

## Electronic Supplementary Information

### Synthesis of Photo-responsive Single-walled Nanoscroll and Photo-reactivity in Nano-layered Microenvironment

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#### ESI-1: Characterization of the single-walled niobate nanosheet

The XRD pattern change by the intercalation of  $\text{PrNH}_2$  into the interlayer spaces of niobate after the acid treatment is shown in Fig. SI-1. After the ion exchange by an acidic aqueous solution (1.6 M  $\text{HNO}_3$ ), the (040) peak shifted to the higher angle ( $d_{040} = 0.80$  nm), but another peak ( $d = 1.08$  nm) was also observed to indicate that only the interlayer I suffered the ion exchange with protons as shown in Fig. SI-1(a). On the other hand, the acid treatment with the more concentrated  $\text{HNO}_3$  (6.5 M), the peak assigned to the insufficient protonated niobate completely disappeared and only the (040) peak at the higher angle ( $d_{040} = 0.80$  nm) was observed as shown in Fig. SI-1(b), clearly indicating the complete substitution of  $\text{K}^+$  into  $\text{H}^+$  in both interlayer I and II of  $\text{K}_4\text{Nb}_6\text{O}_{17}$  under the condition. In the AFM observations, the thickness of niobate nanosheets obtained by the intercalation of  $\text{PrNH}_2$  into the protonated niobate treated by 1.6 M  $\text{HNO}_3$  is measured at ca. 2.5 nm (Fig SI-1(c) and (e)), which is assigned to the double-walled nanosheet, while that of niobate nanosheet by 6.5 M  $\text{HNO}_3$  is done at ca. 1.7 nm (Fig SI-1(d) and (f)), which is assigned to the single-walled nanosheet.

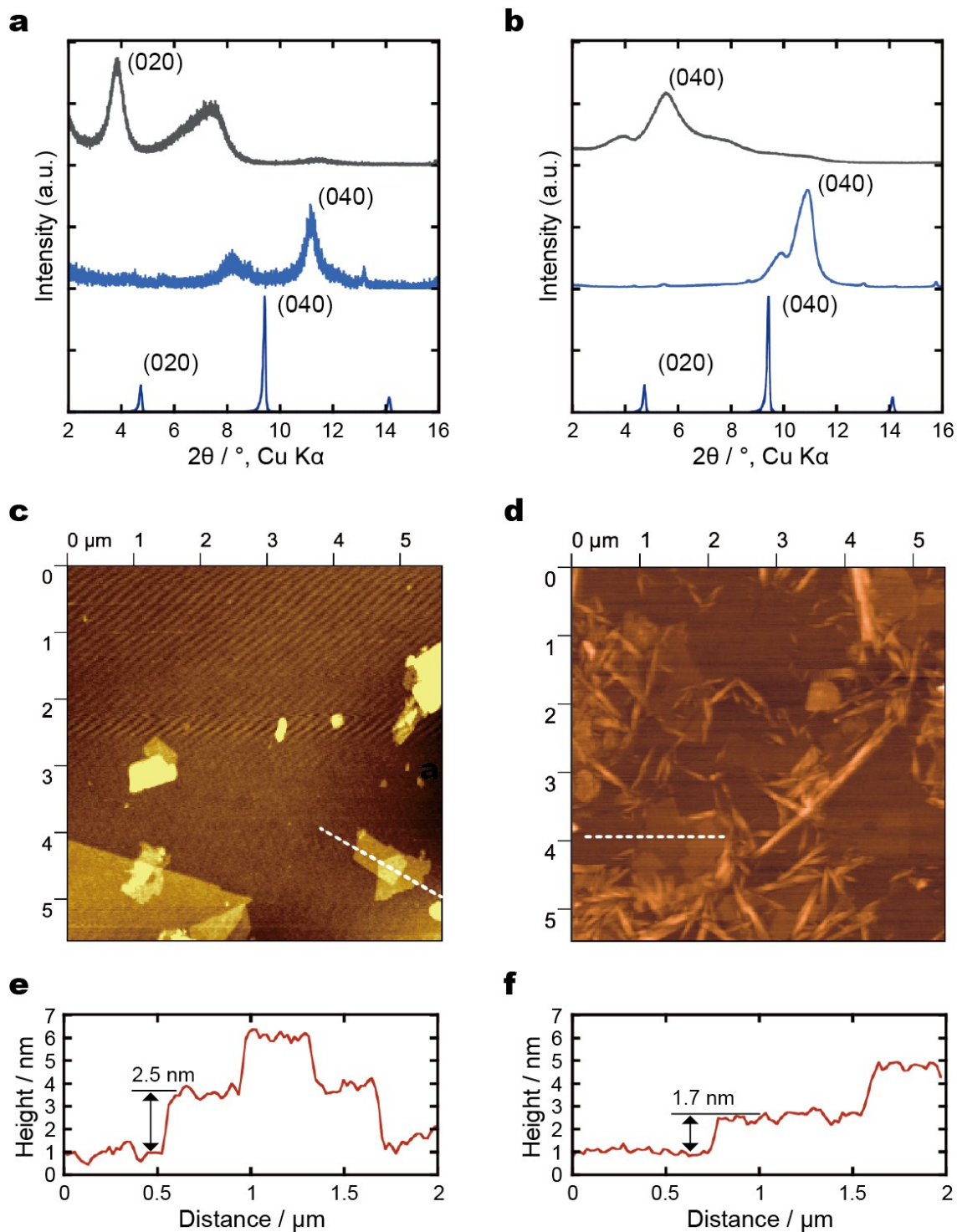


Fig. SI-1. XRD pattern changes of the intercalation of PrNH<sub>2</sub> into the niobates after the (a) 1.6M and (b) 6.5 M acid treatment and AFM morphologies of the PrNH<sub>2</sub>-intercalated niobate obtained from the protonated niobate treated by (c, e) 1.6 M and (d, f) 6.5 M HNO<sub>3</sub>.

**ESI-2:** Nano-layered structure of PrNH<sub>2</sub> intercalated niobate characterized by TG/DTA

The PrNH<sub>2</sub>-intercalated niobate hybrid was analyzed by TG/DTA. The TG/DTA curve for the PrNH<sub>2</sub>-intercalated niobate is shown in Fig. SI-2. The amount of the intercalated molecules in the niobate hybrid was calculated from the weight loss from 100 °C to 750 °C. The adsorption occupied area by one PrNH<sub>3</sub><sup>+</sup> molecule on the SW nanosheet surface was estimated at 31 Å<sup>2</sup>, indicating the molecules form a bilayer in the interlayer space of niobate hybrid.

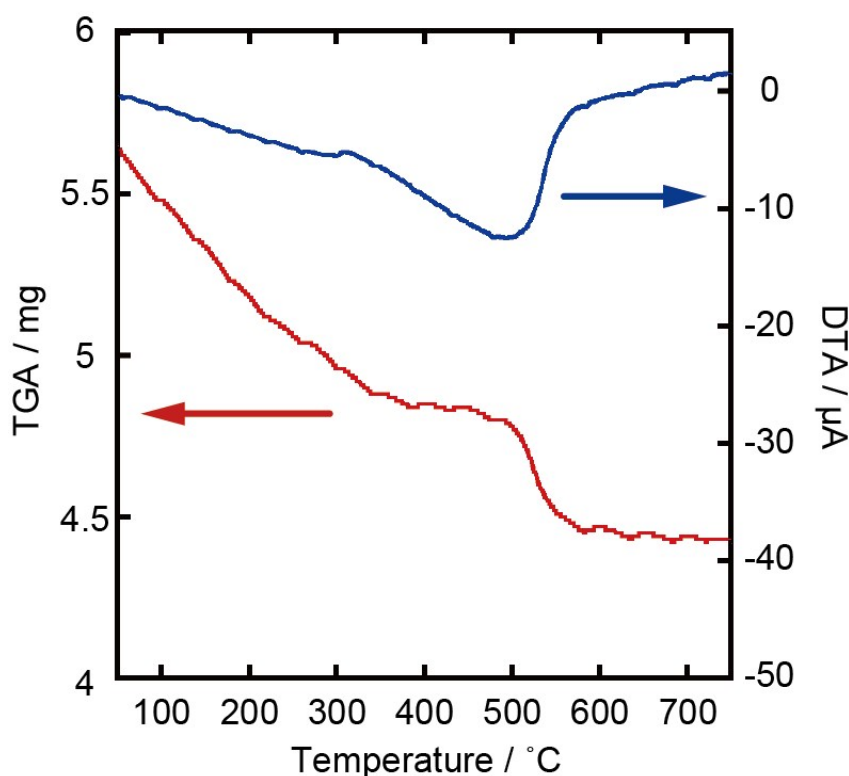


Fig. SI-2. TG/DTA curve for SWNS after the intercalation of PrNH<sub>2</sub>.

**ESI-3:** Estimation of extinction coefficients of C3F-Azo-C6H in the niobate hybrid

The extinction coefficients of C3F-Azo-C6H in the niobate hybrids were estimated by the procedure illustrated in Fig. SI-3(a, (1)-(4)). The absorption spectra of the starting all-*trans* C3F-Azo-C6H in the niobate hybrids before the UV light irradiation and *cis*-rich (photostationary state) upon the UV light irradiation were measured (Fig. SI-3(a, (1))). The hybrid sample was then all removed from the glass plate and was dispersed in CD<sub>2</sub>Cl<sub>2</sub> for <sup>1</sup>H-NMR measurement to determine the *trans*:*cis* ratio in the hybrids (Fig. SI-3(a, (2))). The *trans*- and *cis*-C3F-Azo-C6H mixture was completely desorbed from the niobate hybrids by treating with acid in ethanol (Fig. SI-3(a, (3))) and the filtrate solution containing *trans*- and *cis*-C3F-Azo-C6H mixture was heated at 60°C for 4 h in the dark to revert to all *trans*- form of C3F-Azo-C6H to allow the measurement

of absorption spectrum. The amount of C3F-Azo-C6H was thus determined by the absorbance and the known extinction coefficient in ethanol. The extinction coefficient of C3F-Azo-C6H in niobate hybrids was determined on the assumption that the same amount in the final ethanol solution was intercalated within niobate nanosheets. The concentration of *cis*- form was further determined by the *trans*:*cis* ratio by the  $^1\text{H-NMR}$  measurement and the total concentration of C3F-Azo-C6H. The  $^1\text{H-NMR}$  spectra of C3F-Azo-C6H in the SWNS, nanosheet-stacked film and DWNS are shown in Fig. SI-3(b), SI-3(c) and SI-3(d) before and after UV irradiations, respectively. The *trans*:*cis* ratio in the hybrid after UV irradiation was estimated from the integral of proton around the azobenzene moiety.

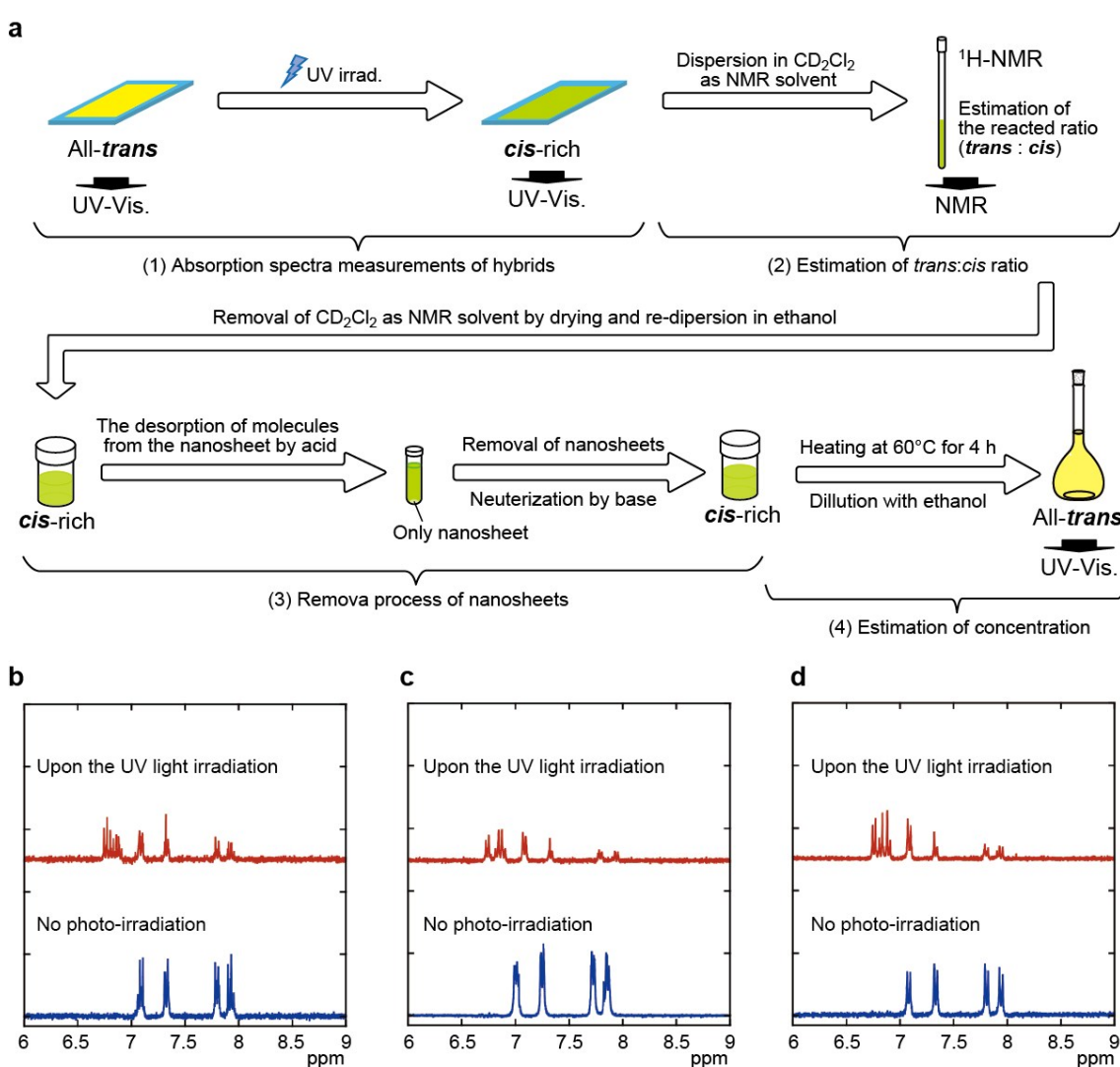


Fig. SI-3. (a) Schematic illustration of the absorption coefficient estimation of C3F-Azo-C6H in the niobate hybrids, and the  $^1\text{H-NMR}$  spectra of C3F-Azo-C6H in (b) SWNS, (c) nanosheet-stacked film and (d) DWNS.