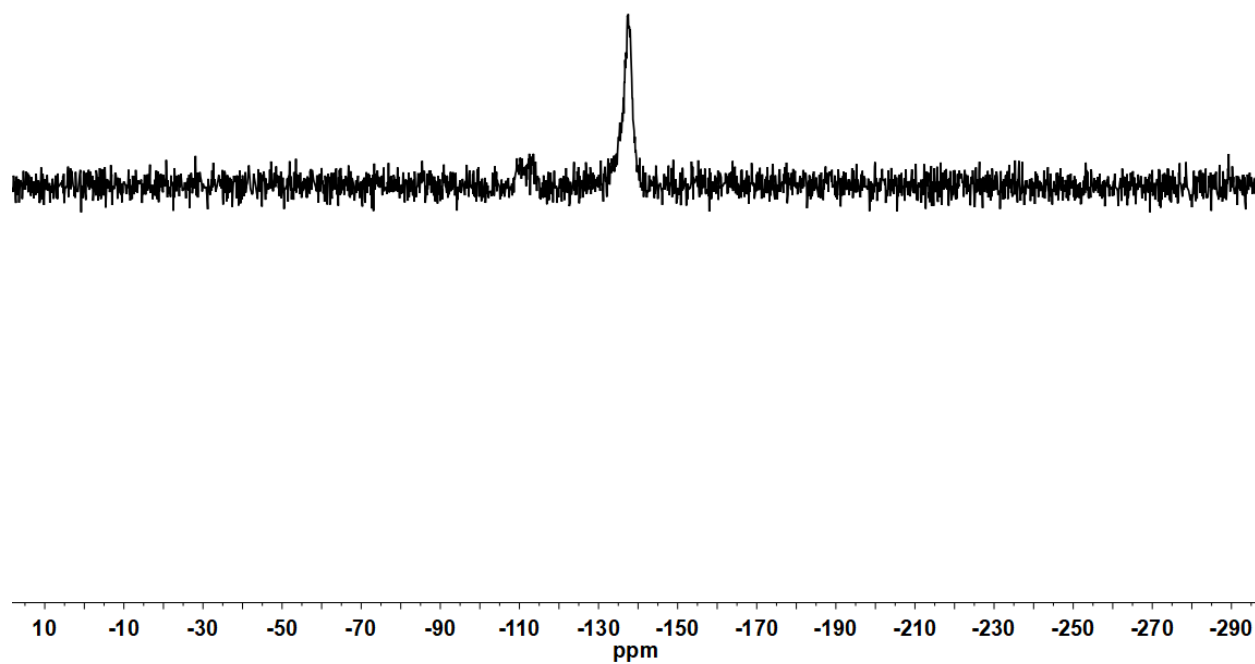


Figure S3. The growth of *M. smegmatis* in the presence of DMSO.

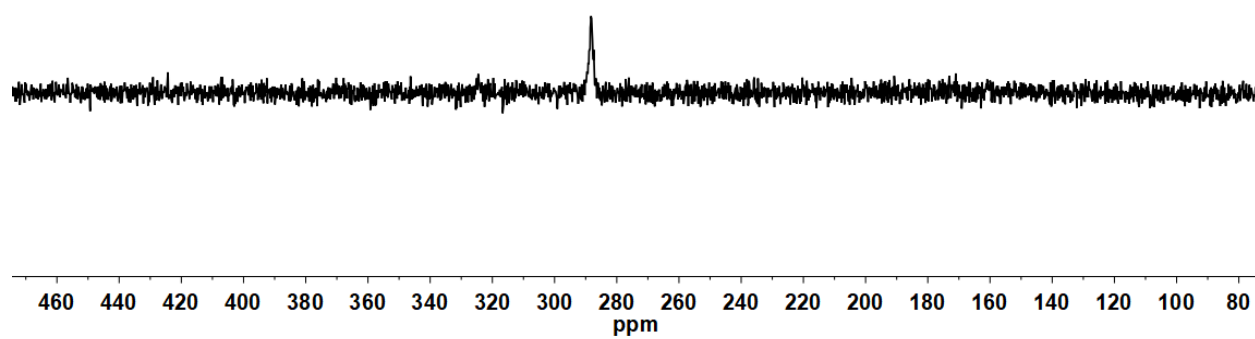


**Figure S4.** The  $^{51}\text{V}$  NMR spectrum of  $[\text{VO}(\text{Hshed})(4\text{NO}_2)]$  in  $\text{CD}_3\text{CN}$ .

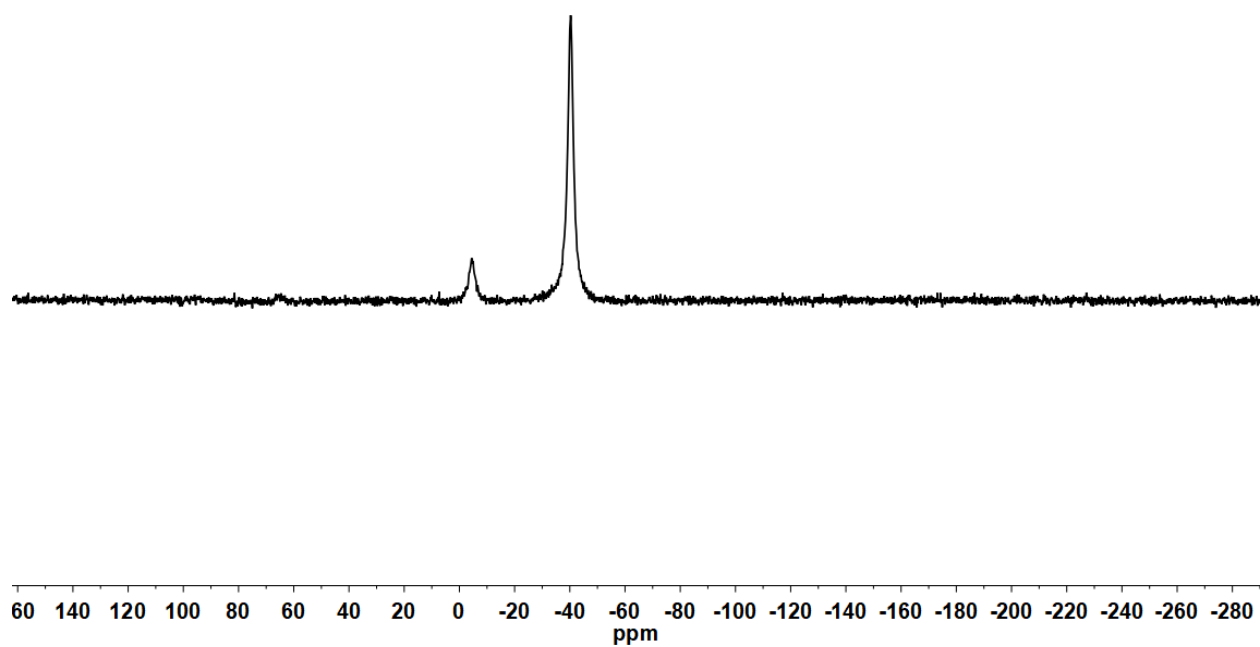


**Figure S5.** The  $^{51}\text{V}$  NMR spectrum of  $[\text{VO}(\text{Hshed})(\text{CN})]$  in  $\text{CD}_3\text{CN}$ .

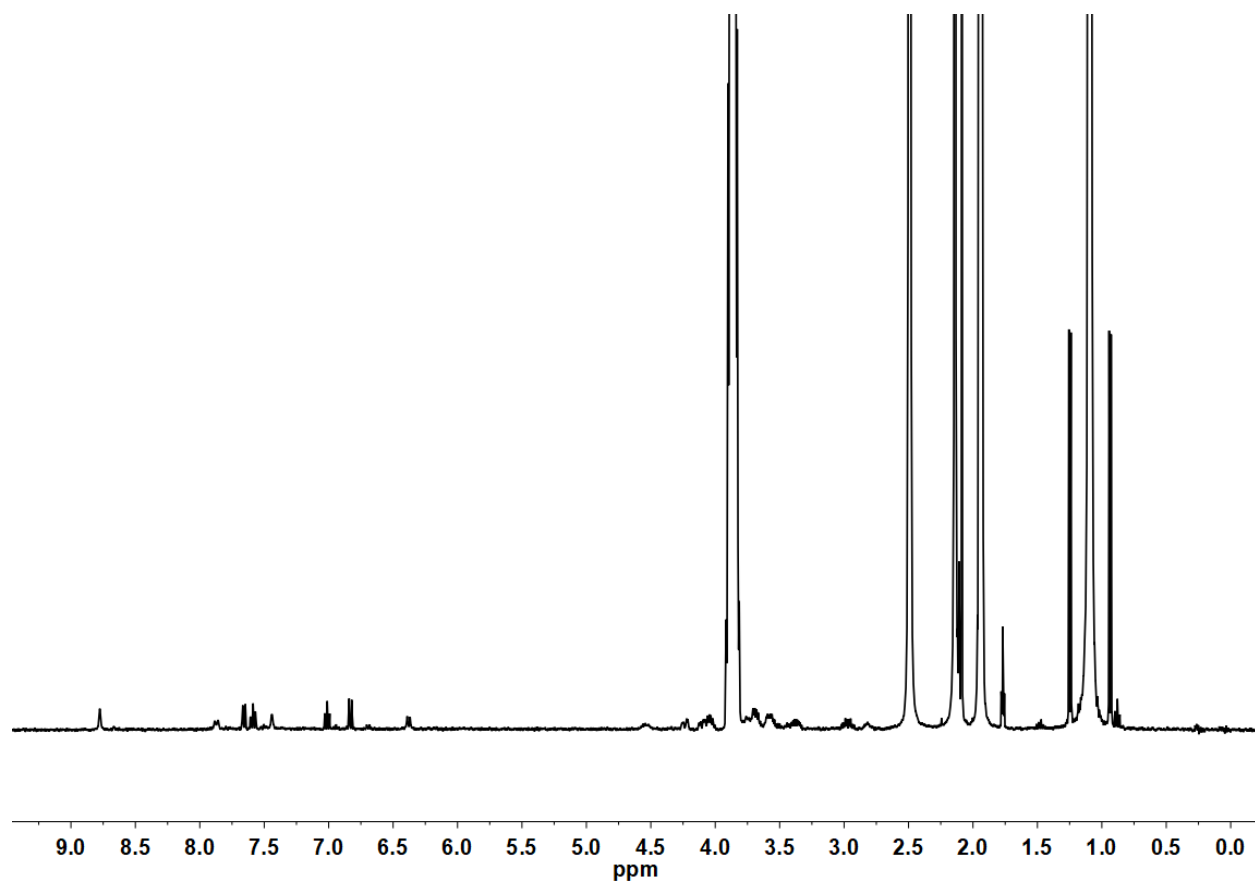




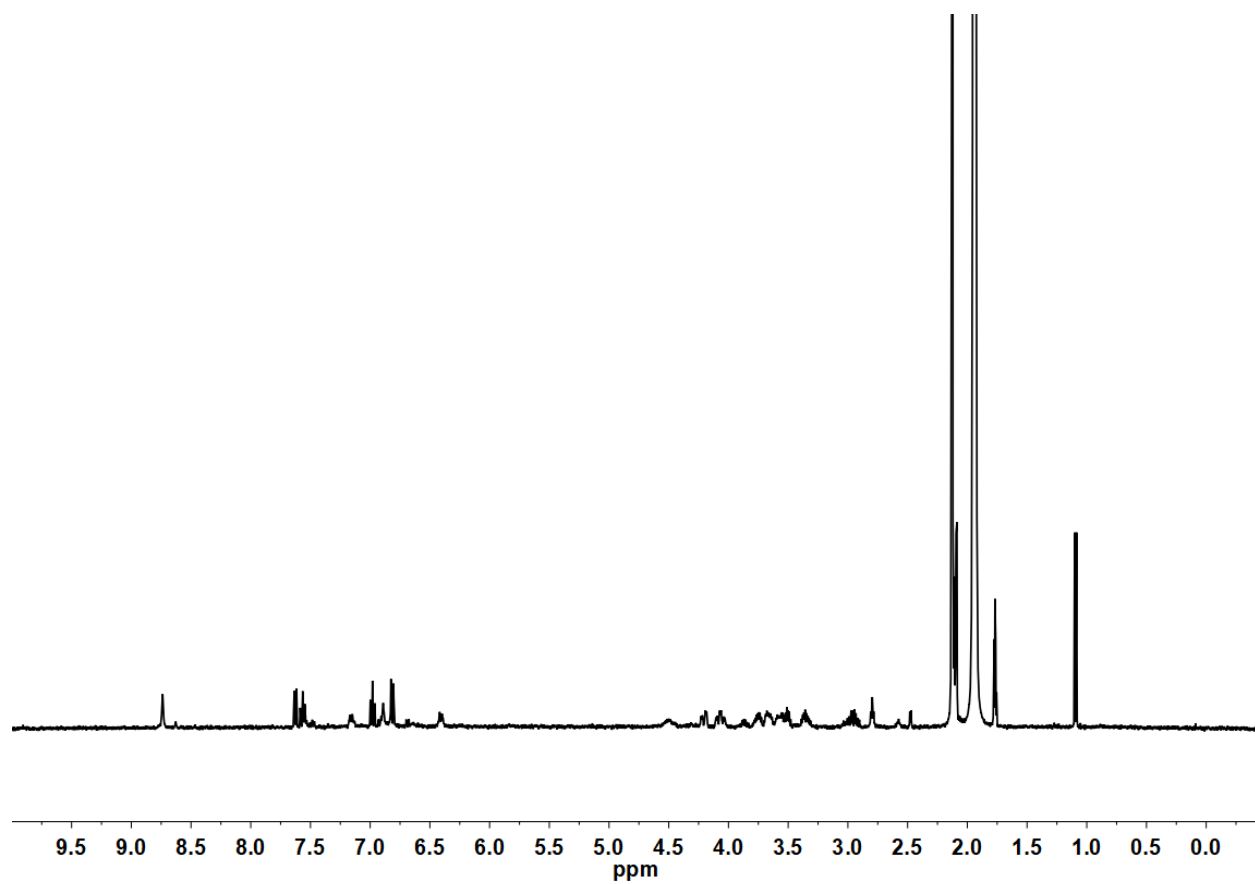
**Figure S6.** The  $^{51}\text{V}$  NMR spectrum of  $[\text{VO}(\text{Hshed})(3\text{OMet})]$  in  $\text{CD}_3\text{CN}$ .



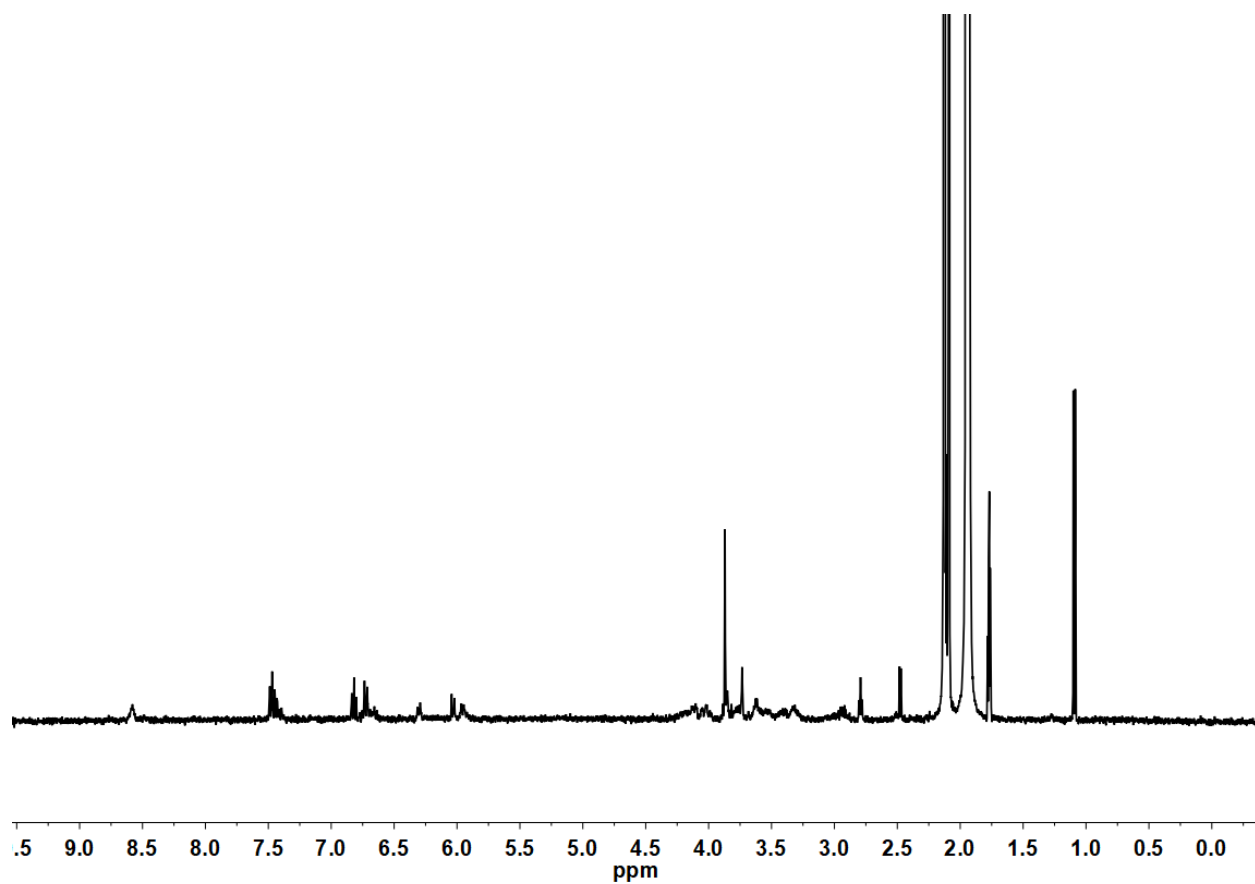
**Figure S7.** The  $^{51}\text{V}$  NMR spectrum of  $[\text{VO}(\text{Hshed})(\text{Coum})]$  in  $\text{CD}_3\text{CN}$ .



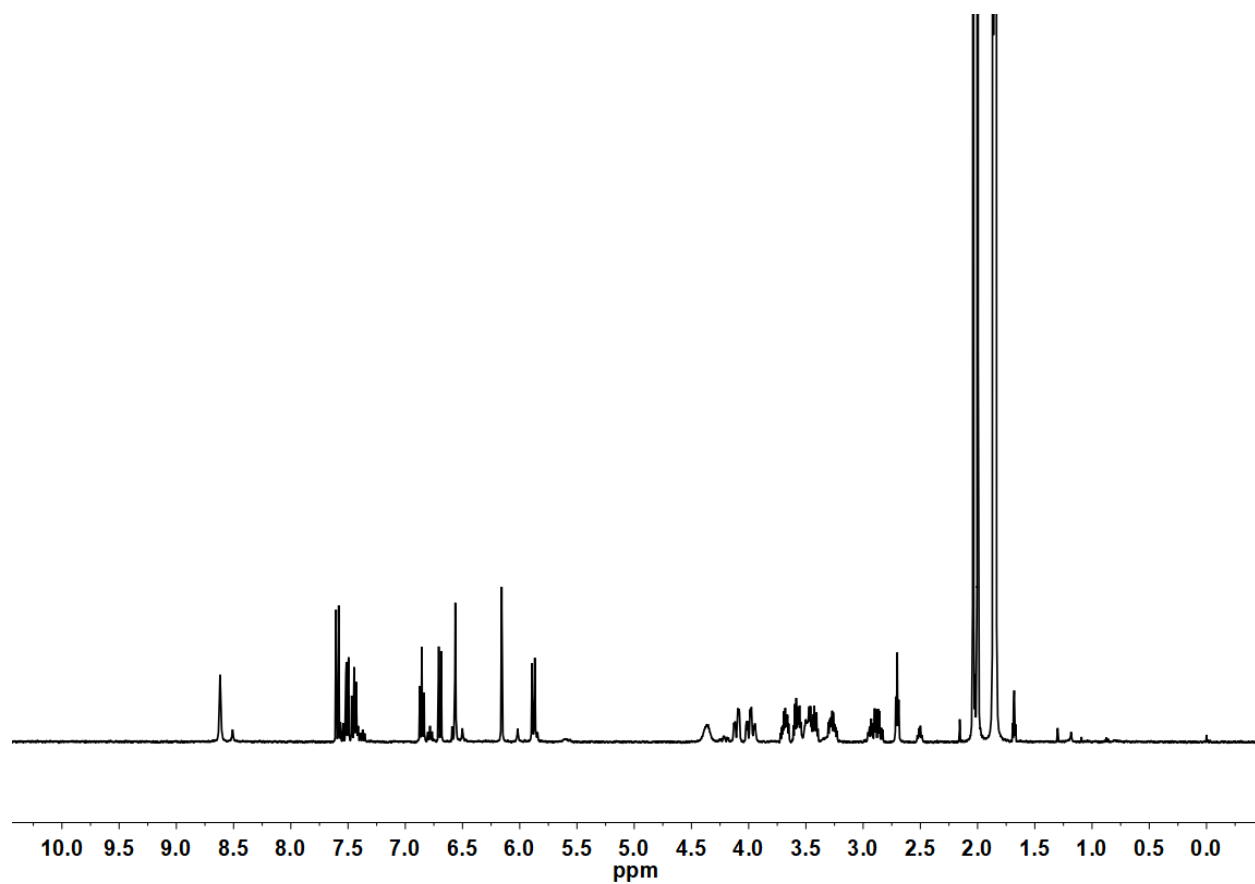
**Figure S8.** The  $^1\text{H}$  NMR spectrum of  $[\text{VO}(\text{Hshed})(4\text{NO}_2)]$  in  $\text{CD}_3\text{CN}$ .



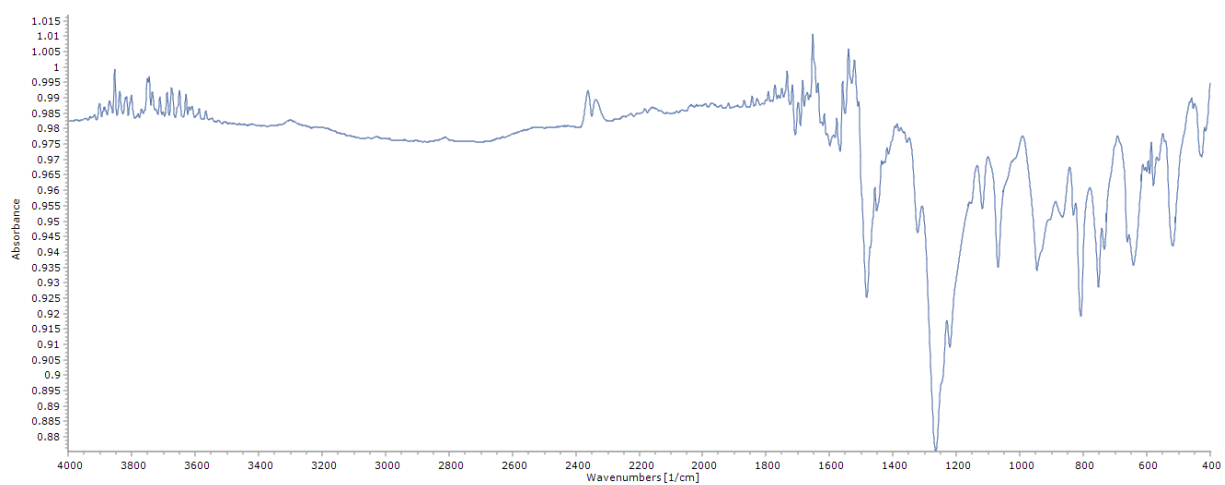
**Figure S9.** The  $^1\text{H}$  NMR spectrum of  $[\text{VO}(\text{Hshed})(\text{CN})]$  in  $\text{CD}_3\text{CN}$ .



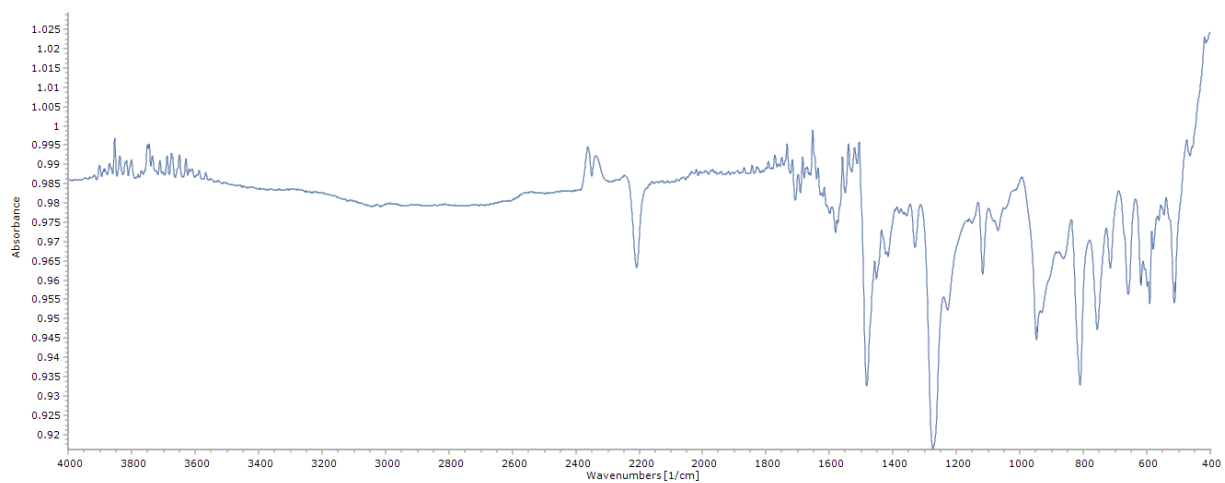
**Figure S10.** The  $^1\text{H}$  NMR spectrum of  $[\text{VO}(\text{Hshed})(3\text{OMet})]$  in  $\text{CD}_3\text{CN}$ .



**Figure S11.** The  $^1\text{H}$  NMR spectrum of  $[\text{VO}(\text{Hshed})(\text{Coum})]$  in  $\text{CD}_3\text{CN}$ .

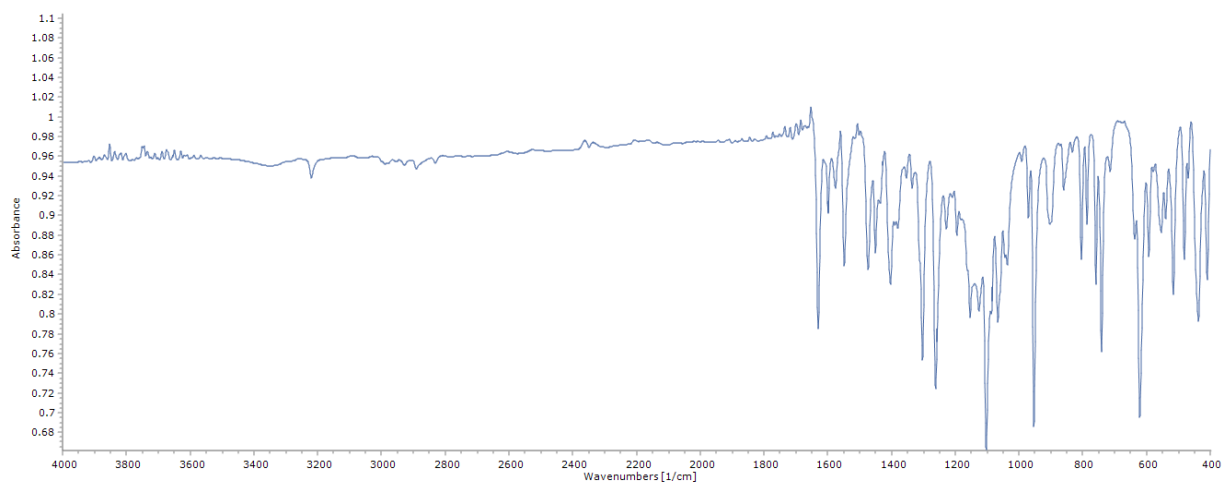


**Figure S12.** The IR spectrum of [VO(Hshed)(4NO<sub>2</sub>)].

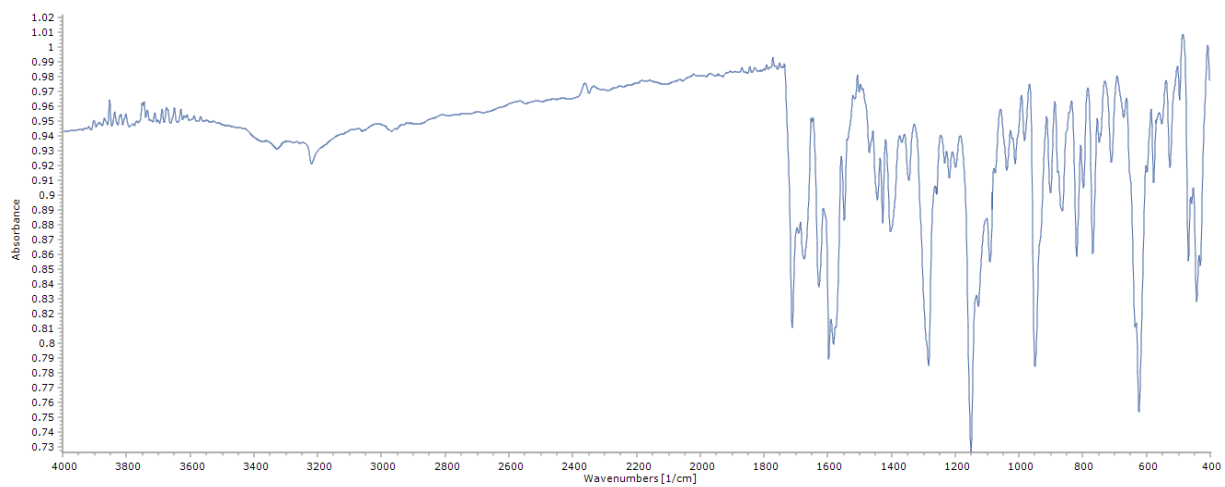


**Figure S13.** The IR spectrum of [VO(Hshed)(CN)].

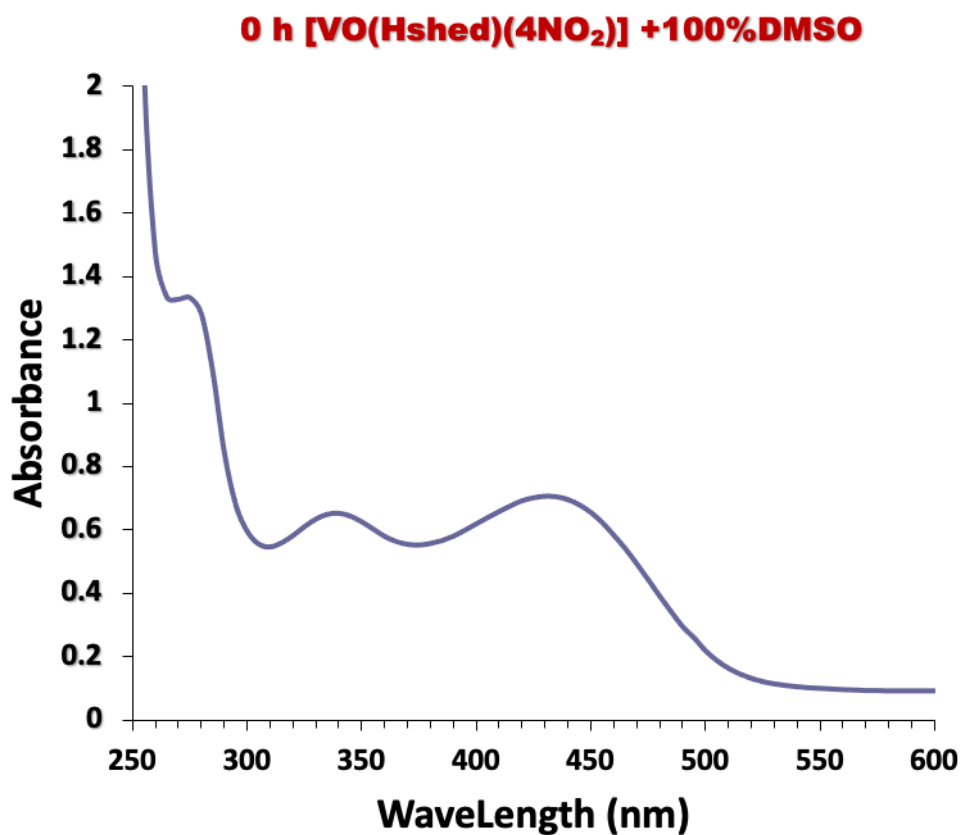




**Figure S14.** The IR spectrum of [VO(Hshed)(3OMet)].

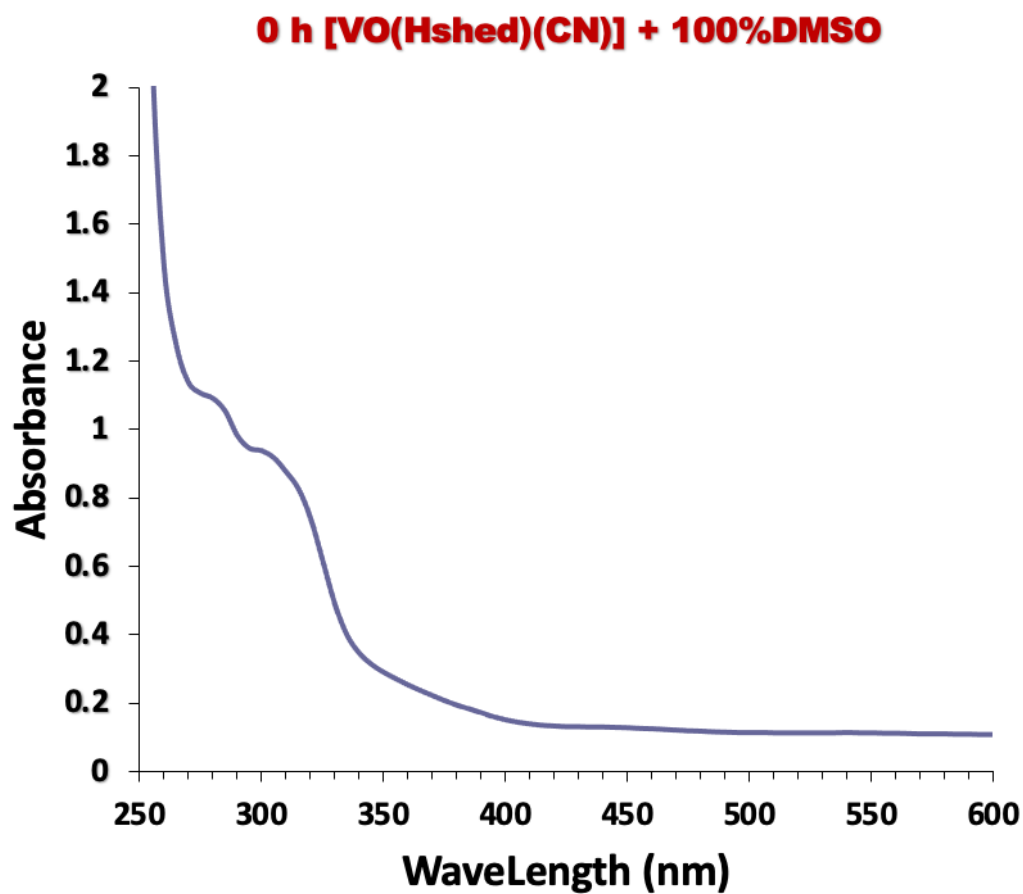


**Figure S15.** The IR spectrum of [VO(Hshed)(Coum)].



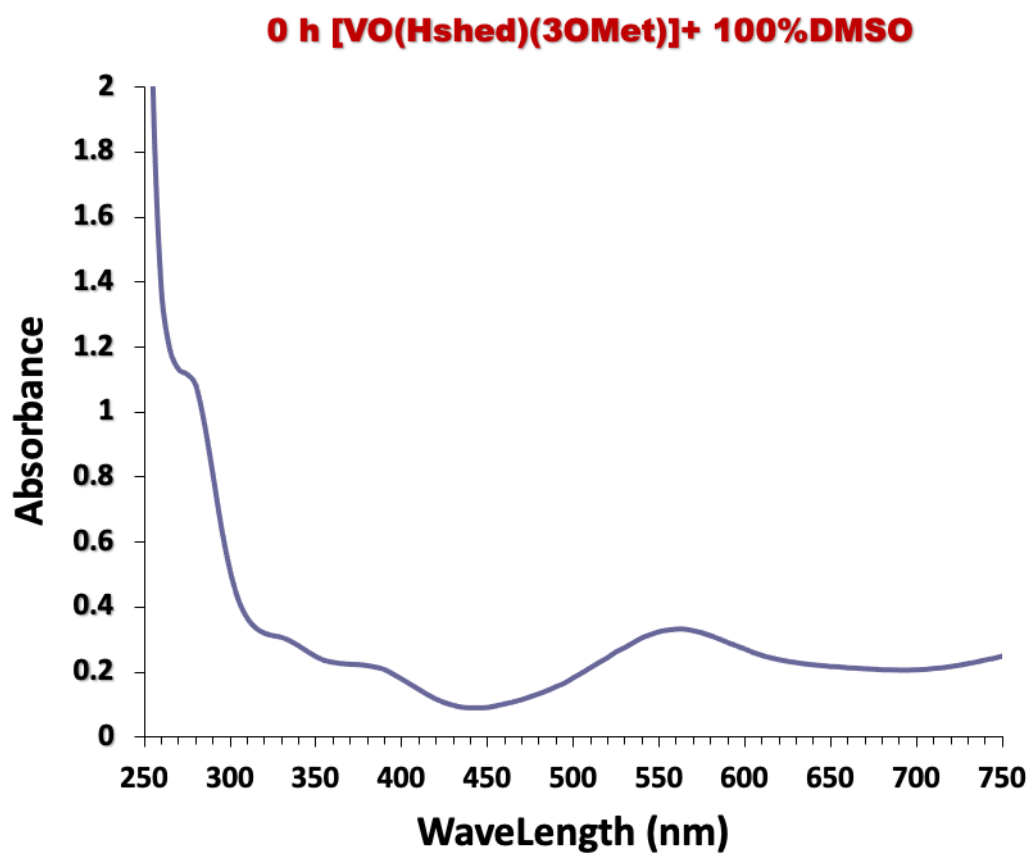
Complex	[complex] mM	Complex (100%DMSO) absorbance (nm)/ $\epsilon$ ( $\times 10^3 \text{ M}^{-1}$ )
[VO(Hshed)(4NO <sub>2</sub> )] (100% DMSO)	0.250	(280, 1.282, 5.1);
		(340, 0.653, 2.6);
		(435, 0.705, 2.8)

**Figure S16.** The UV-vis spectrum of [VO(Hshed)(4NO<sub>2</sub>)].



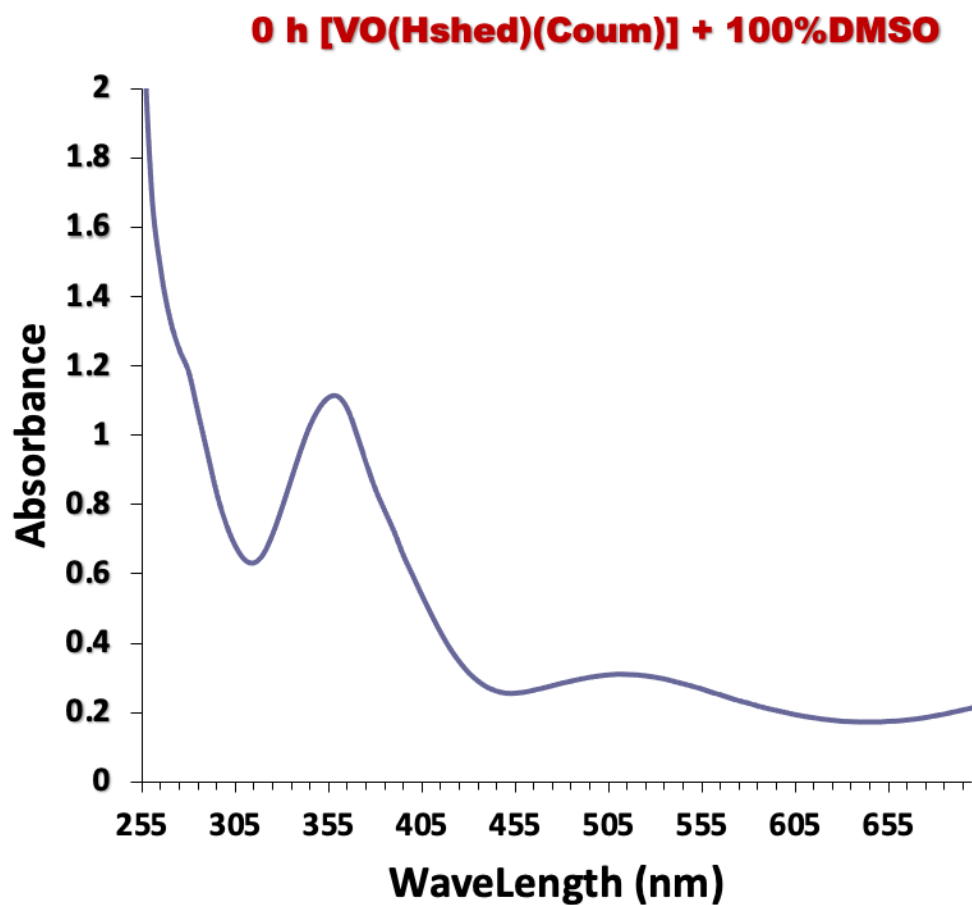
Complex	[complex] mM	Complex (100%DMSO) absorbance (nm)/ $\epsilon$ ( $\times 10^3 \text{ M}^{-1}$ )
([VO(Hshed)(CN)] (100% DMSO))	0.250	(280, 1.091, 4.4);
		(305, 0.917, 3.7)

**Figure S17.** The UV-vis spectrum of [VO(Hshed)(CN)].



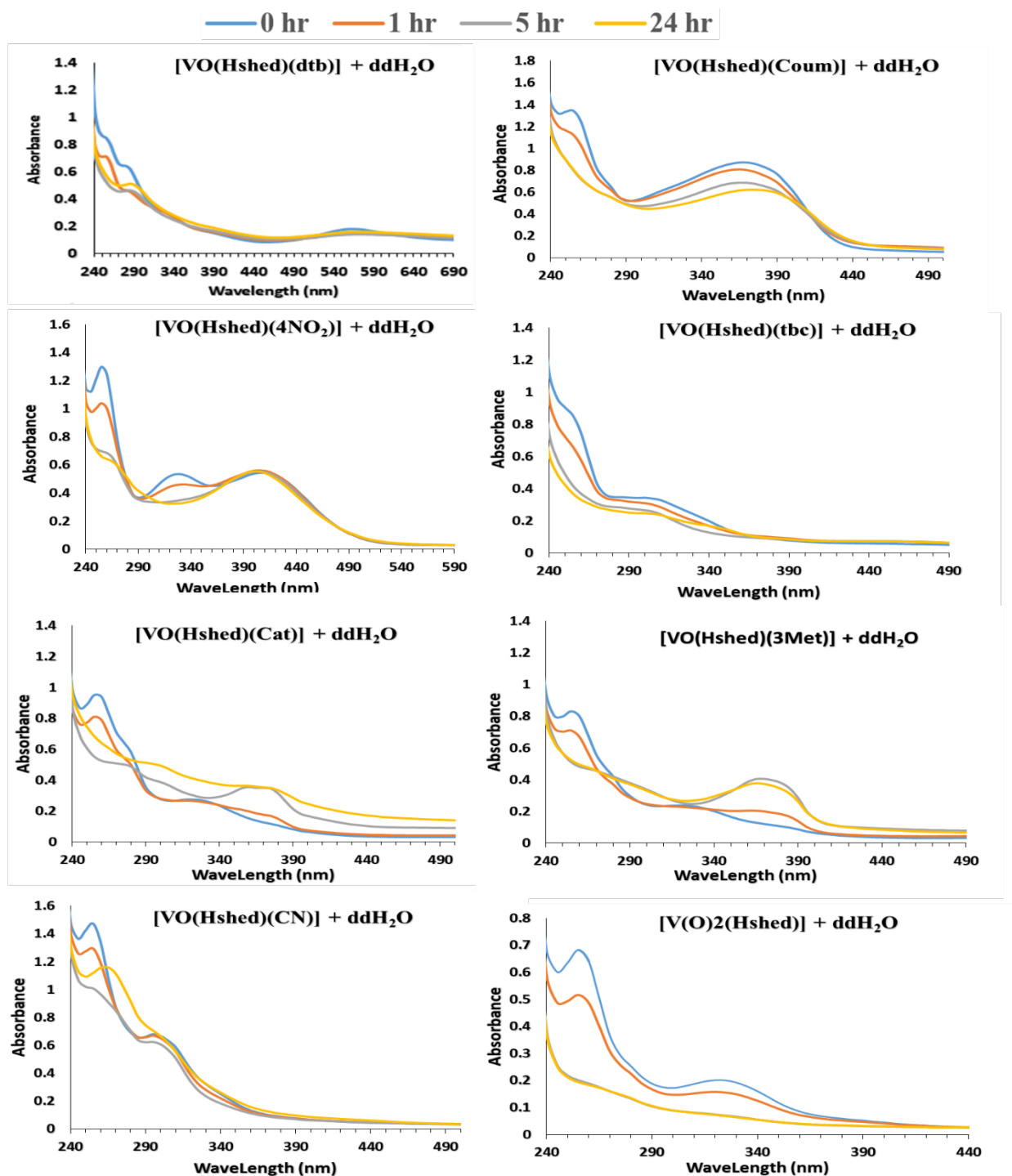
Complex	[complex] mM	Complex (100%DMSO) absorbance (nm)/ $\epsilon$ ( $\times 10^3 \text{ M}^{-1}$ )
[VO(Hshed)(3OMet)] / (100%DMSO) /	0.250	(280, 1.086, 4.3);
		(335, 0.298, 1.2);
		(390, 0.21, 0.84);
		(565, 0.334, 1.3)

**Figure S18.** The UV-vis spectrum of [VO(Hshed)(3OMet)].



Complex	[complex] mM	Complex (100%DMSO) absorbance (nm)/ $\epsilon$ ( $\times 10^3 \text{ M}^{-1}$ )
[VO(Hshed)(Coum)] (100% DMSO)	0.250	(280, 1.183, 4.7);
		(360, 1.112, 4.4);
		(520, 0.309, 1.2)

Figure S19. The UV-vis spectrum of [VO(Hshed)(Coum)].



**Figure S20.** The UV-vis spectra are recorded of all the complexes at 0.250 mM in ddH<sub>2</sub>O as a function of time (0, 1, 5, 24 h).

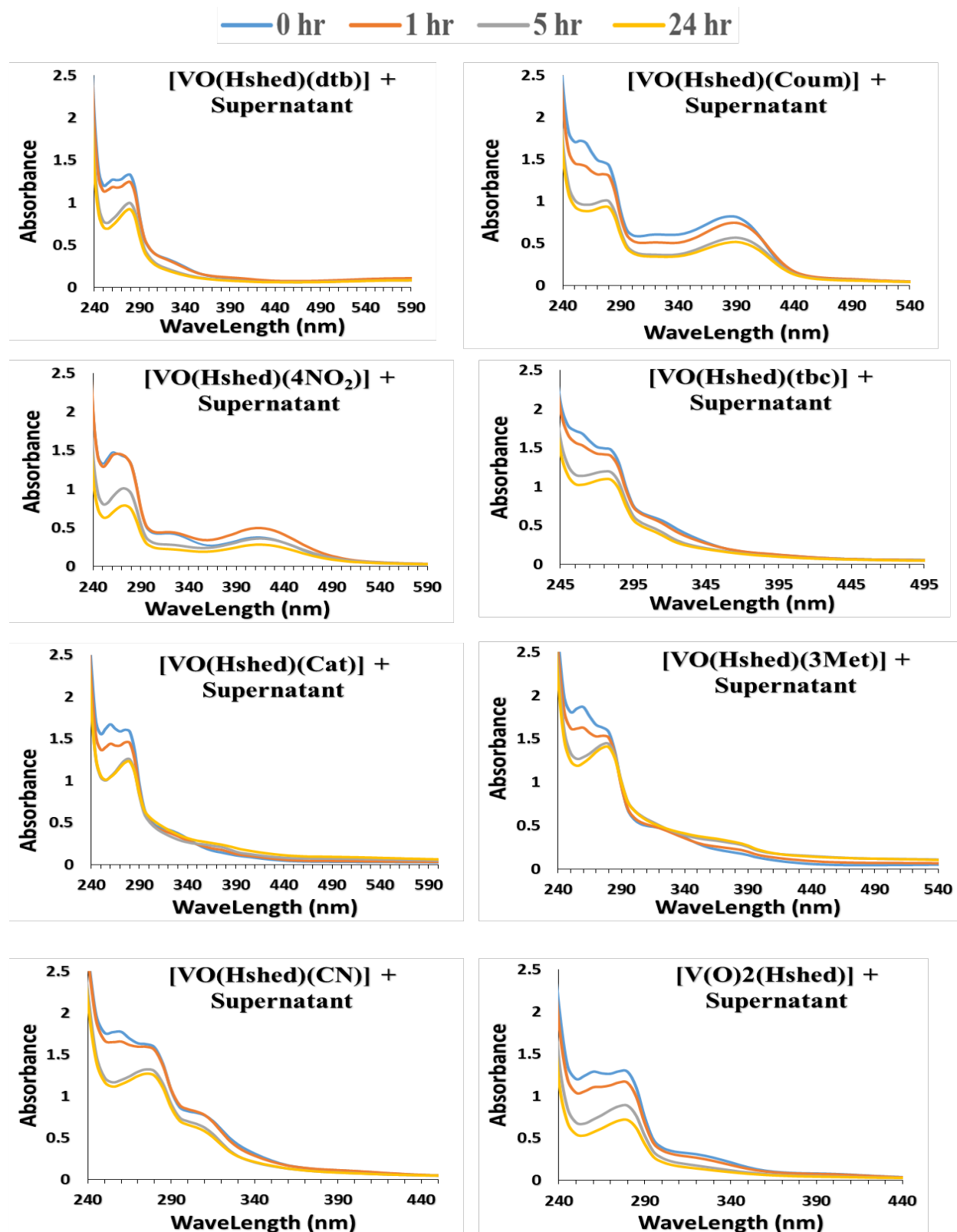
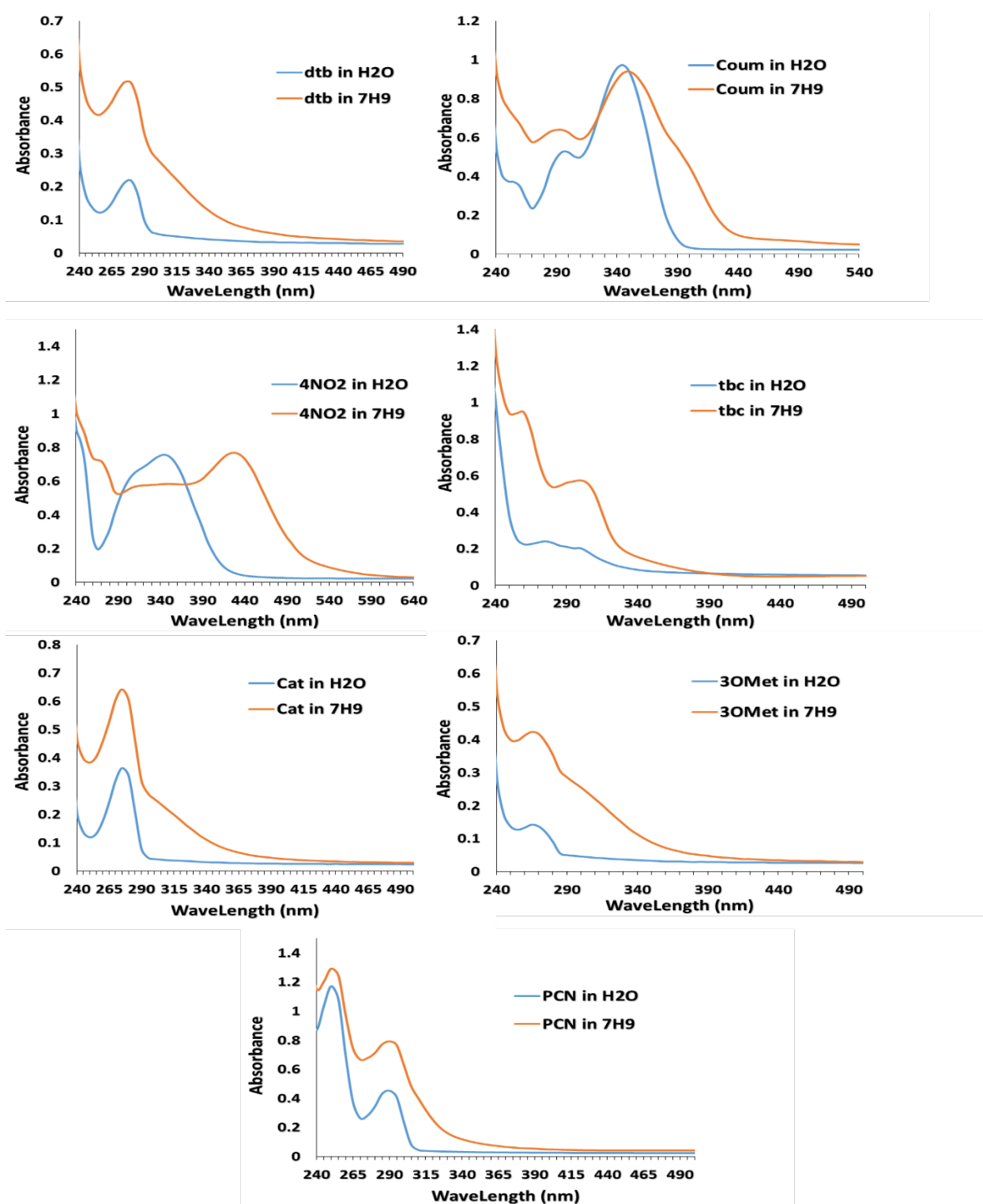
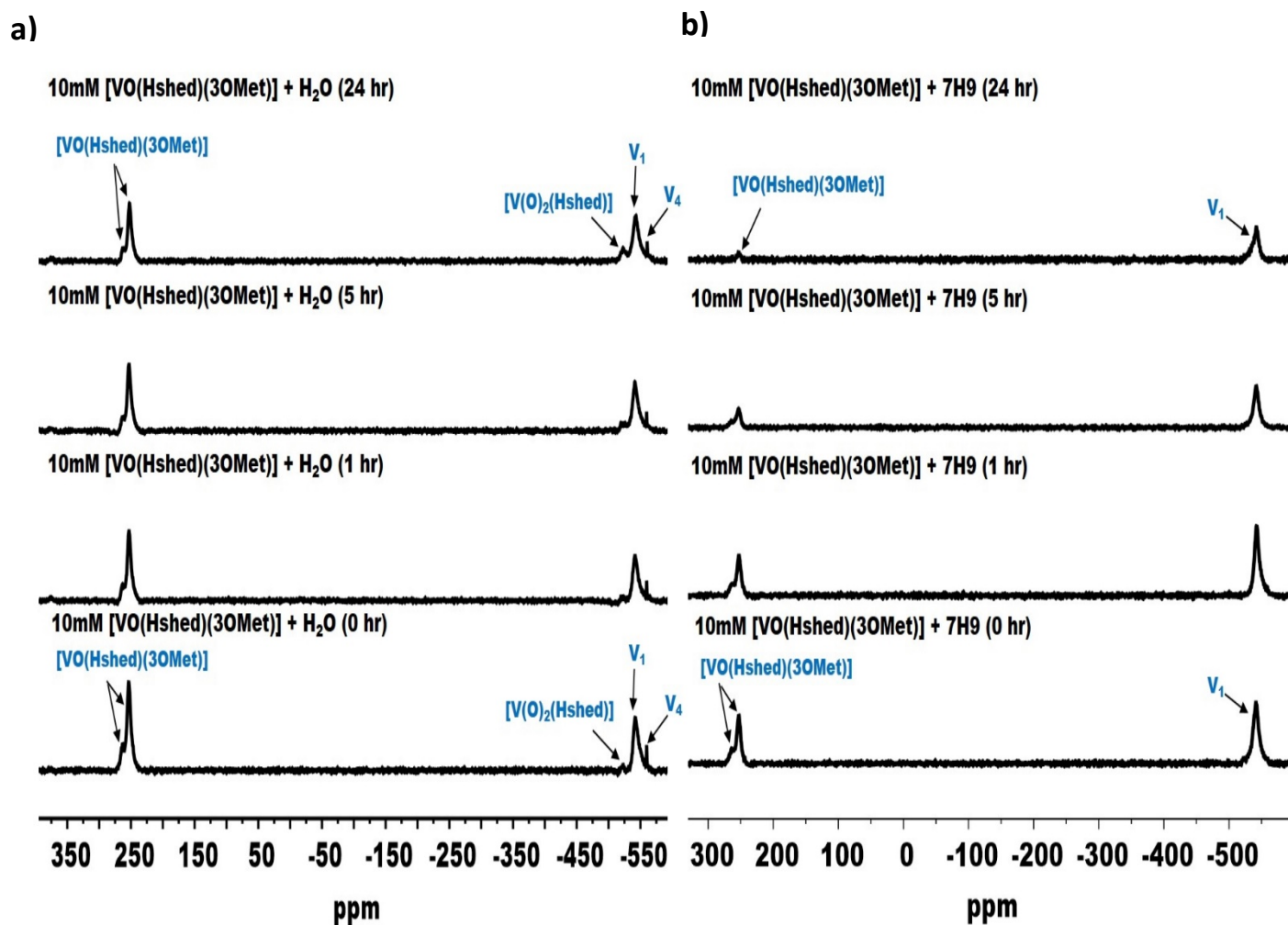


Figure S21. The UV-vis spectra are recorded of all the complexes at 0.250 mM in supernatant fraction as a function of time (0,1, 5, 24 h).

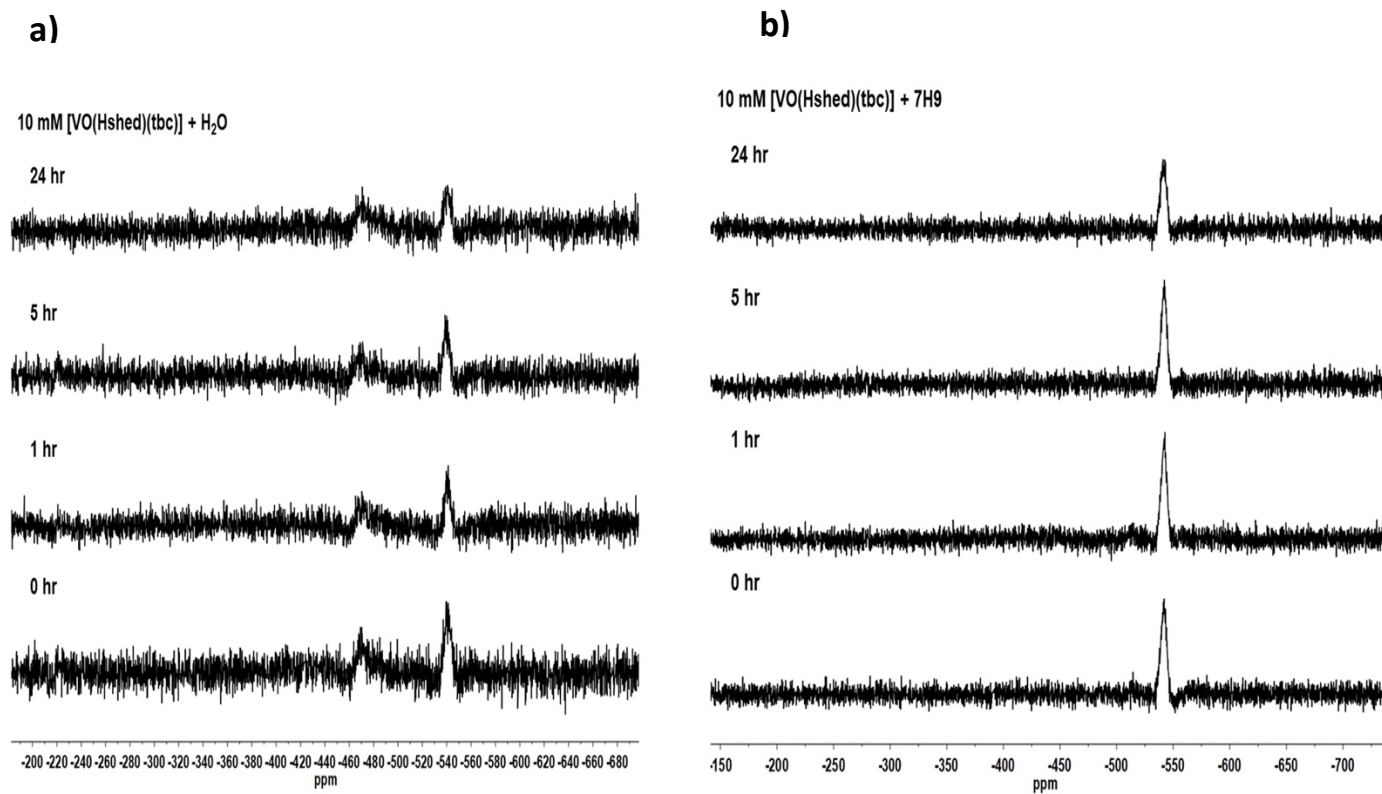




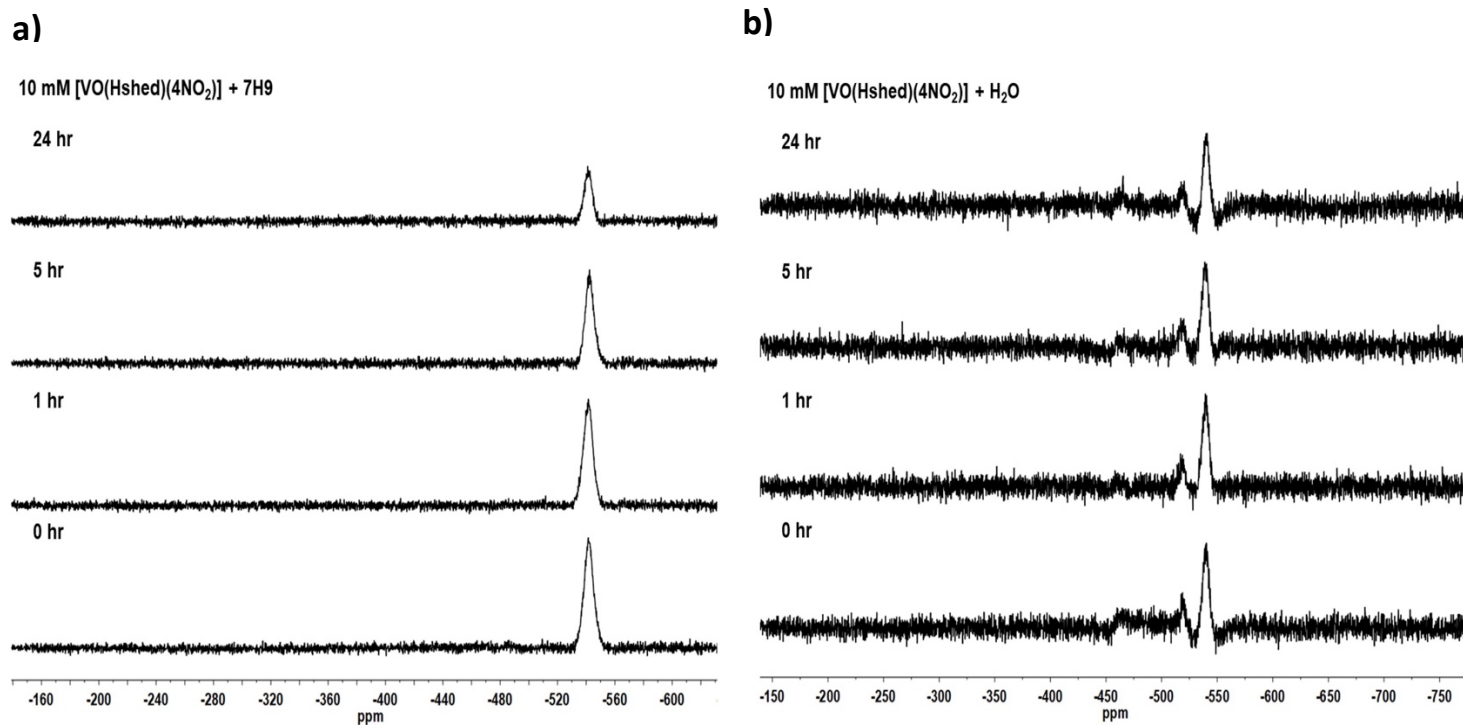
**Figure S22.** The UV-vis spectra are recorded of all the free ligands at 0.250 mM in ddH<sub>2</sub>O and 7H9 medium at time zero (0 h).



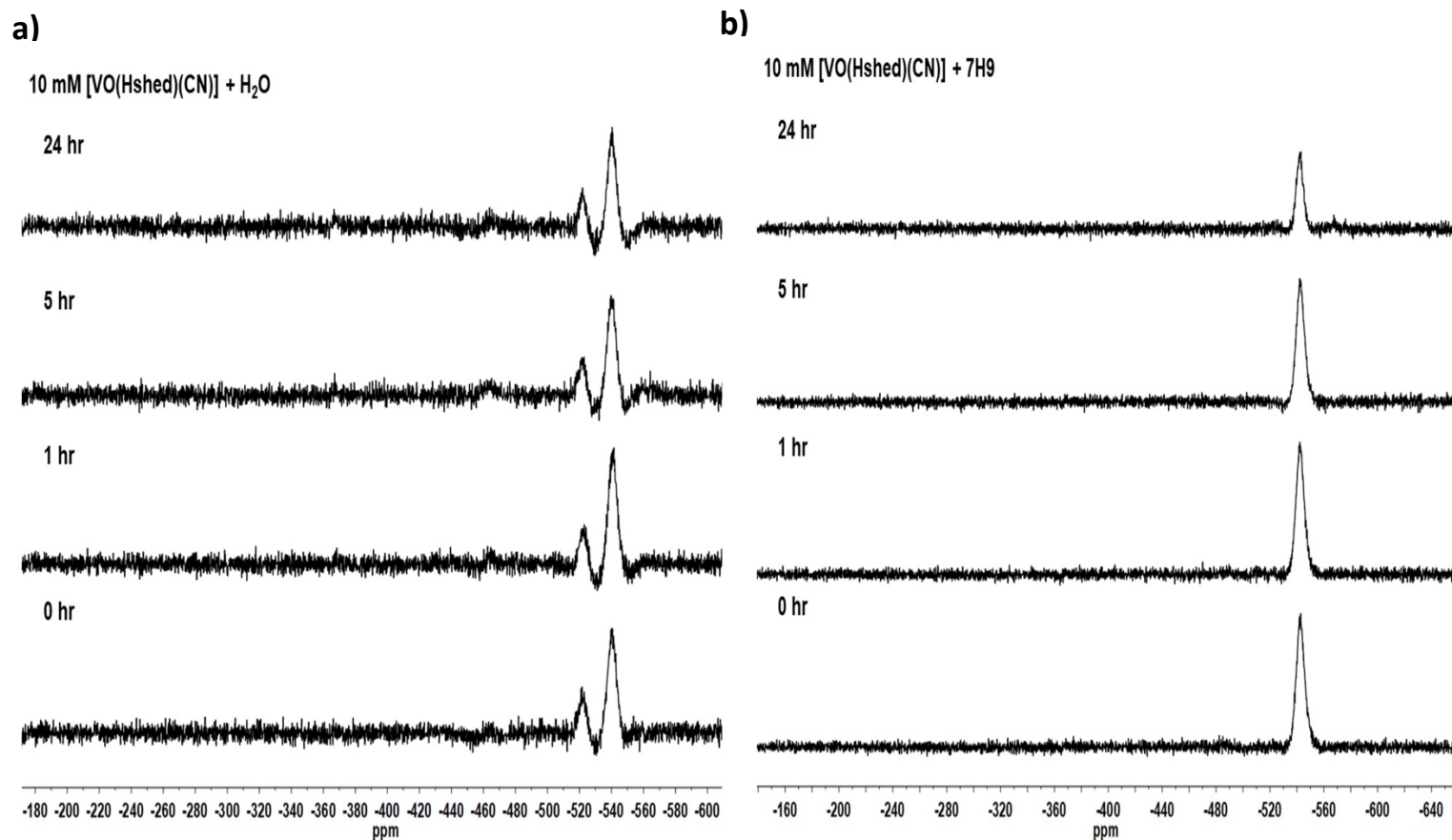
**Figure S23.** The  $^{51}\text{V}$  NMR spectra of 10mM  $[\text{VO}(\text{Hshed})(3\text{OMet})]$  at 0, 1, 5, and 24 h time points in a) 50:50  $\text{H}_2\text{O}$ :DMSO and in b) 50:50 7H9 growth medium: DMSO.



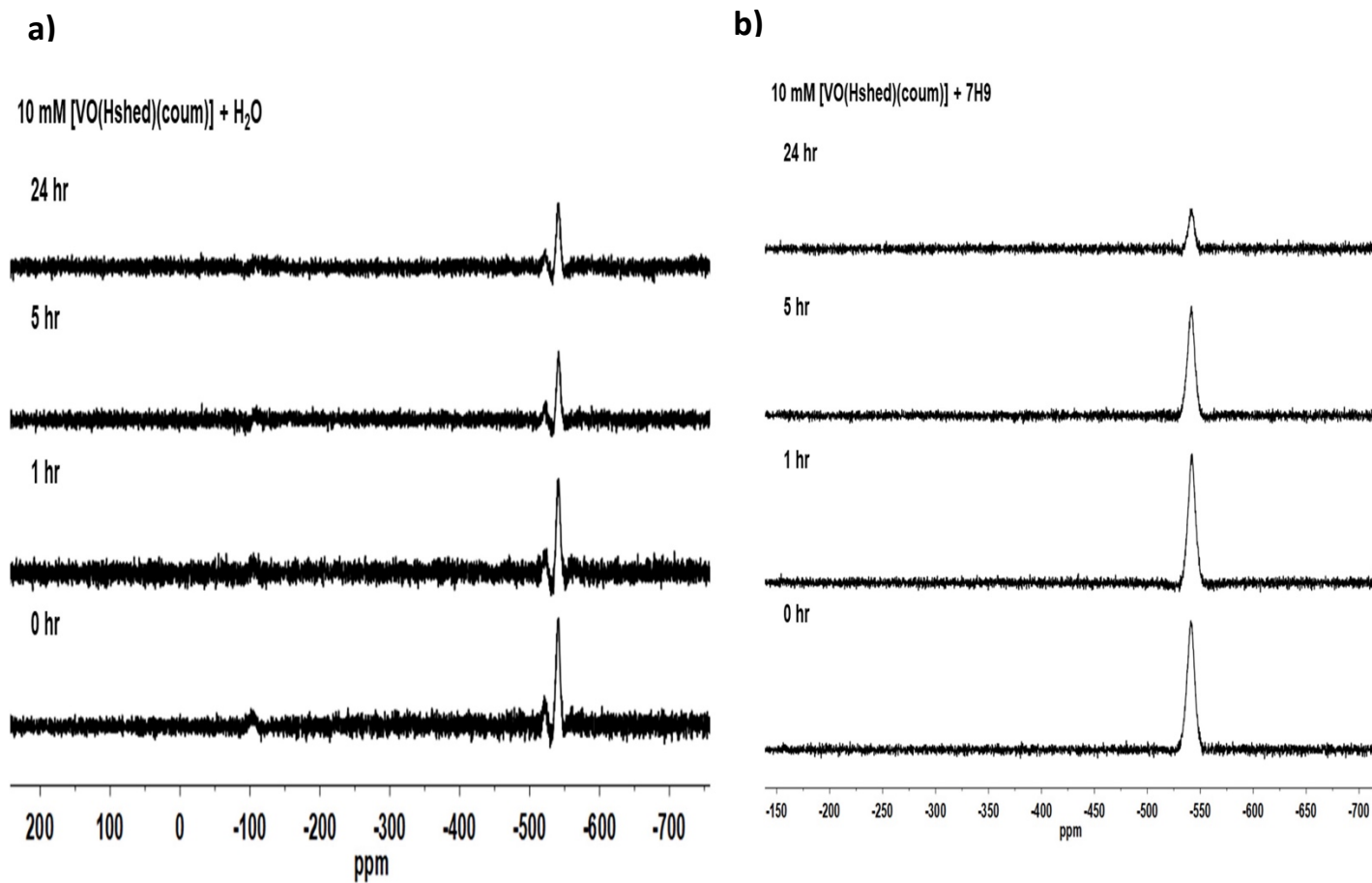
**Figure S24.** The  $^{51}\text{V}$  NMR spectra of 10mM [VO(Hshed)(tbc)] at 0, 1, 5, and 24 h time points in **a)** 50:50 H<sub>2</sub>O:DMSO and in **b)** 50:50 7H9 growth medium: DMSO.



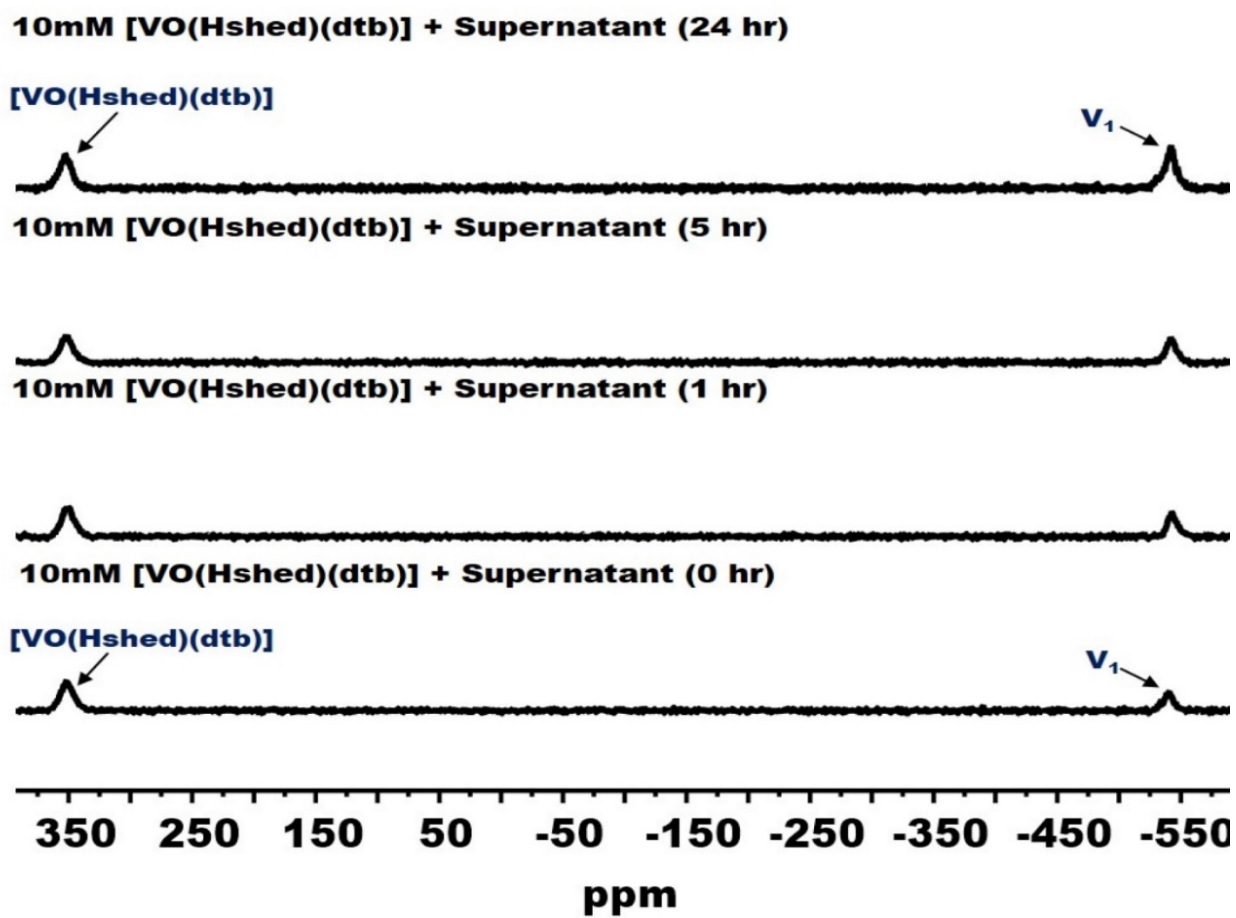
**Figure S25.** The <sup>51</sup>V NMR spectra of 10mM [VO(Hshed)(4NO<sub>2</sub>)] at 0, 1, 5, and 24 h time points in **a)** 50:50 H<sub>2</sub>O:DMSO and in **b)** 50:50 7H9 growth medium: DMSO.



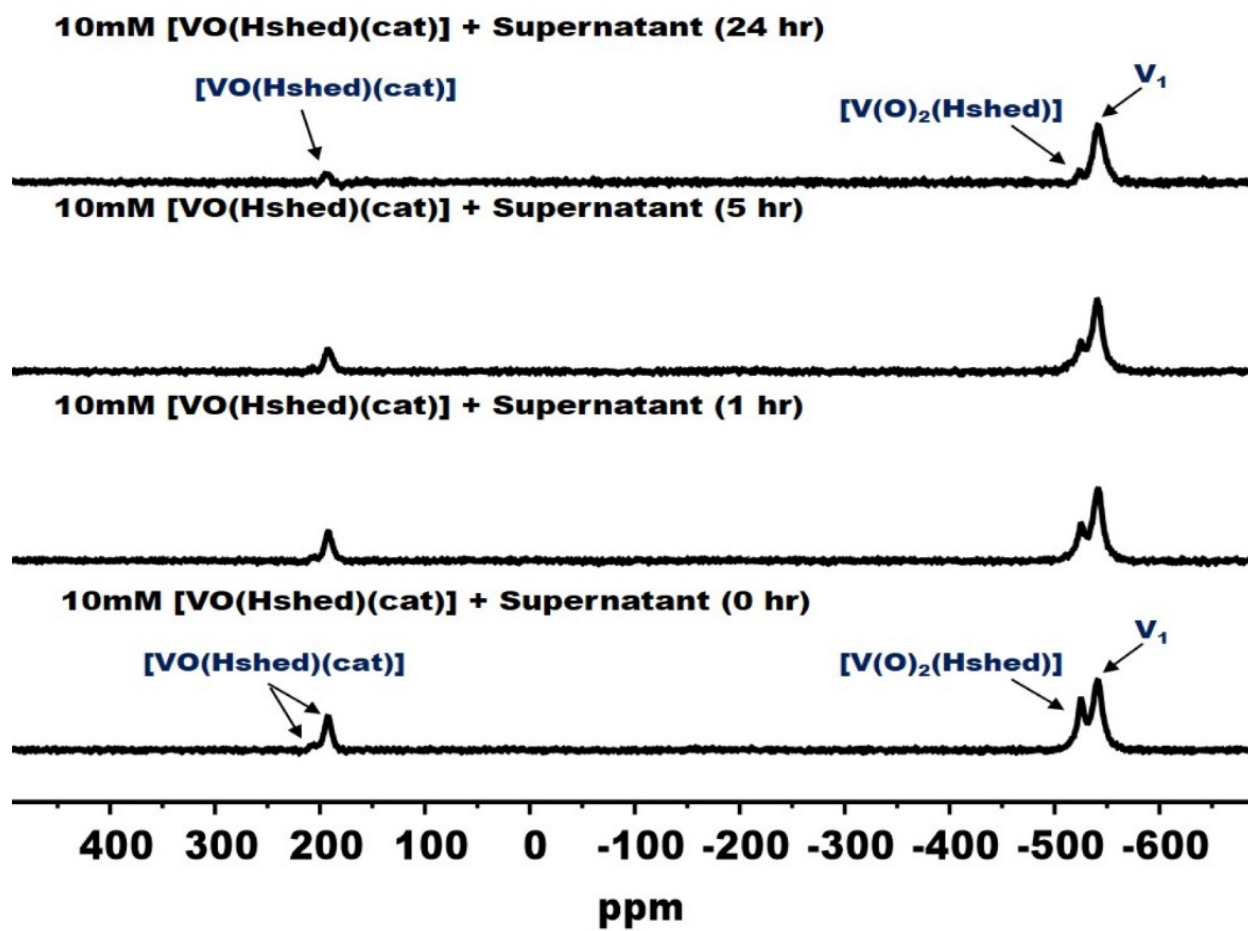
**Figure S26.** The  $^{51}\text{V}$  NMR spectra of 10mM [VO(Hshed)(CN)] at 0, 1, 5, and 24 h time points in **a)** 50:50 H<sub>2</sub>O:DMSO and in **b)** 50:50 7H9 growth medium: DMSO.



**Figure S27.** The  $^{51}\text{V}$  NMR spectra of 10mM [VO(Hshed)(Coum)] at 0, 1, 5, and 24 h time points in **a)** 50:50 H<sub>2</sub>O:DMSO and in **b)** 50:50 7H9 growth medium: DMSO.

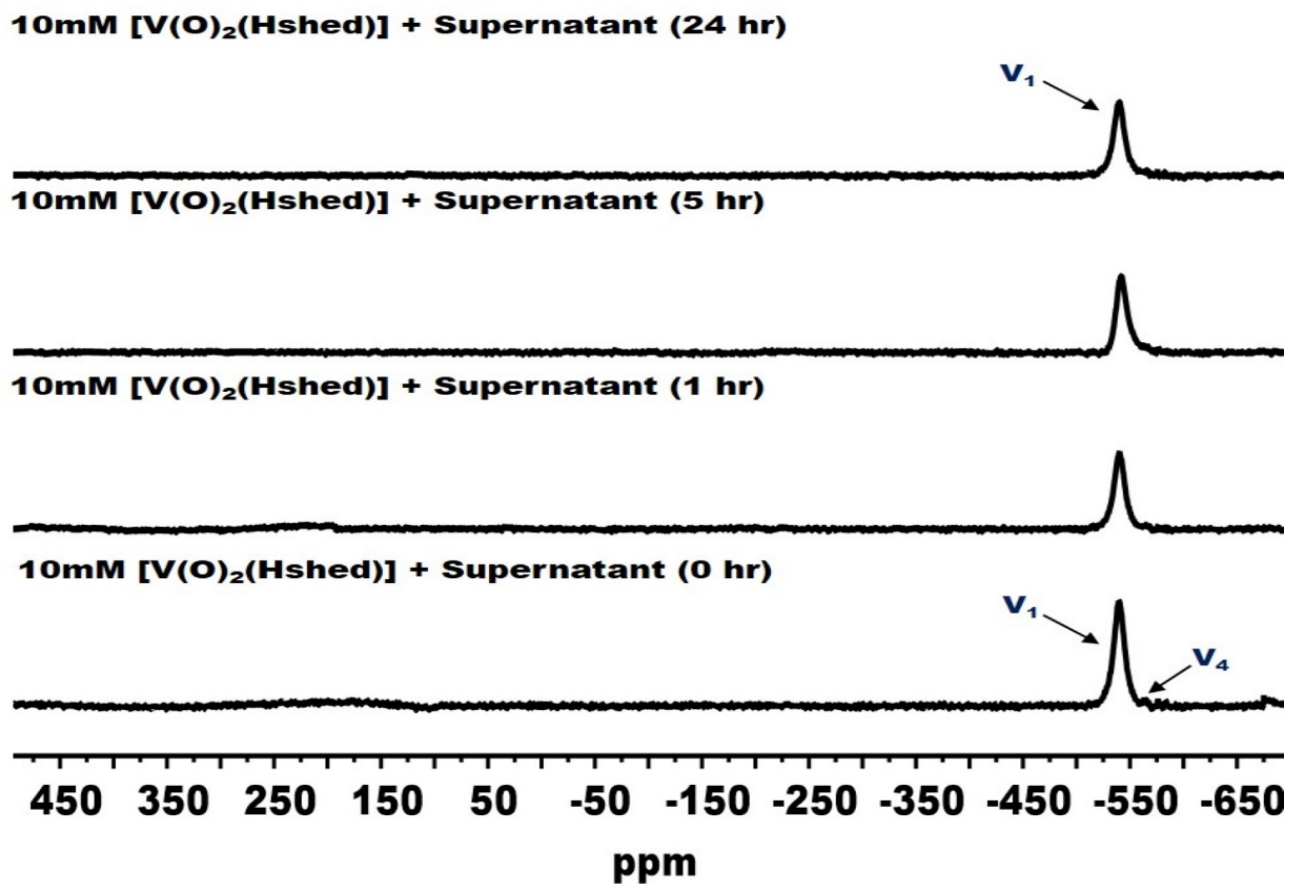


**Figure S28.** The  $^{51}\text{V}$  NMR spectra of 10mM [VO(Hshed)(dtb)] at 0, 1, 5, and 24 h time points in supernatant fraction.

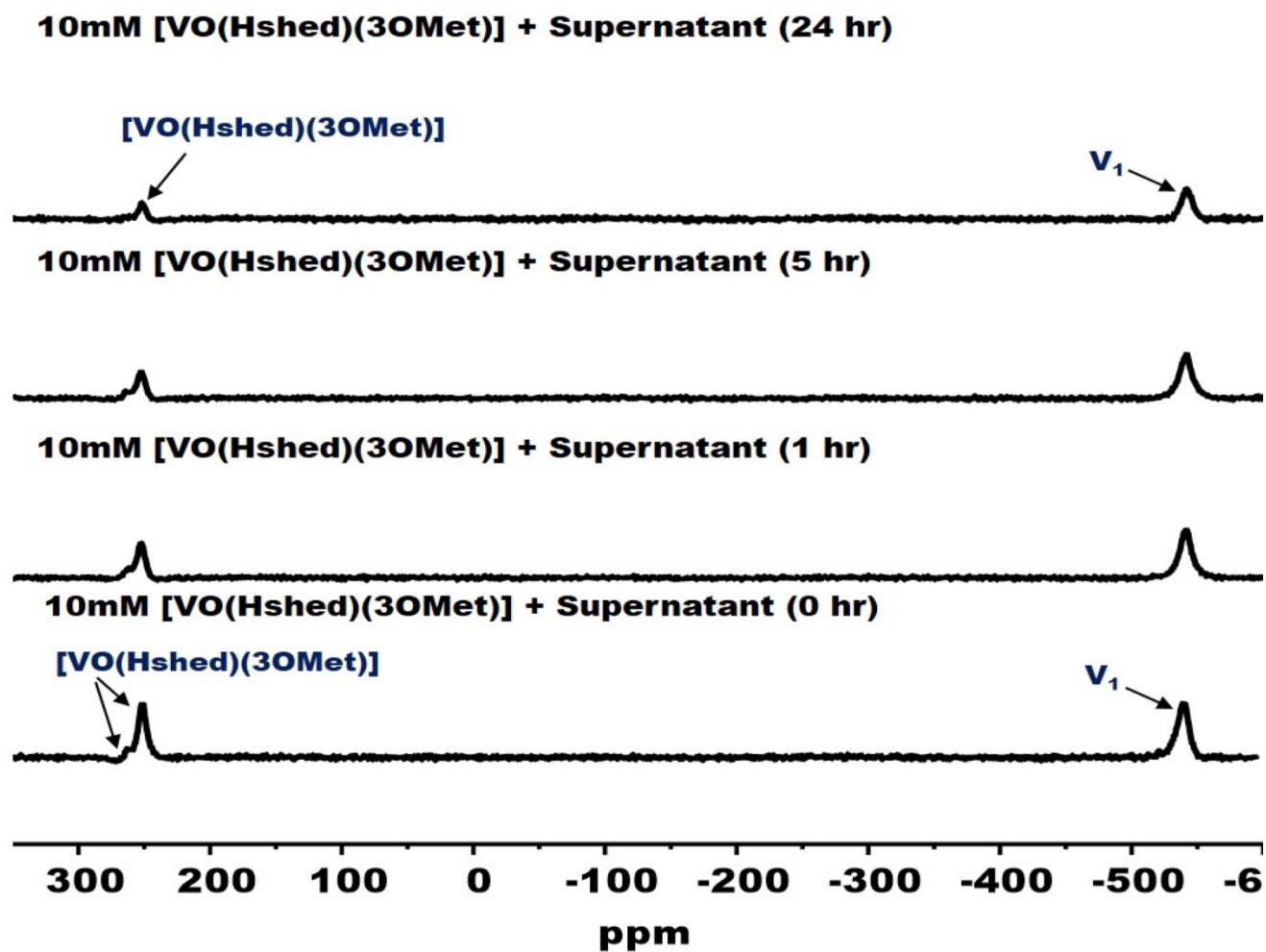


**Figure S29.** The  $^{51}\text{V}$  NMR spectra of 10mM  $[\text{VO}(\text{Hshed})(\text{cat})]$  at 0, 1, 5, and 24 h time points in supernatant fraction.





**Figure S30.** The <sup>51</sup>V NMR spectra of 10mM [V(O)<sub>2</sub>(Hshed)] at 0, 1, 5, and 24 h time points in supernatant fraction.



**Figure S31.** The  $^{51}\text{V}$  NMR spectra of 10mM [VO(Hshed)(3OMet)] at 0, 1, 5, and 24 h time points in supernatant fraction.