Supplementary Information

**Fig S1.** Raman spectrum of the Ta₃N₅ synthesised under high pressure in autoclave showing the signature peaks of graphitic carbon at 1570 and 1325 cm⁻¹

**Fig S2.** TGA trace showing the decomposition of Ta₃N₅ to Ta₅N₆ under nitrogen - the product is Ta₅N₆ by PXD
Fig S3. SEM micrograph of the product of reaction of TaCl₅ with commercial LiNH₂ under ambient pressure before washing with THF. Li is not detectable in the EDX.
Fig S4. SEM micrograph of the product of reaction of TaCl$_5$ with commercial LiNH$_2$ under reflux after washing with THF (sample 1).
Fig S5. Infrared spectrum of commercial LiNH₂ highlighting the peak corresponding to OH stretching.

Fig S6. TGA profile for the decomposition of Ta₃N₅ produced by the reaction between TaCl₅ and pure LiNH₂ under high pressure (sample 3) in oxygen.
Fig S7. SEM micrograph of the product of the reaction of TaCl₅ with Mg₃N₂ under ambient pressure after washing (sample 4)
Fig S8. Electron Diffraction Pattern of the product of the reaction of TaCl₅ with Mg₃N₂ under ambient pressure after washing (sample 4)