

## Supplementary Information

### A new synthetic methodology in the preparation of bimetallic chalcogenide clusters via cluster-to-cluster transformations

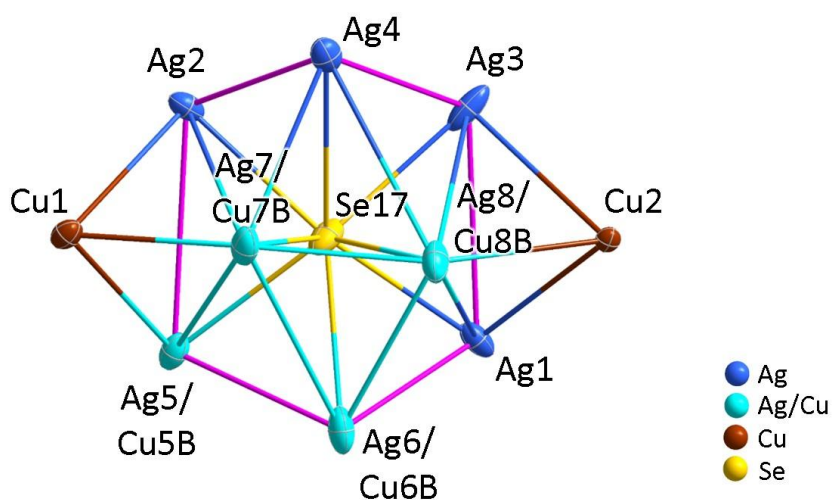
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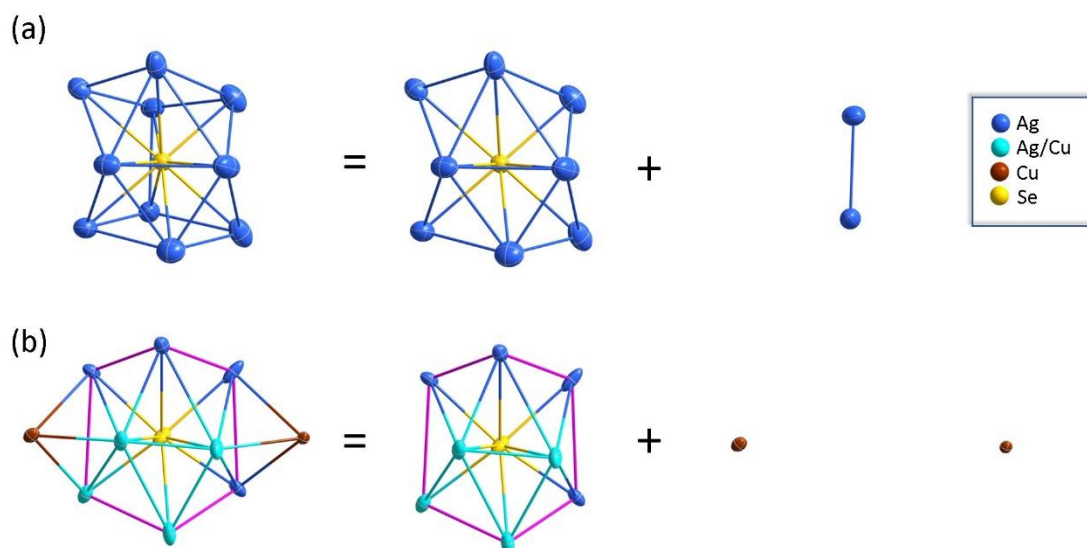
<http://faculty.ndhu.edu.tw/~cwl/index.htm>



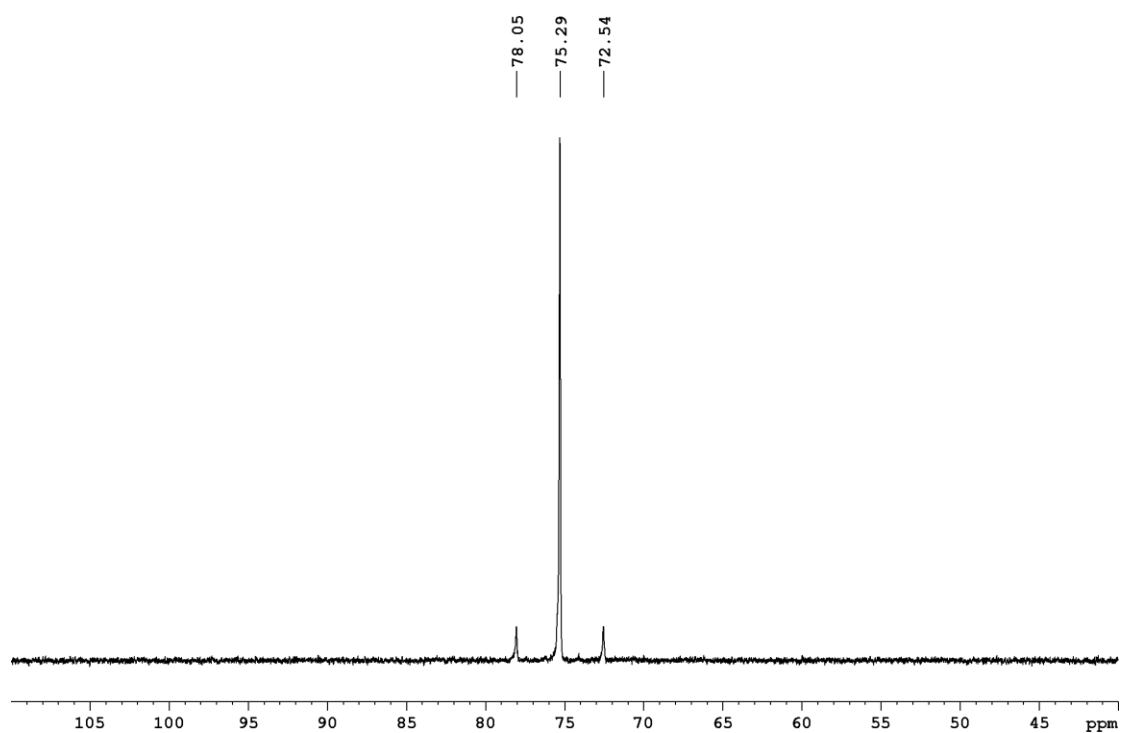
| Atomic site     | Cu1 | Cu2 | Ag1 | <sup>[a]</sup> Ag2/<br>Ag2B | Ag3 | Ag4 | Ag5/<br>Cu5B | Ag6/<br>Cu6B | Ag7/<br>Cu7B | Ag8/<br>Cu8B | Total occupancy |
|-----------------|-----|-----|-----|-----------------------------|-----|-----|--------------|--------------|--------------|--------------|-----------------|
| Cu occupancy    | 1   | 1   | 0   | 0                           | 0   | 0   | 0.2          | 0.2          | 0.5          | 0.5          | 3.4             |
| Ag occupancy    | 0   | 0   | 1   | 0.8/<br>0.2                 | 1   | 1   | 0.8          | 0.8          | 0.5          | 0.5          | 6.6             |
| Total occupancy | 1   | 1   | 1   | 1                           | 1   | 1   | 1            | 1            | 1            | 1            | 10              |

<sup>[a]</sup> The Ag2 was disordered and split into two sites (Ag2 and Ag2B) with occupancy ratio 80:20. Herein, only the major distribution is discussed.

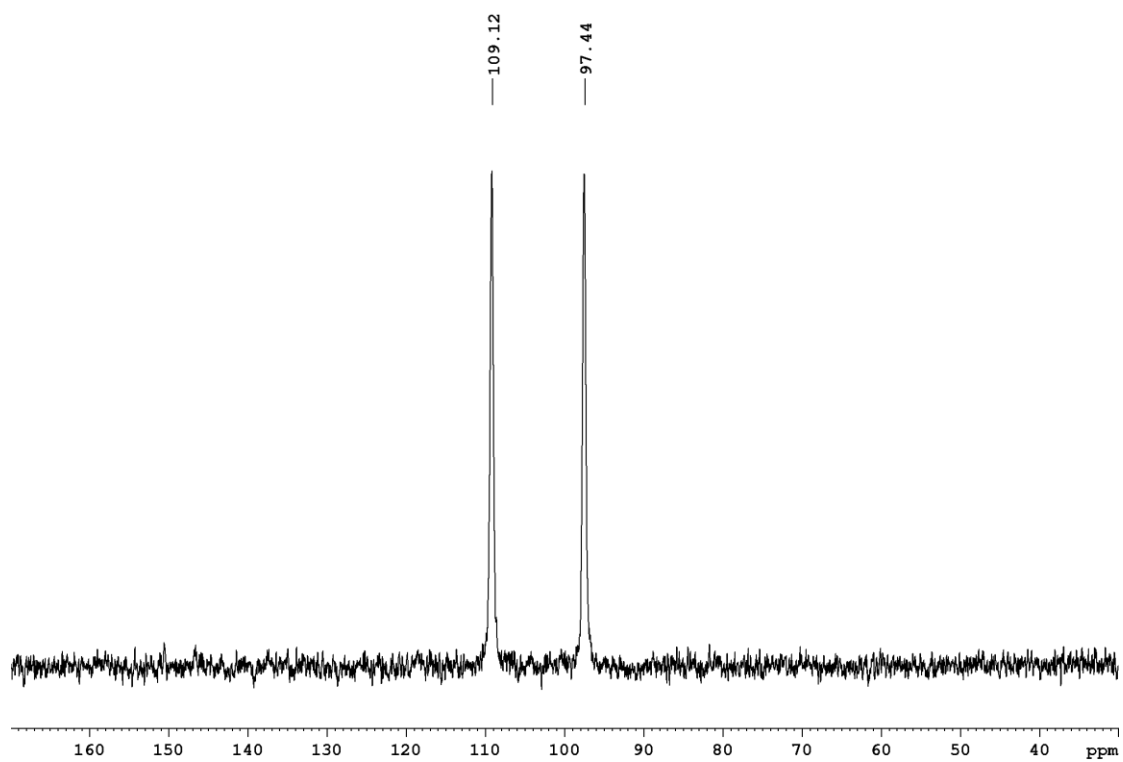
**Figure S1.** The M<sub>10</sub>(Se) framework of [3a]<sub>0.6</sub>[3b]<sub>0.4</sub>, (thermal ellipsoid plots were drawn at the 30 % probability level), with the refined occupancy ratios on each position.



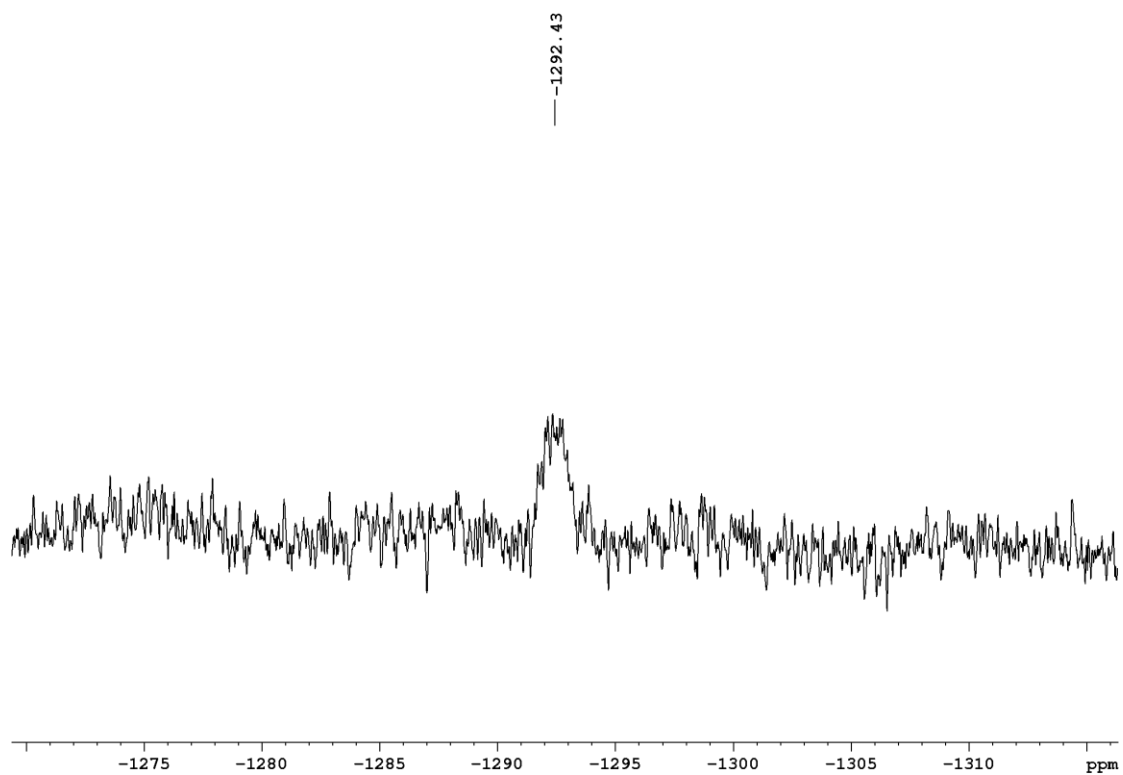
**Figure S2.** (a) The schematic representation of  $\text{Ag}_8(\text{Se})$  skeleton in **1** and (b)  $\text{M}_8(\text{Se})$  skeleton in  $[\mathbf{3a}]_{0.6}[\mathbf{3b}]_{0.4}$ .



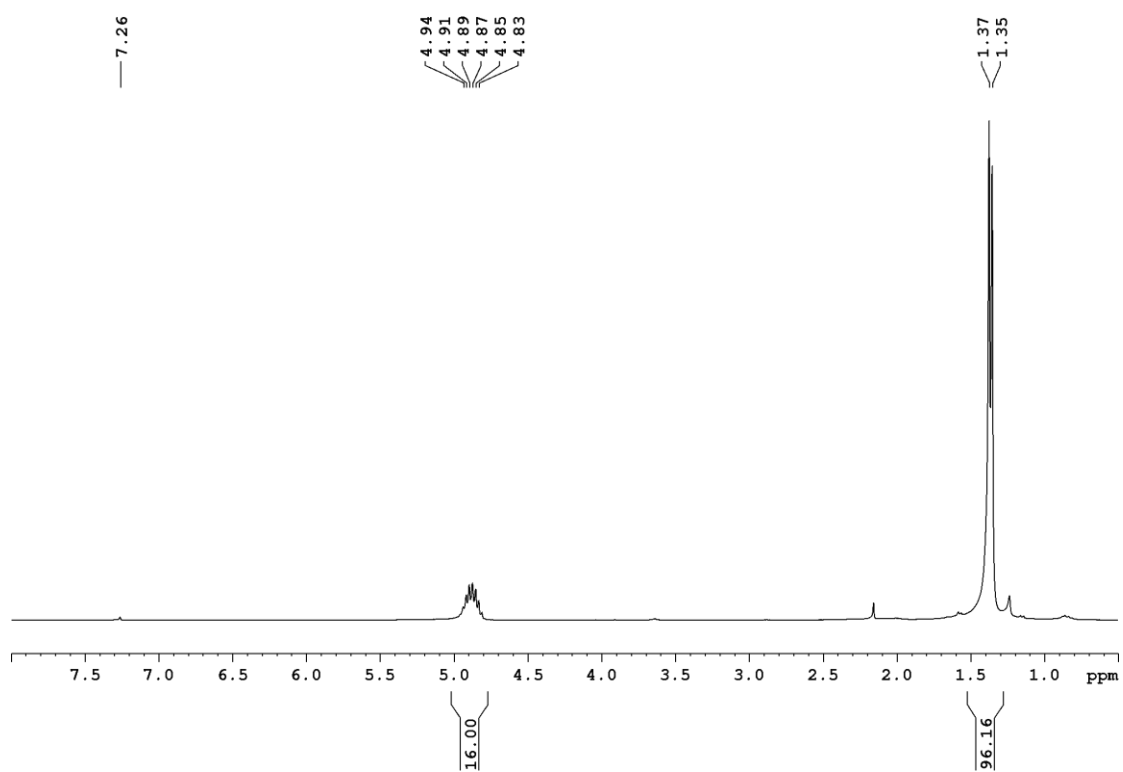
**Figure S3.**  $^{31}\text{P}\{^1\text{H}\}$  NMR spectrum of **2**.



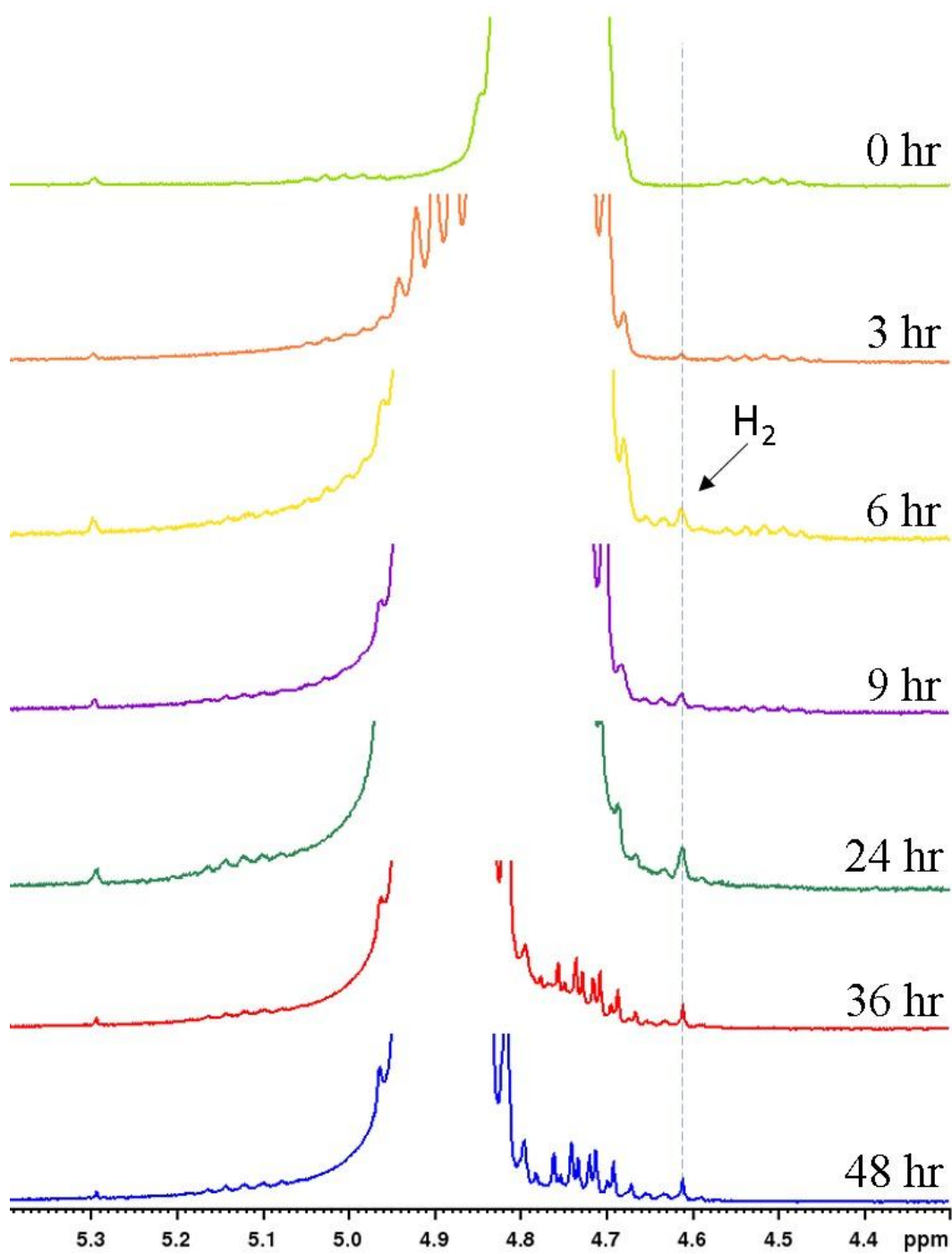
**Figure S4.**  $^{77}\text{Se}$  NMR spectrum of **2**.



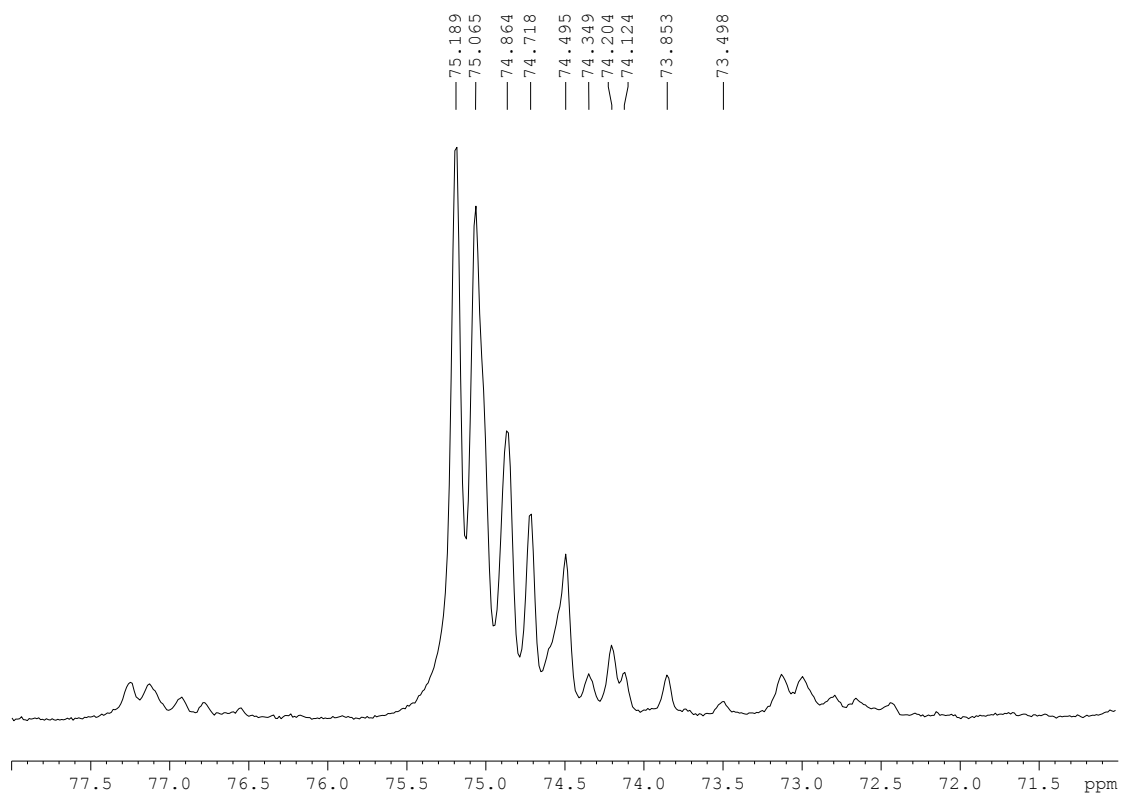
**Figure S5.**  $^{77}\text{Se}$  NMR spectrum of **2**.



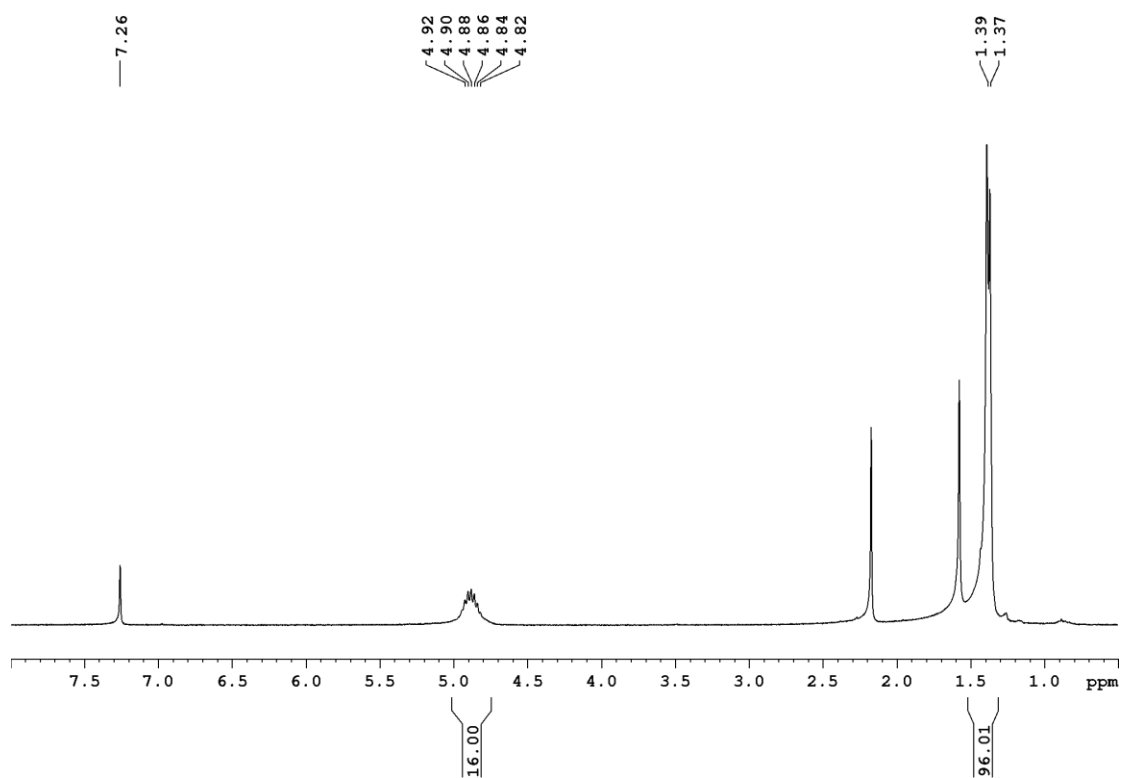
**Figure S6.**  $^1\text{H}$  NMR spectrum of **2**.



**Figure S7.** The magnified time-dependent  $^1\text{H}$  NMR spectrum during the reaction of 2.



**Figure S8.**  $^{31}\text{P}\{^1\text{H}\}$  NMR spectrum of **3**.



**Figure S9.**  $^1\text{H}$  NMR spectrum of **3**.