

*Eur. J. Inorg. Chem.* ISSN 1099–0682

<https://doi.org/10.1002/ejic.201800168>

**SUPPORTING INFORMATION**

**Title:** Nickel–Alkyl Complexes with a Reactive PNC-Pincer Ligand

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Multinuclear NMR spectra ( $^1\text{H}$ ,  $^{13}\text{C}$ ,  $^{31}\text{P}$ ,  $^{19}\text{F}$ ) for complexes **2CH<sub>3</sub>** and **2CF<sub>3</sub>**

Cyclic voltammogram of **2CF<sub>3</sub>** (anodic scan)

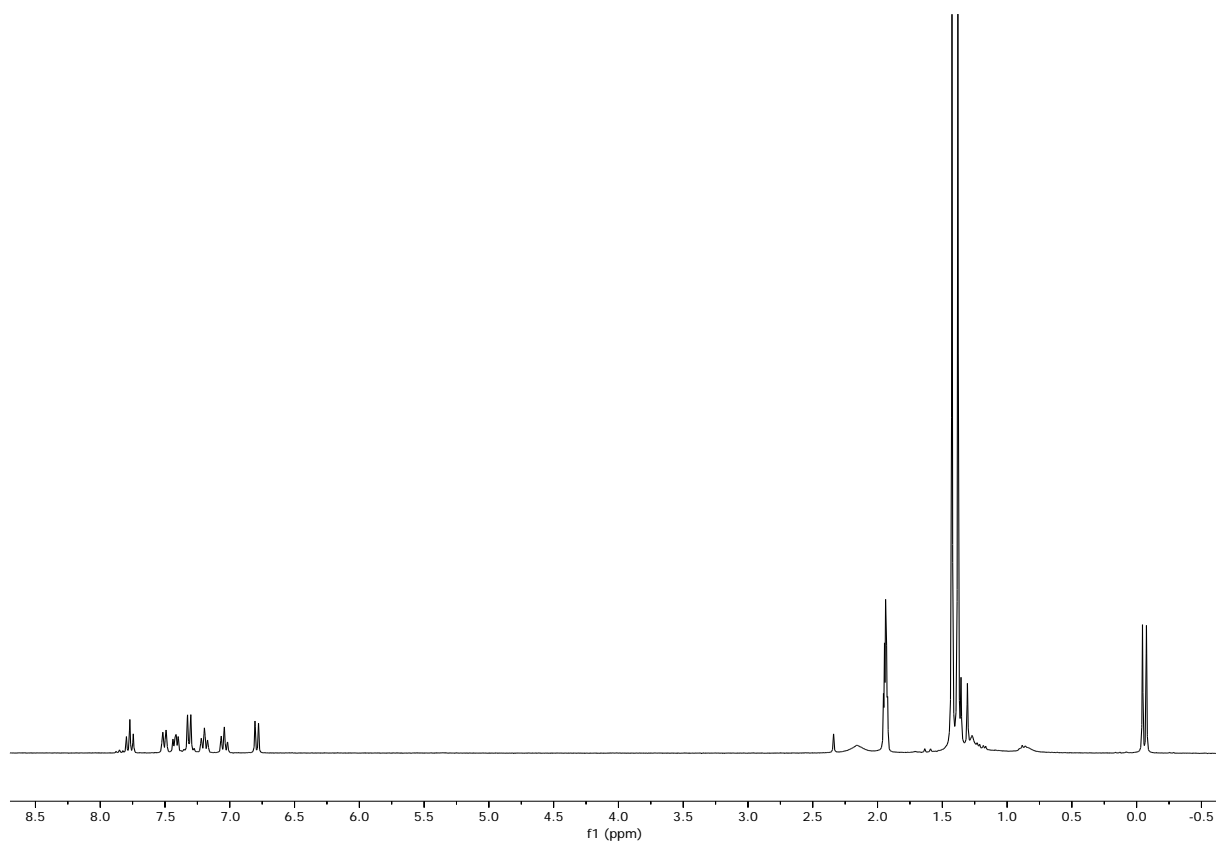
UV/VIS Spectroelectrochemistry of **2CF<sub>3</sub>** (anodic scan)

NMR spectra ( $^{31}\text{P}$  and  $^{19}\text{F}$ ) from reaction of **2CF<sub>3</sub>** with (thianthrenium)BF<sub>4</sub> and AgBF<sub>4</sub>

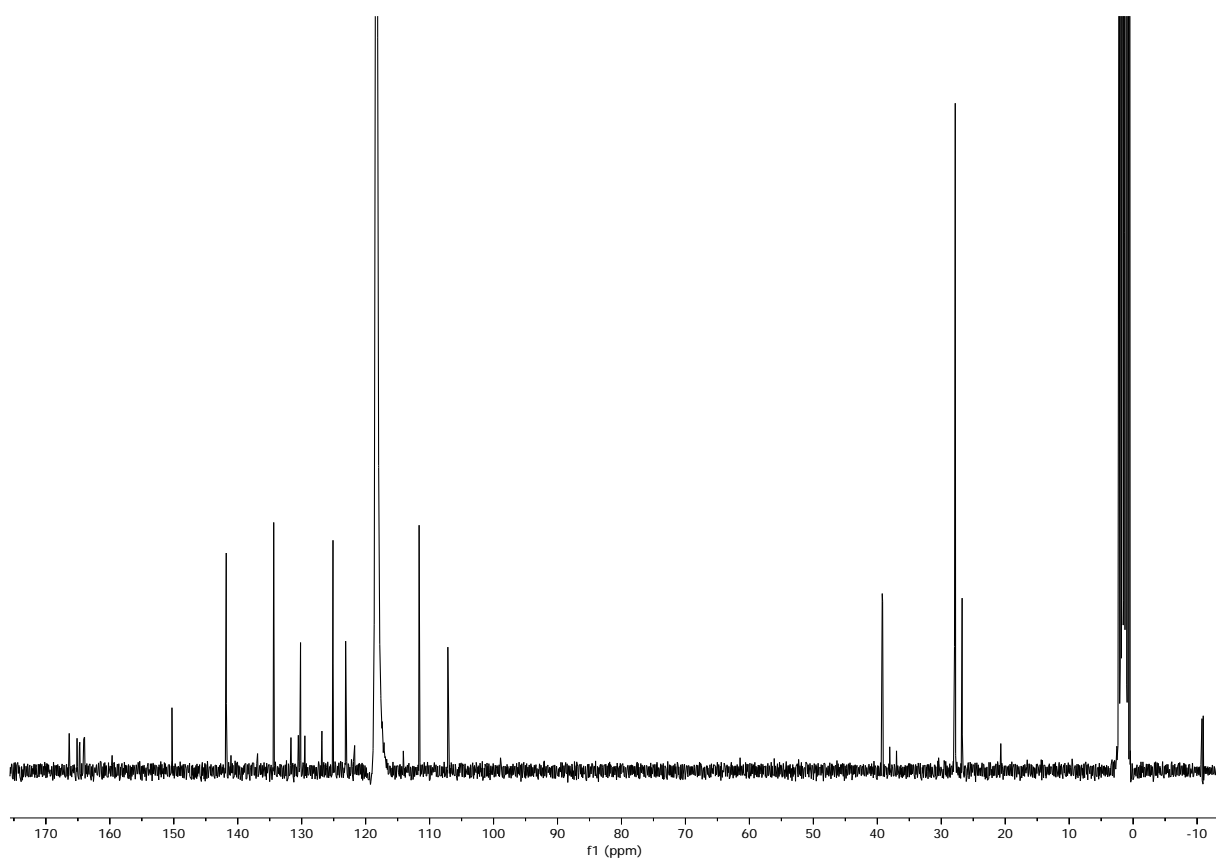
EPR spectrum for reaction of **2CF<sub>3</sub>** with KC<sub>8</sub>

EPR spectrum (r.t.) obtained from cathodic reduction of **2CF<sub>3</sub>**

Cathodic EPR spectroelectrochemistry of **2CH<sub>3</sub>**



**Figure S1**  $^1\text{H}$  NMR (300 MHz) spectrum of  $2\text{CH}_3$  in  $\text{MeCN-}d_3$



**Figure S2**  $^{13}\text{C}$  NMR (75 MHz) spectrum of  $2\text{CH}_3$  in  $\text{MeCN-}d_3$

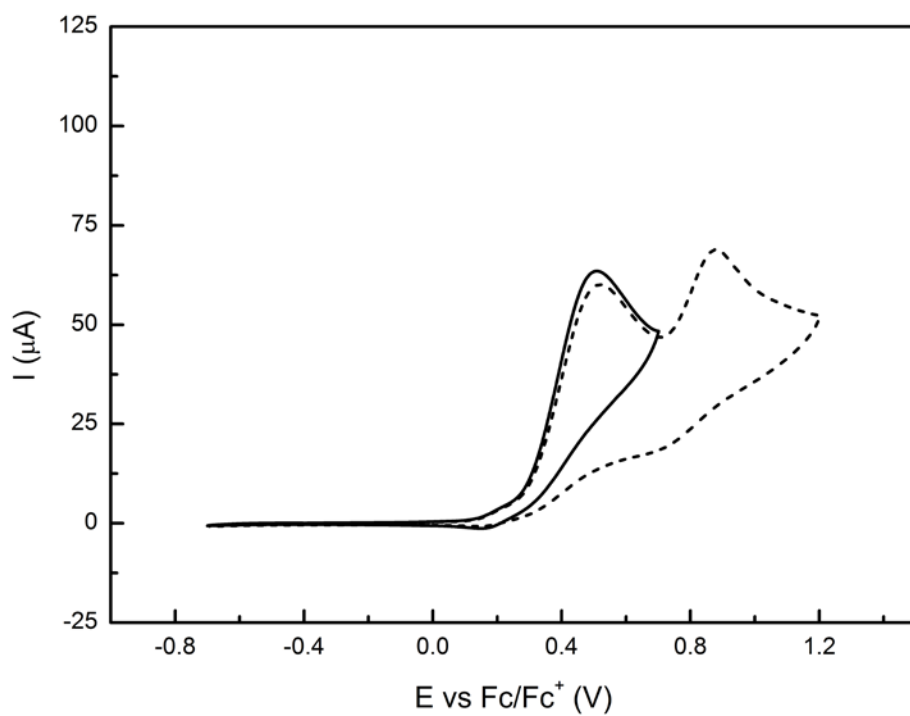
**Figure S3**  $^{31}\text{P}$  NMR (121 MHz) spectrum of **2CH<sub>3</sub>** in MeCN-*d*<sub>3</sub>

**Figure S4**  $^1\text{H}$  NMR (300 MHz) spectrum of **2CF<sub>3</sub>** in MeCN-*d*<sub>3</sub>

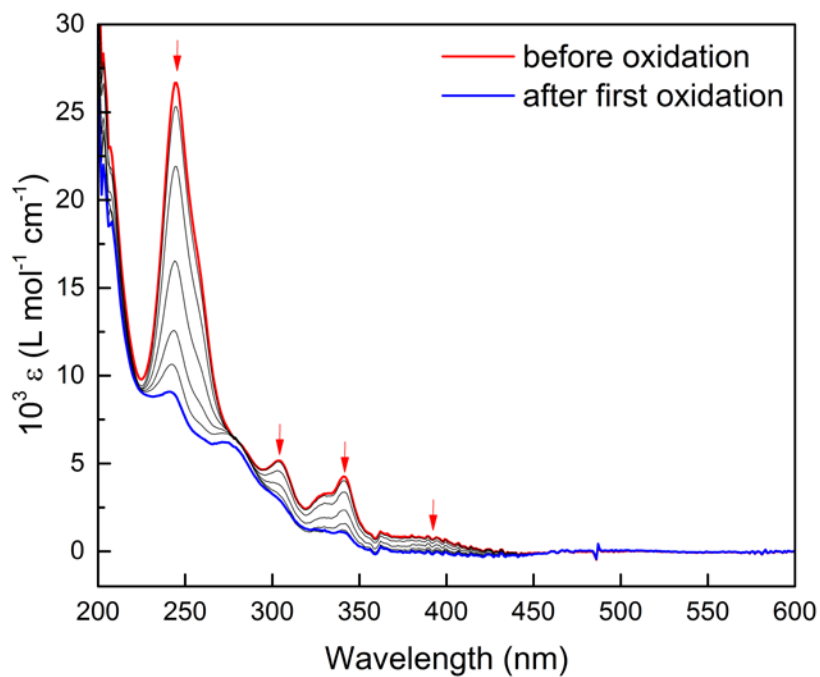
**Figure S5**  $^{13}\text{C}$  NMR (75 MHz) spectrum of **2CF<sub>3</sub>** in MeCN-*d*<sub>3</sub>

**Figure S6**  $^{31}\text{P}$  NMR (121 MHz) spectrum of **2CF<sub>3</sub>** in MeCN-*d*<sub>3</sub>

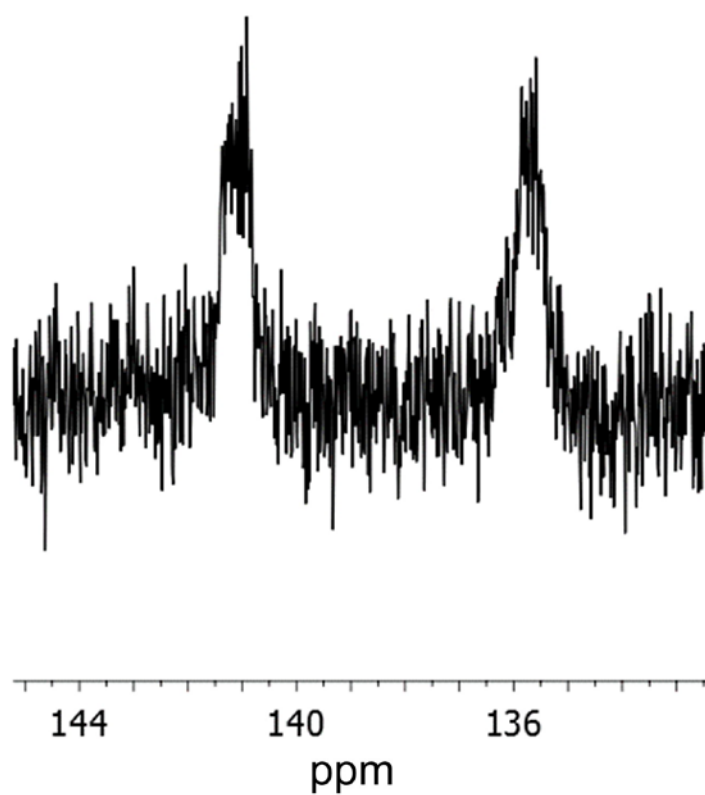
**Figure S7**  $^{19}\text{F}$  NMR (282 MHz) spectrum of  $2\text{CF}_3$  in  $\text{MeCN-}d_3$



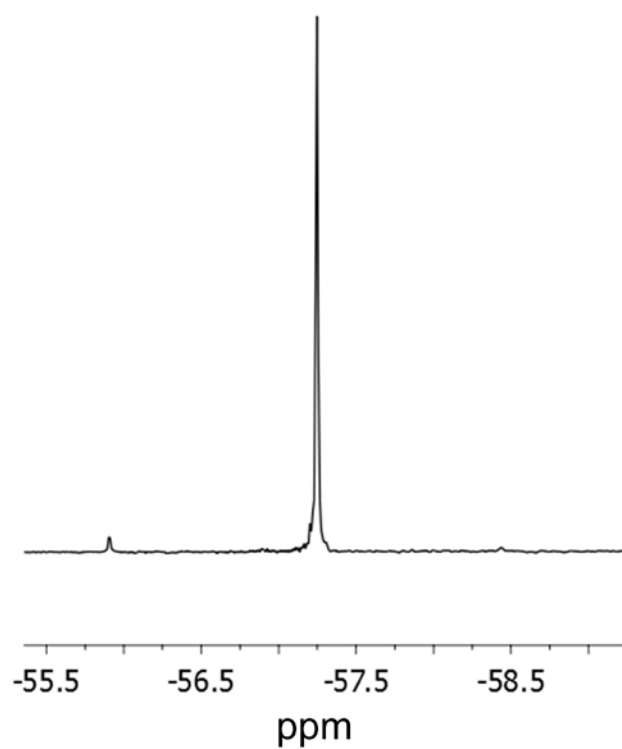
**Figure S8** Cyclic voltammogram of  $2\text{CF}_3$  (anodic scan wave only) in  $\text{PrCN}/\text{NnBu}_4\text{PF}_6$  at 298 K, with a scan rate of 100 mV/s



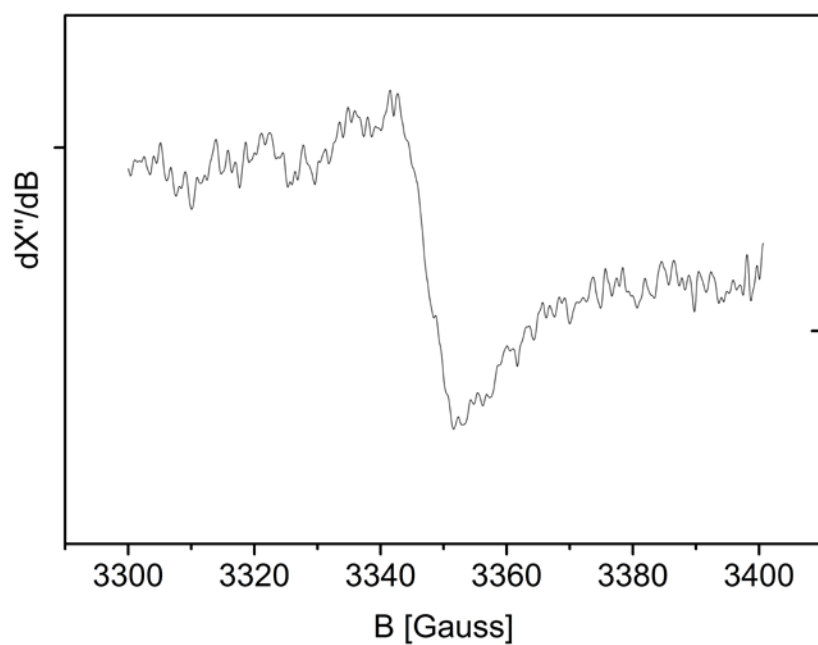
**Figure S9.** UV/Vis spectroelectrochemical oxidation of a solution of complex **2CF<sub>3</sub>** in THF/*n*Bu<sub>4</sub>NPF<sub>6</sub>; anodic scan from 0 V to +1.8 V (referenced to Ag/Ag<sup>+</sup>).



**Figure S10** <sup>31</sup>P NMR spectrum of product from reaction of **2CF<sub>3</sub>** with (thianthrenium)BF<sub>4</sub> and AgBF<sub>4</sub>

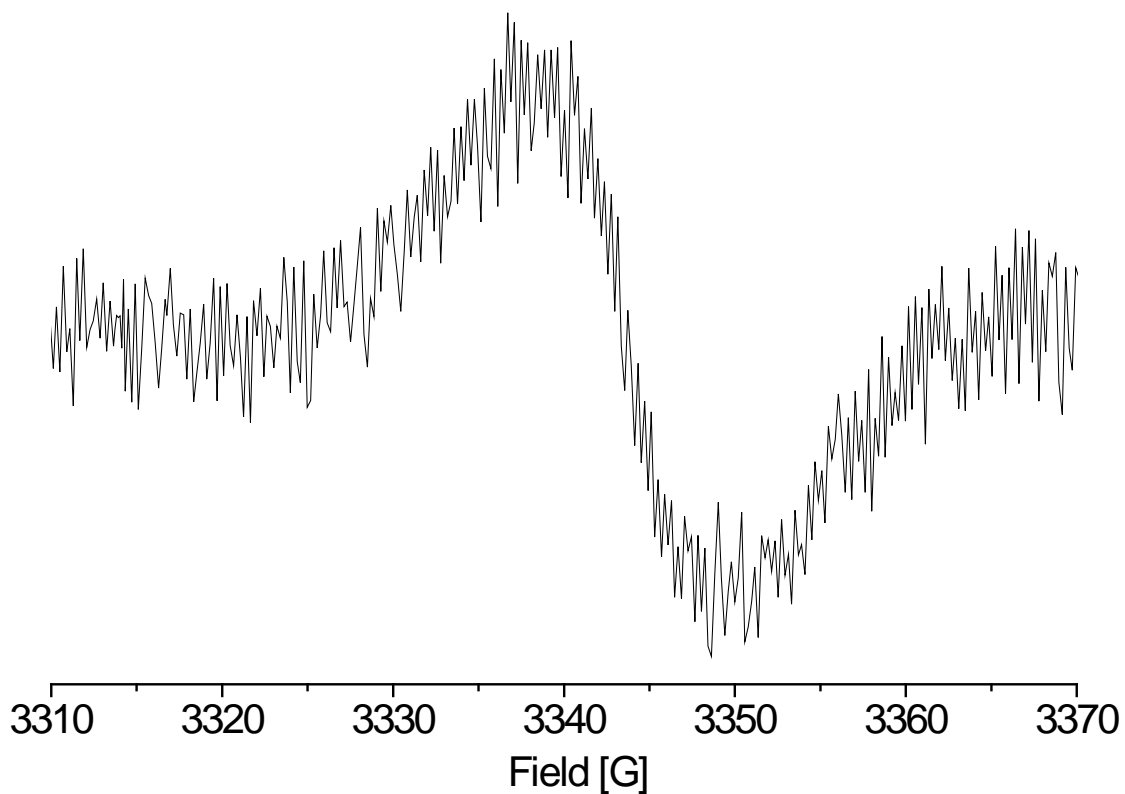


**Figure S11**  $^{19}\text{F}$  NMR spectrum of product from reaction of  $2\text{CF}_3$  with (thianthrenium) $\text{BF}_4$  and  $\text{AgBF}_4$

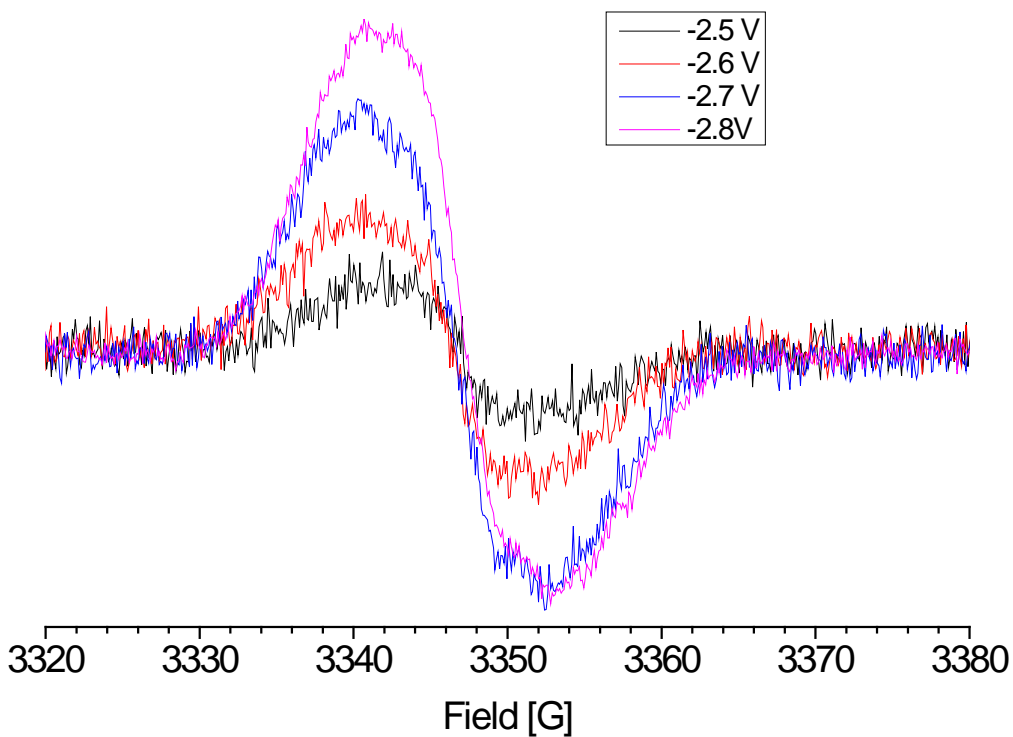


**Figure S12** EPR spectrum (r.t.) after reduction of  $2\text{CF}_3$  with  $\text{KC}_8$





**Figure S13** EPR spectrum obtained from cathodic reduction of  $2\text{CF}_3$  at  $-2.4\text{ V}$  in  $\text{THF}/n\text{Bu}_4\text{PF}_6$  at  $298\text{ K}$ .



**Figure S14** EPR spectra recorded during cathodic reduction of  $2\text{CH}_3$  in  $\text{THF}/n\text{Bu}_4\text{NPF}_6$  at  $298\text{ K}$ .