Supporting Information

Cyclic gas-phase heterogeneous process in a metal-organic framework involving a nickel nitrosyl complex

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FT-IR spectra

Fig. S1  FT-IR spectrum of Ni-MFU-4l-NO₂.

Fig. S2  FT-IR spectrum of Ni-MFU-4l-NO.
Fig. S3  FT-IR spectra of Co-MFU-4l-NCO (green) and Co-MFU-4l-NO₂ after treatment with CO at 350 °C (red).

**DRIFT spectra**

Fig. S4  DRIFT spectra of Ni-MFU-4l-NO₂ after treatment with CO at 350 °C and subsequent treatment with NO at 40 °C (marks over the spectrum indicate a gas and cycle number); • - bands, accumulating due to NO₂ / N₂O₄ impurities.
**UV-vis-NIR spectra**

**Fig. S5**  UV-vis-NIR spectra of Ni-MFU-4I-NO$_2$ (green) and Ni-MFU-4I-NO (blue).

**Fig. S6**  UV-vis-NIR spectrum of Ni-MFU-4I-NO$_2$. 
Fig. S7  In situ UV-vis-NIR spectra of Ni-MFU-4I-NO before (blue) and after (red) reaction with 5% NO in Ar; green curve shows a reference spectrum of Ni-MFU-4I-NO$_2$.

Fig. S8  Zoom of the in situ UV-vis-NIR spectra shown in Fig. S7 (the spectrum of Ni-MFU-4I-NO is not shown).
Powder X-ray diffraction measurements

Fig. S9  XRPD patterns of MFU-4I (red), Ni-MFU-4I-NO₂ (green) and Ni-MFU-4I-NO (blue).
Gas sorption measurements

![Graph showing nitrogen adsorption isotherms at 77.3 K for Ni-MFU-4/NO\(_2\) (green) and Ni-MFU-4/NO (blue).](image)

**Fig. S10** Nitrogen adsorption isotherms at 77.3 K for Ni-MFU-4/NO\(_2\) (green) and Ni-MFU-4/NO (blue).