

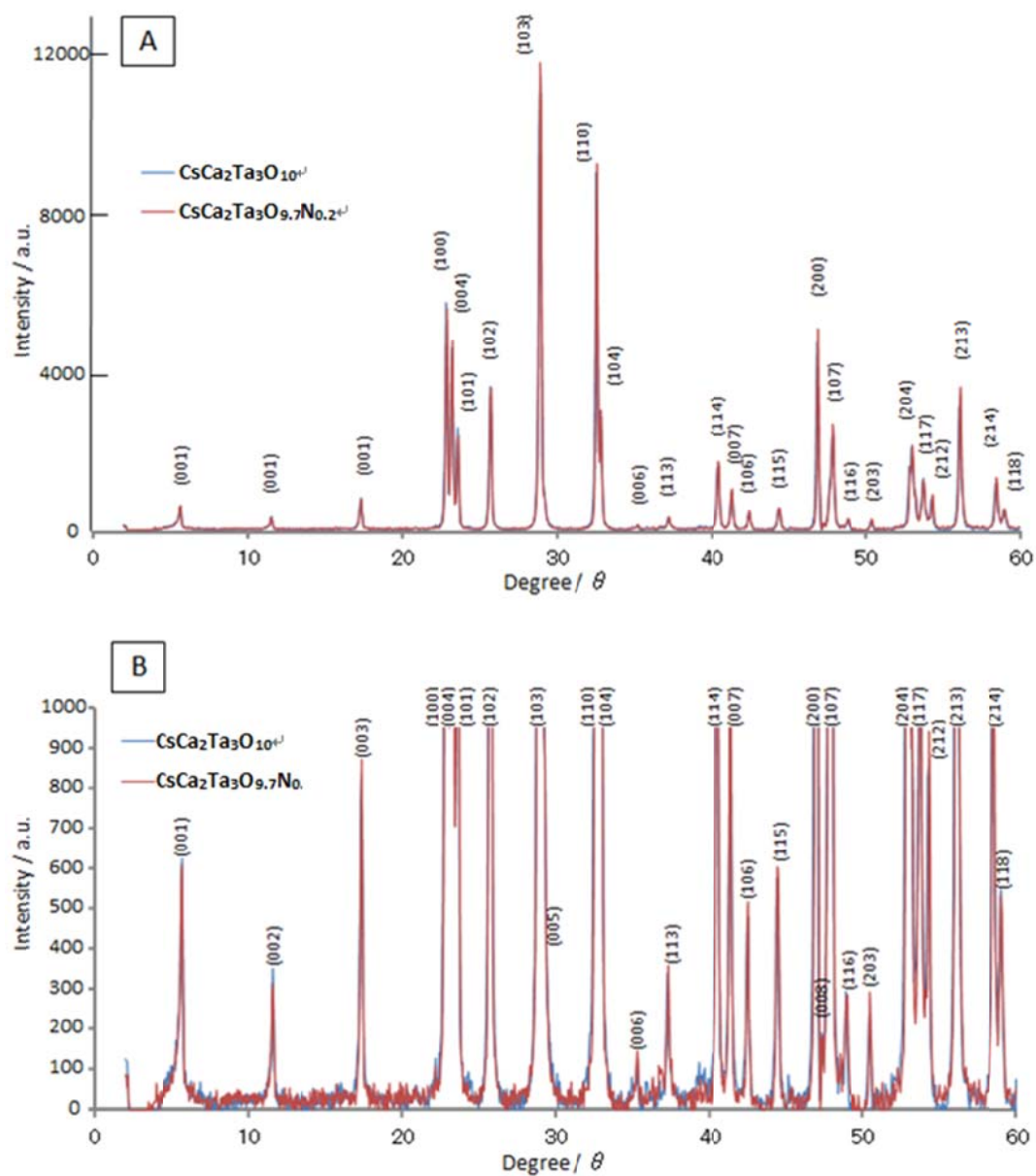
## Supporting Information

### Preparation of Tantalum-Based Oxynitride Nanosheets by Exfoliation of a Layered Oxynitride, $\text{CsCa}_2\text{Ta}_3\text{O}_{10-x}\text{N}_y$ , and Their Photocatalytic Activity

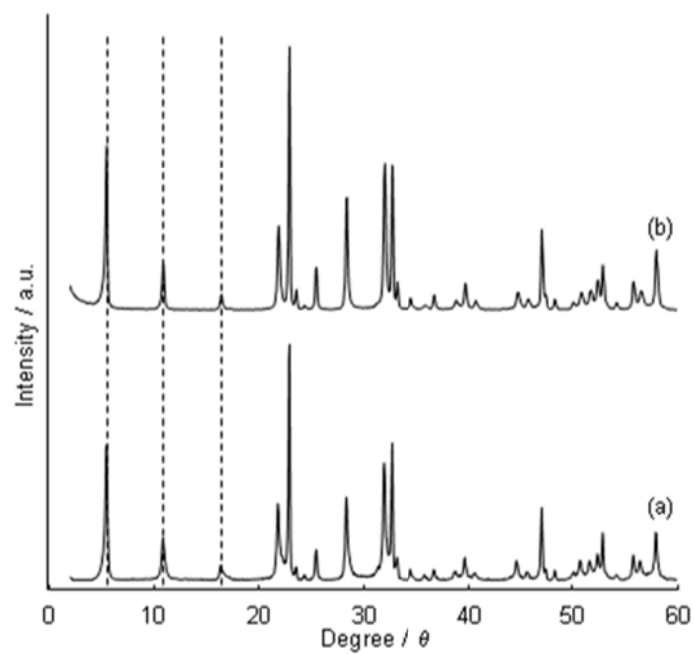
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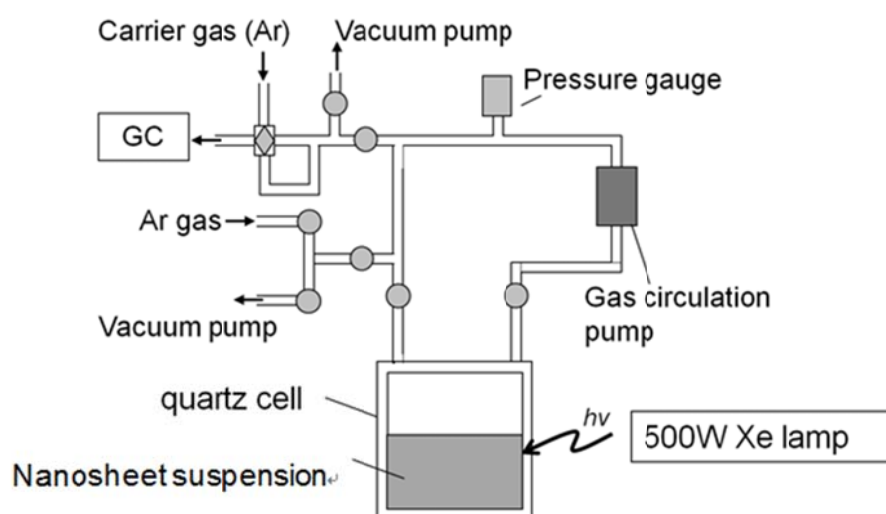


**Figure S1.** XRD patterns of  $\text{CsCa}_2\text{Ta}_3\text{O}_{10}$  and  $\text{CsCa}_2\text{Ta}_3\text{O}_{9.7}\text{N}_{0.2}$ : (A) overall spectra, (B) spectra in the region of weak intensity.

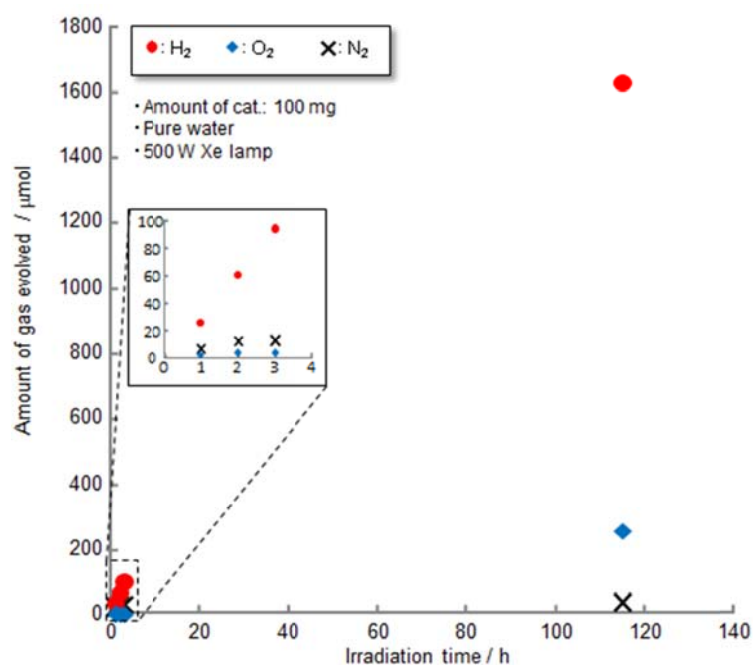


**Figure S2.** XRD patterns of  $\text{H-Ca}_2\text{Ta}_3\text{O}_{9.7}\text{N}_{0.2}$ ; (a) before and (b) after intercalation reaction in TBAOH aqueous solution.

S3



**Figure S3.** Apparatus for photocatalytic reaction. The reaction apparatus has a one-chamber system. The reaction was carried out in Ar atmosphere.



**Figure S4.** Time course of photocatalytic hydrogen and oxygen generation in pure water over Rh(0.15 wt.%) loaded  $\text{Ca}_2\text{Ta}_3\text{O}_{9.7}\text{N}_{0.2}$  nanosheets restacked with proton under UV-light irradiation (500 W Xe-lamp).