

Supporting information

Probing dynamic covalent chemistry in a 2D boroxine framework by *in-situ* near-ambient pressure X-ray photoelectron spectroscopy

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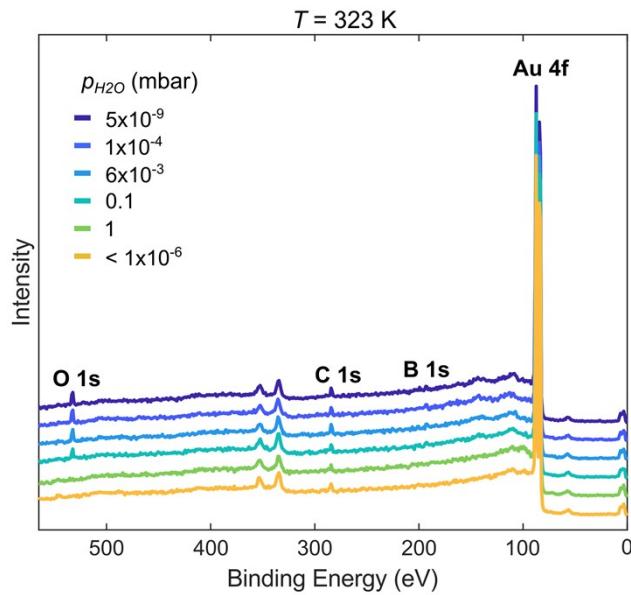


Fig. SI1. Survey spectra acquired at $T = 323\text{ K}$ upon sequential increase of $p_{\text{H}_2\text{O}}$ and after cell evacuation. Photon energy: 650 eV.

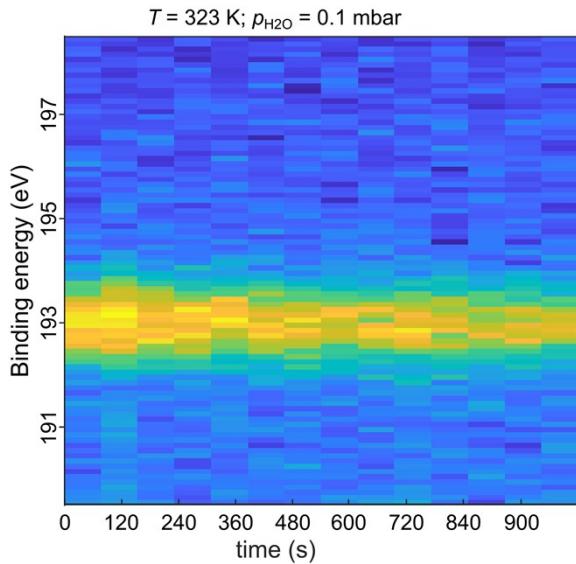


Fig. SI2. Time-resolved B 1s spectra acquired at $T = 323\text{ K}$ and $p_{\text{H}_2\text{O}} = 0.1\text{ mbar}$. Photon energy: 650 eV.

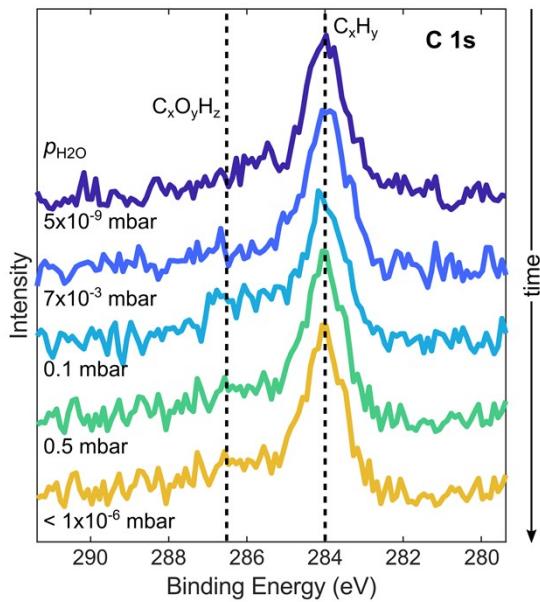


Fig. S13. Carbon-containing contaminations at NAP. C 1s spectra acquired at $T = 300$ K upon sequential increase of $p_{\text{H}_2\text{O}}$ and after cell evacuation. Photon energy: 650 eV. The intensity of hydrocarbon species (C_xH_y , 284.0 eV) is rather constant. Oxygen-containing carbon species ($\text{C}_x\text{O}_y\text{H}_z$) are detected at 0.1 mbar at 286.5 eV, while their intensity diminish at 0.5 mbar.

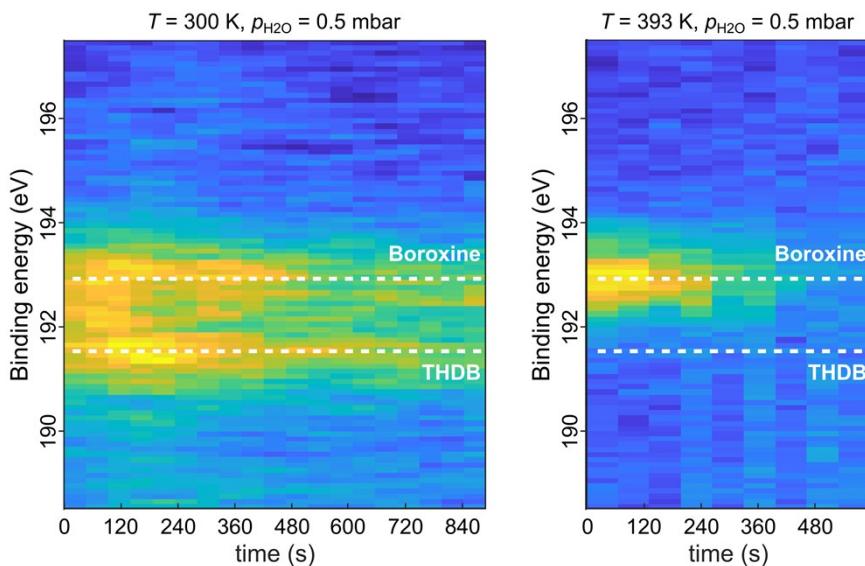


Fig. S14. *In-situ* NAP-XPS characterization at $p_{\text{H}_2\text{O}} = 0.5$ mbar. Time-resolved B 1s spectra acquired at $T = 300$ K (left) and $T = 393$ K (right). Photon energy: 650 eV. The binding energies corresponding to boroxine and THDB are indicated.

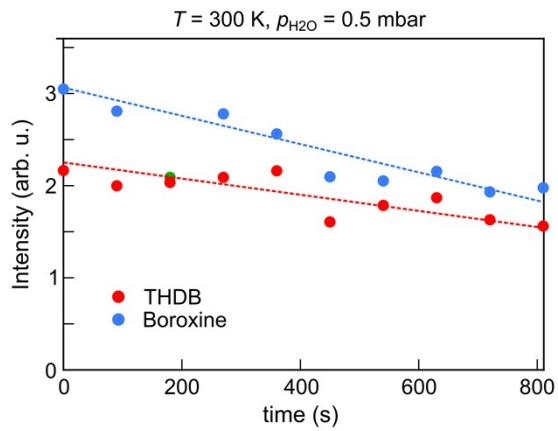


Fig. S15. *In-situ* NAP-XPS characterization at $p_{\text{H}_2\text{O}} = 0.5 \text{ mbar}$. Plot of the time-resolved B 1s spectra components: THDB (red) and boroxine (blue), with dashed lines as a guide to the eye.