Electronic Supplementary Material (ESI) for Dalton Transactions.

Supporting Information

Effect of isothermal holding time on hydrogen-induced structural transitions of WO_3

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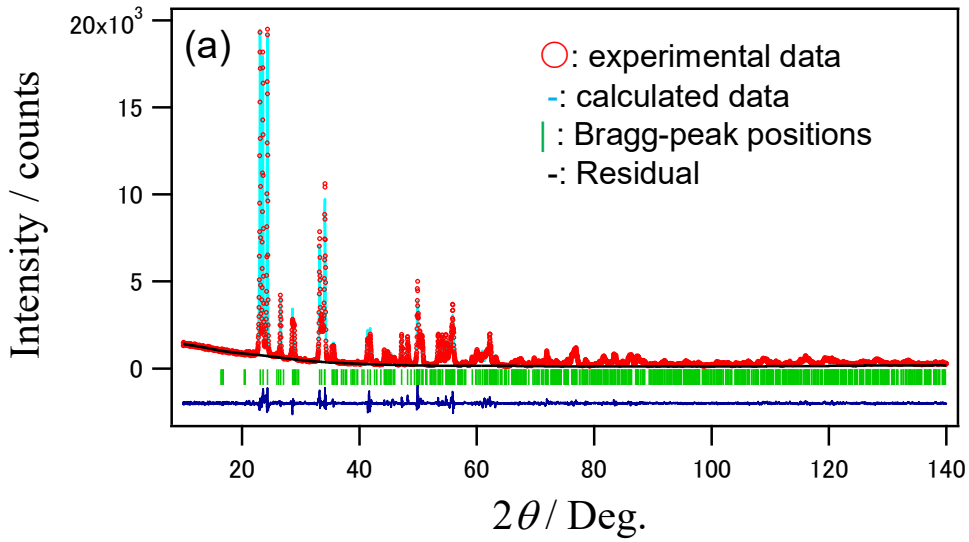
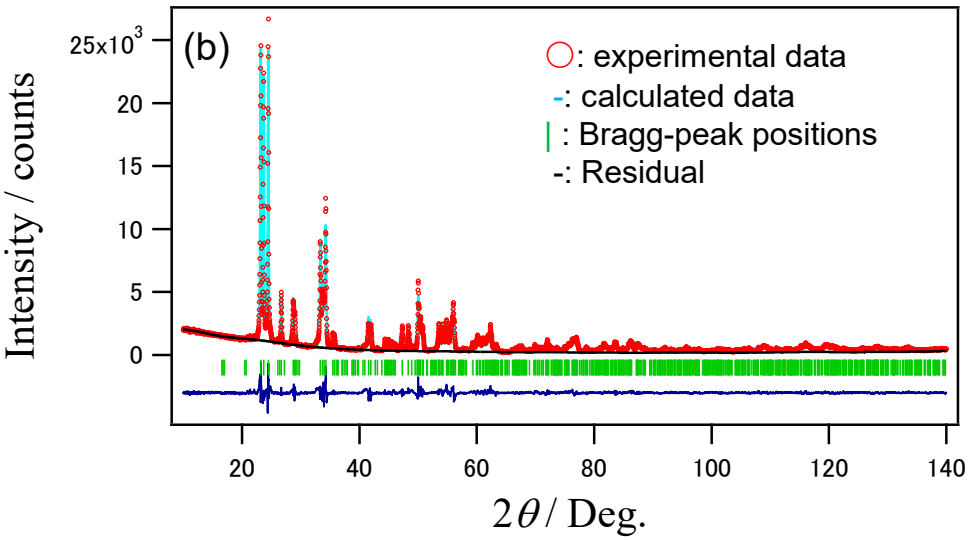
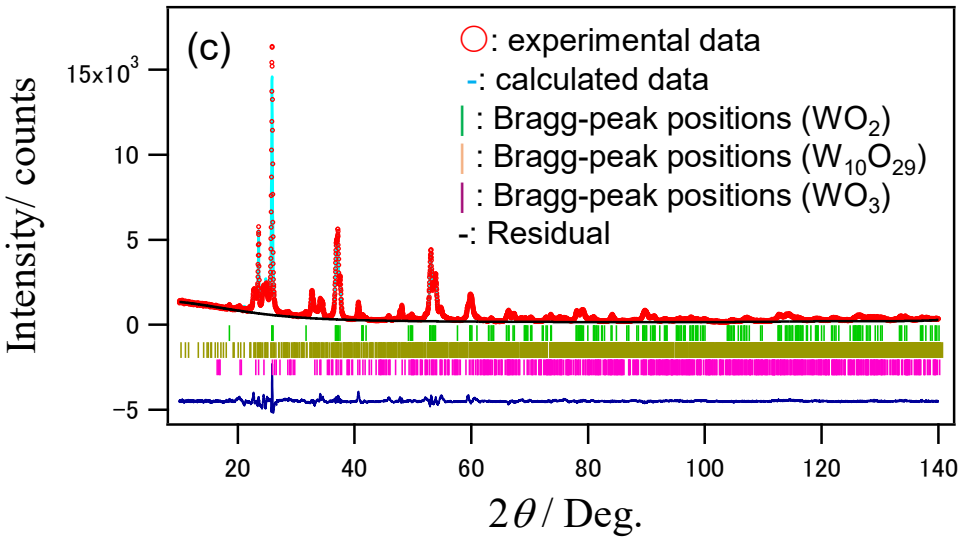

(a)
20x10^3
15
10
5
0
Intensity / counts
○: experimental data
-: calculated data
| : Bragg-peak positions
-: Residual
20
40
60
80
100
120
140
2θ / Deg.


(b)
25x10^3
20
15
10
5
0
Intensity / counts
○: experimental data
-: calculated data
| : Bragg-peak positions
-: Residual
20
40
60
80
100
120
140
2θ / Deg.


(c)
15x10^3
10
5
0
−5
Intensity/ counts
○: experimental data
-: calculated data
| : Bragg-peak positions (WO2)
| : Bragg-peak positions (W10O29)
| : Bragg-peak positions (WO3)
-: Residual
20
40
60
80
100
120
140
2θ / Deg.

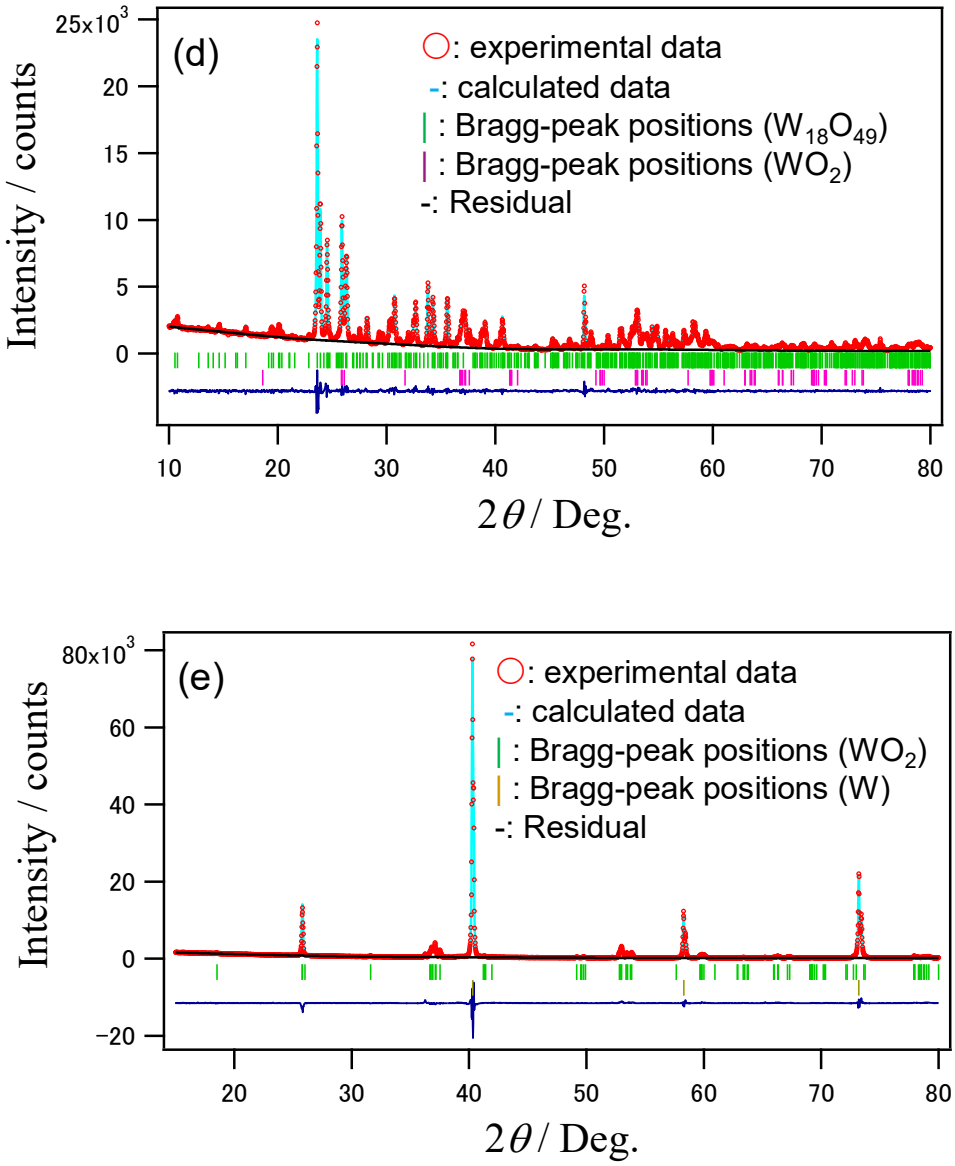


Figure S1. Rietveld refinement results of XRPD measurements of WO_3 samples before and after hydrogen treatments: (a) WO_3, (b) bule WO_3 (500 °C for 30 min), (c) mazarine WO_3 (500 °C for 22 h), (d) mazarine WO_3 (800 °C for 30 min), and (e) gray WO_3 (800 °C for 22 h).

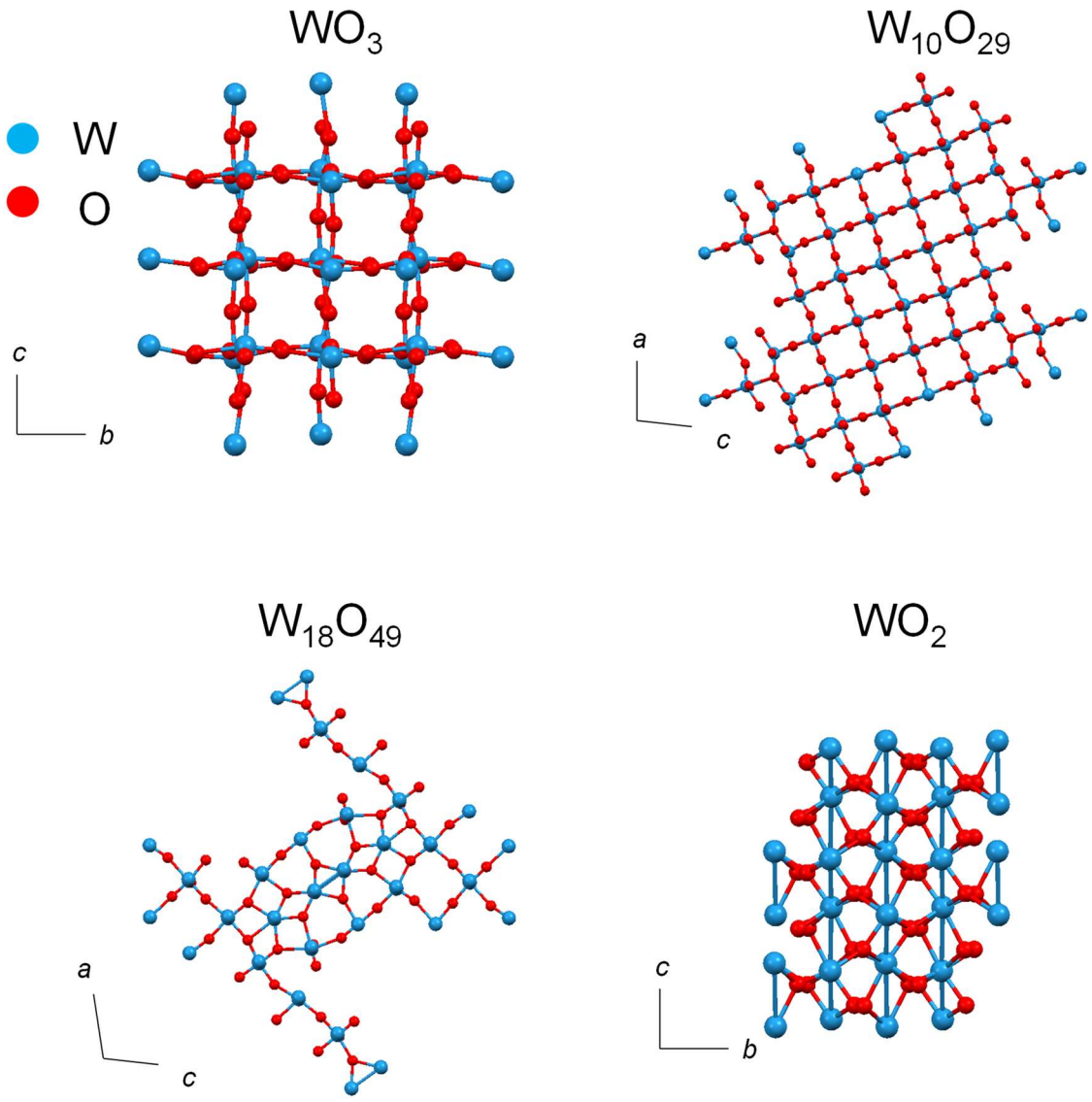


Figure S2. Crystal structures of WO_3, $W_{10}O_{29}$, $W_{18}O_{49}$, and WO_2.

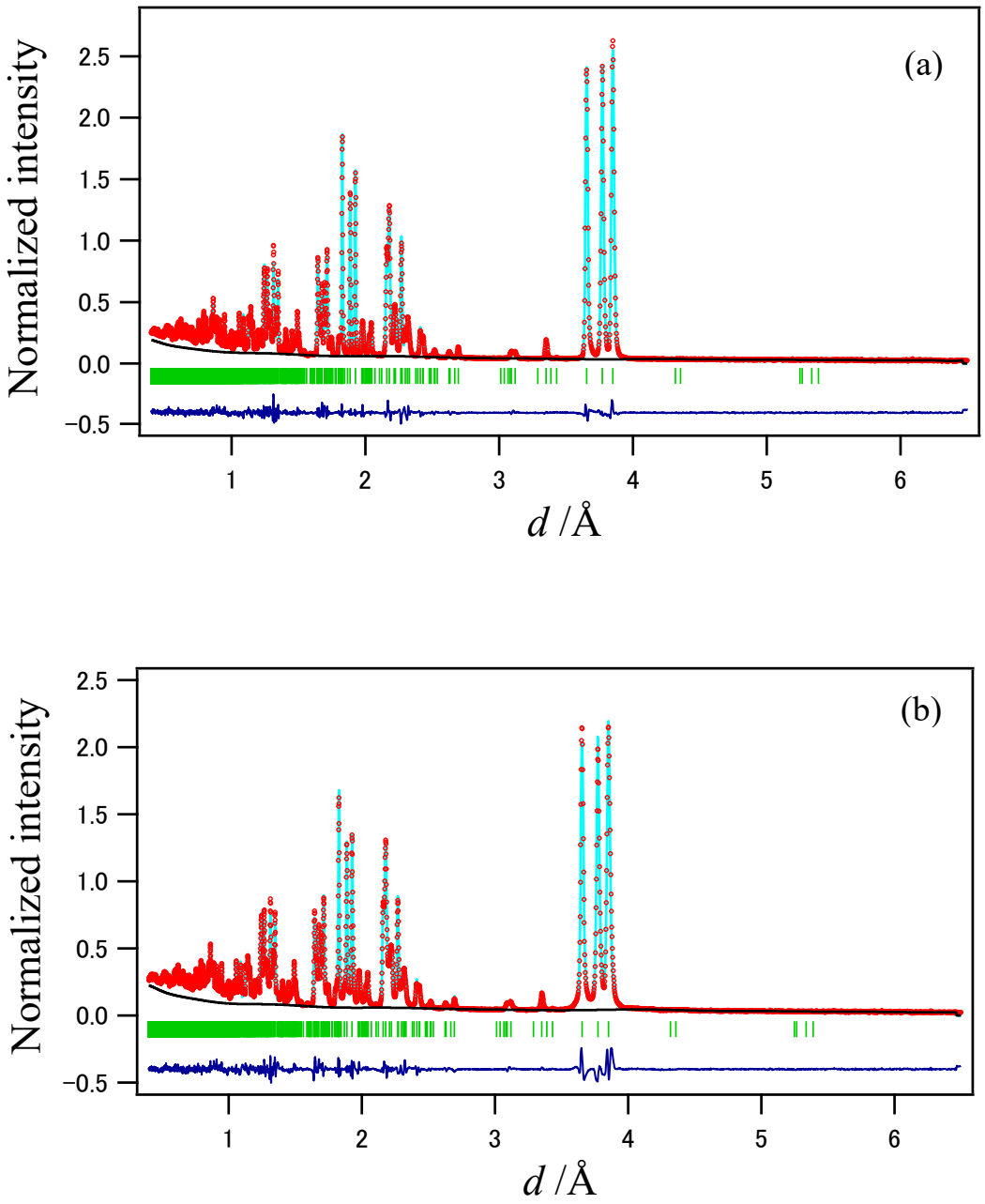


Figure S3. Rietveld refinement results of NPD measurements of WO_3 and WO_3-H samples before and after hydrogen treatment: (a) WO_3 and (b) WO_3-H.

Table S1. Crystallographic information (obtained by NPD) of WO_3 and WO_3-H samples

Temperature (°C)	500	
Isothermal holding time	0	30 min
Chemical formula	WO_3	WO_3
Crystal system	Monoclinic	Monoclinic
Space group	$P\,2_1/n$	$P\,2_1/n$
a (Å)	7.30959(1)	7.30759(1)
b (Å)	7.54425(1)	7.54425(1)
c (Å)	7.70015(1)	7.70552(1)
β (°)	90.7661(3)	90.7661(4)
V (Å^3)	424.6	424.8
R_{wp} (%)	6.25	7.21

Isothermal holding time at 500°C	Element	site	g	x	y	z	B_{iso} (Å^2)
WO_3 (0 min)	W (1)	4*e*	1	0.2516(1)	0.0248(1)	0.28381(7)	0.5
	W (2)	4*e*	1	0.2473(1)	0.0297(1)	0.78055(7)	0.5
	O (1)	4*e*	1	0.00068(9)	0.4660(1)	0.21425(8)	0.355(3)
	O (2)	4*e*	1	1.0010(1)	0.4660(1)	0.21780(8)	0.355(3)
	O (3)	4*e*	1	0.28473(8)	0.26144(8)	0.28197(8)	0.355(3)
	O (4)	4*e*	1	0.21082(8)	0.2582(1)	0.73406(8)	0.355(3)
	O (5)	4*e*	1	0.28510(8)	0.03963(8)	0.00664(8)	0.355(3)
	O (6)	4*e*	1	0.28521(7)	0.48837(7)	0.99337(4)	0.355(3)
WO_3 (30 min)	W (1)	4*e*	1	0.2540(1)	0.0245(1)	0.28182(8)	0.5
	W (2)	4*e*	1	0.2475(1)	0.0271(1)	0.77918(8)	0.5
	O (1)	4*e*	1	-0.00014(1)	0.0406(1)	0.21588(8)	0.406(3)
	O (2)	4*e*	1	0.9986(1)	0.4692(1)	0.22012(7)	0.406(3)
	O (3)	4*e*	1	0.28457(7)	0.2624(1)	0.27901(8)	0.406(3)
	O (4)	4*e*	1	0.21080(7)	0.2582(1)	0.73584(8)	0.406(3)
	O (5)	4*e*	1	0.28572(9)	0.03502(7)	0.00499(8)	0.406(3)
	O (6)	4*e*	1	0.28253(8)	0.49050(7)	0.99405(8)	0.406(3)