Figure S1. Examples of neutron diffraction diagrams obtained during the in situ H/D exchange reaction at 20°C. H content inside the Ti-material is indicated in front of each pattern in relation with Figures 2 and 3. Since neutrons show a huge incoherent scattering cross section for hydrogen of 80.26 barn for \(^{1}\)H, which falls off to 2.05 barn for deuterium \(^{2}\)H [12], the related contrast of both isotopes has been used to follow this D/H isotope exchange reaction in a specially adapted solid-gas reaction cell. Plotted diagrams with 0% and 100% of H content inside the Ti-material well illustrate the intensity increase due to the absorption of hydrogen atoms instead of the initial deuterium ones.

Figure S2. Normalized raw data diagrams measured at 20°C. (a) Neutron pattern obtained during the in situ H/D exchange reaction (powder Ti-material and sample environment), it shows 0% of H content inside the Ti-material (5 s collecting time). (b) Neutron pattern of the sample environment with empty quartz tube (120 s collecting time). (c) Subtraction between diagram (a) and diagram (b) illustrating the raw data collected from only the Ti-sample (representing a 5 s collecting time diagram). Diagram (c) well shows the poor-crystallized form of the studied TiO(OD)\(_2\) compound. It proves the necessity to use the intrinsic interaction properties of neutrons to investigate with accuracy kinetic exchange reaction involving hydrogen atoms close to room temperature.