

Supplementary information for

Synthesis of Cyanooxovanadate and Cyanosilylation of Ketones

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Table S1. Crystallographic data for $(\text{Et}_4\text{N})_2[\text{VO}_2(\text{CN})_3]$

formula	$\text{C}_{57}\text{H}_{120}\text{N}_{15}\text{O}_6\text{V}_3$
fw	1264.49
crystal system	monoclinic
space group	$P2_1/c$ (#14)
a (Å)	8.2400(2)
b (Å)	34.3199(8)
c (Å)	12.7556(3)
β (deg)	93.1970(10)
V (Å ³)	3601.62(15)
Z	2
μ (mm ⁻¹)	3.616
R_1 ($I > 2\sigma(I)$)	0.0711
wR_2	0.1940

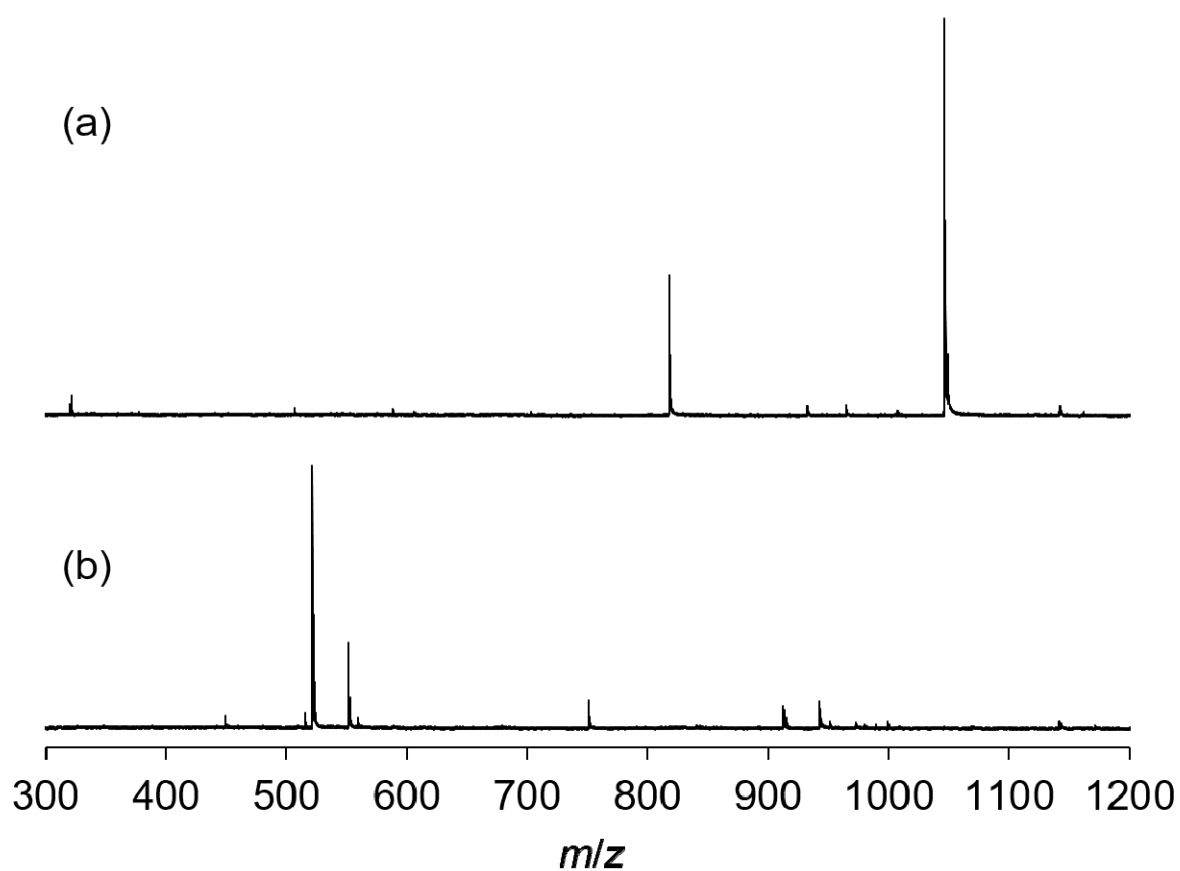


Figure S1. Positive mode of CSI-MS spectra of acetonitrile solution of tetraethylammonium salt of methavanadate (a) without addition of TMSCN and (b) in the presence of TMSCN. The main signal sets in (a) at m/z 817 and 1047 are assignable to $\{(\text{Et}_4\text{N})_4[\text{V}_3\text{O}_9]\}^+$ and $\{(\text{Et}_4\text{N})_5[\text{V}_4\text{O}_{12}]\}^+$, respectively. The main signal sets in (b) at m/z 521 and 551 are assignable to $\{(\text{Et}_4\text{N})_2\text{TMS}_2\text{VO}_4\}^+$ and $\{(\text{Et}_4\text{N})_3\text{VO}_2(\text{CN})_3\}^+$, respectively.

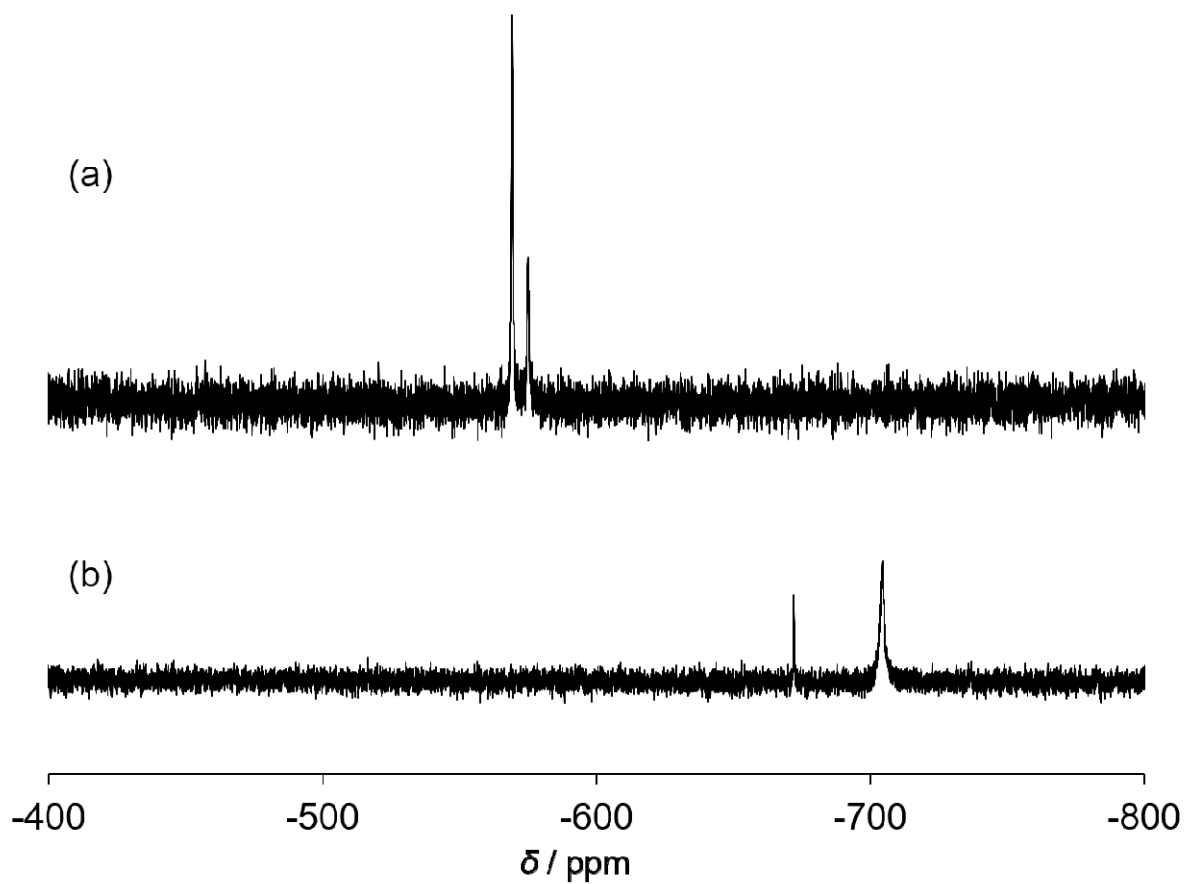


Figure S2. ^{51}V NMR spectrum of tetraethylammonium salt of methavanadate (a) without addition of TMSCN and (b) in the presence of TMSCN. Vanadium concentration is 2 mM.

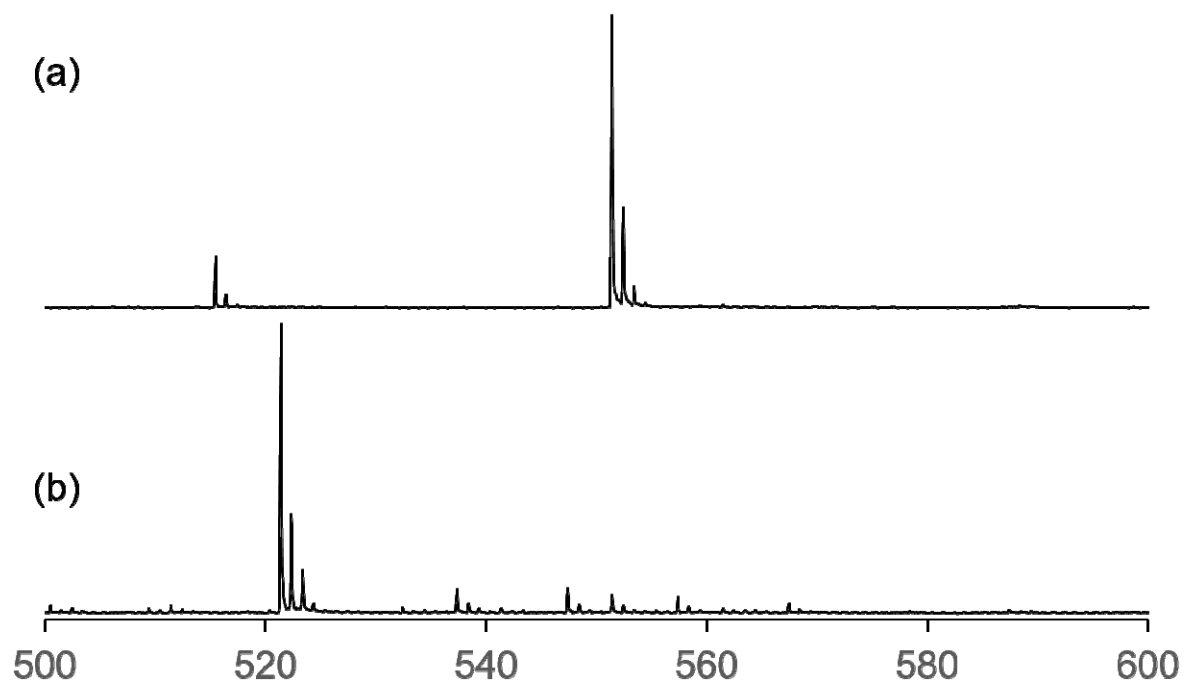


Figure S3. Positive mode of CSI-MS spectra of the reaction solution of cyanosilylation of 2-adamantanone with $(\text{Et}_4\text{N})_2[\text{VO}_2(\text{CN})_3]$ (a) before and (b) during the reaction. The main signal sets at m/z 551 in (a) and m/z 521 in (b) are assignable to $\{(\text{Et}_4\text{N})_3\text{VO}_2(\text{CN})_3\}^+$ and $\{(\text{Et}_4\text{N})_2\text{TMS}_2\text{VO}_4\}^+$, respectively.

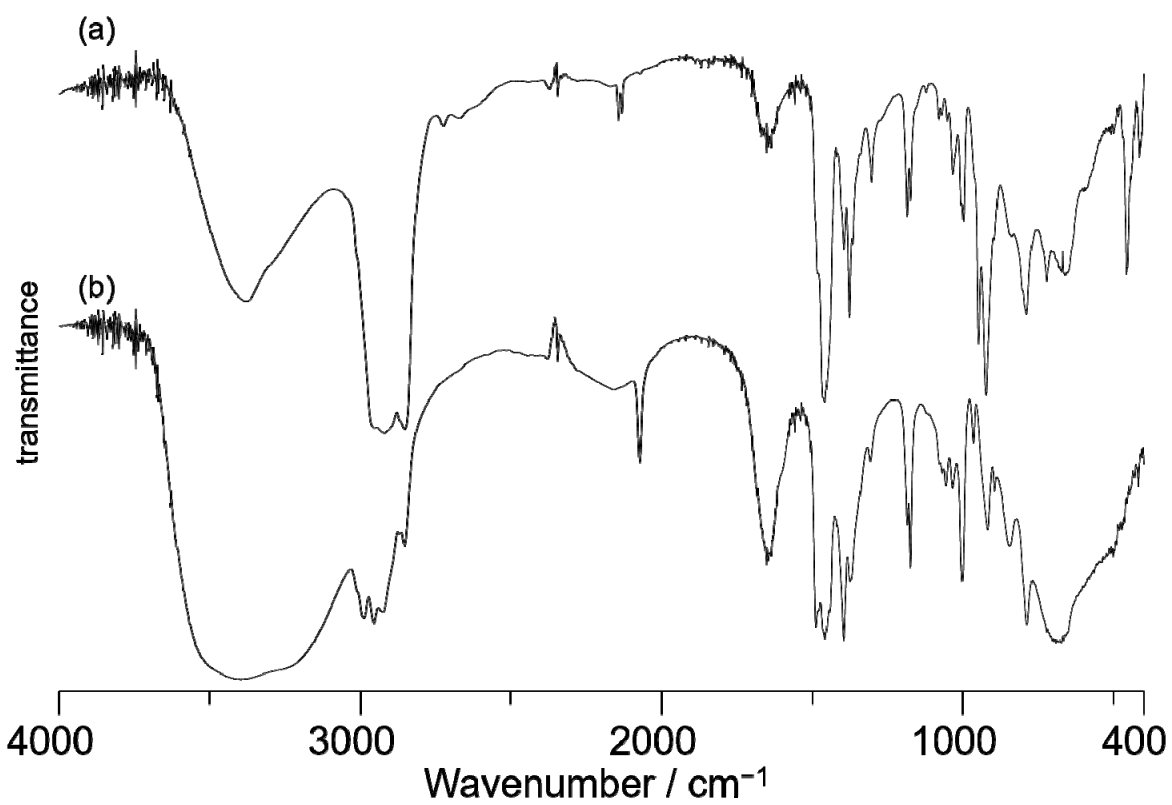


Figure S4. IR spectra of (a) $(\text{Et}_4\text{N})_2[\text{VO}_2(\text{CN})_3]$ and (b) Et_4NCN . Data are recorded with a nujol method.

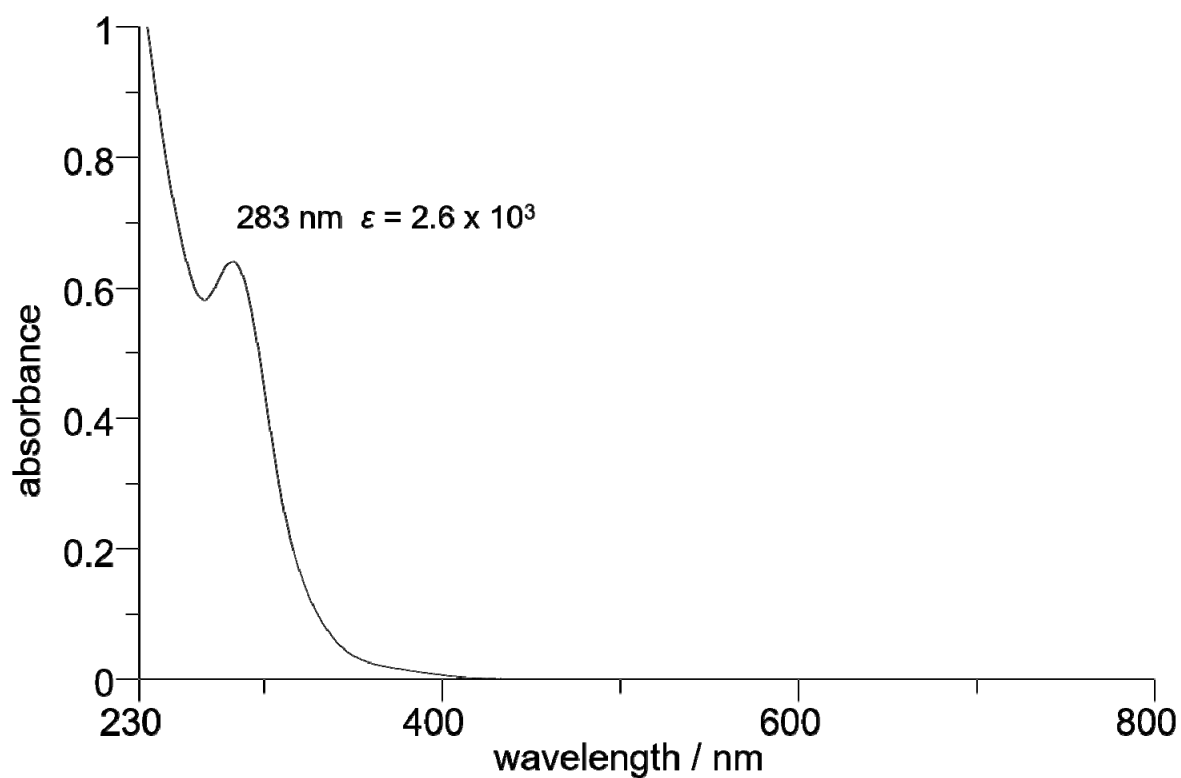


Figure S5. UV spectrum of $(\text{Et}_4\text{N})_2[\text{VO}_2(\text{CN})_3]$ in acetonitrile. The concentration of $(\text{Et}_4\text{N})_2[\text{VO}_2(\text{CN})_3]$ was 0.25 M.