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Supporting information for article:

The influence of XRD pattern angular range on Rietveld refinement results at its use for quantitative analysis, crystallite size calculation and unit cell parameters refinement

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Supplementary materials

The influence of XRD pattern angular range on Rietveld refinement results at its using for quantitative analysis, crystallite size calculation and unit cell parameters refinement

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1. Table S1-1. Information about the crystalline phases (structure, space group, unit cell parameters) from Crystallographic Information Files used at Rietveld refinement.

Sample name	Patameters				
	Chemical composition	Cryst. System	Space Group	Unit cell parameters	*ICSD code
SRM 660	LaB_6	cubic	Pm-3m (221)	a=b=c=4.156950(6)	194636
Hematite	Fe_2O_3	rhombohedral	R-3cH (167)	a=b=5.0259(2) c=13.735(1)	415251
Magnetite	Fe_3O_4	cubic	Fd-3m (227)	a=b=c=8.370(2)	247034
Barium Titanate	$BaTiO_3$	tetragonal	P4mm (99)	a=b=3.9961(5) c=4.029(1)	245945
Copper	Cu	cubic	Fm-3m (225)	a=b=c= 3.61505	43493
Cuprite	Cu_2O	cubic	Pn-3mZ	a=b=c= 4.267(2)	63281
Whewellite	$CaC_2O_4 \cdot H_2O$	monoclinic	P21/c (14)	a= 16.250(1) b= 14.271(2) c= 10.114(2) β= 109.978(5)°	153499
Uricite	$C_5H_4N_4O_3$	monoclinic	P21/a (14)	a= 14.464(3) b= 7.404(2) c= 6.208(1) β= 109.978(5)°	56916
Quartz low	SiO_2	rhombohedral	P3221 (154)	a=b=4.9134 c=5.4052	174
Cristobalite low	SiO_2	tetragonal	P41212 (92)	a=b=4.978 c=6.948	9327
Anatase	TiO_2	tetragonal	I4/amd (141)	a=b= 3.799(1) c= 9.509(3)	154602
Rutile	TiO_2	tetragonal	P42/mnm (136)	a=b= 4.5911(7) c= 2.9924(9)	167953
Calcite	$CaCO_3$	rhombohedral	R-3cH(167)	a=b=4.9887(1) c=17.05289(80)	79673
Gypsum	$CaSO_4 \cdot 2H_2O$	monoclinic	C12/c1(15)	a= 6.28655 b= 15.202 c= 5.679 β= 114.17°	246243
Dimagnesium Diphosphate	$Mg_2P_2O_7$	monoclinic	P21/c(14)	a= 6.9443(4) b= 8.2861(4) c= 9.0438(5) β= 113.816(3)	261230
Kaolinite	$Al_2Si_2O_5(OH)_4$	triclinic	P1(1)	a=5.14971(4) b=8.93507(7) c=7.38549(5) α=91.9280(4)° β=105.0440(4)° γ=89.7920(5)°	63316
Muscovite 2M1	$KAl_{2.9}Si_{3.1}O_{10}(OH)_2$	monoclinic	C12/c1(15)	a= 5.189(1) b= 9.004(1) c= 20.256(6) β= 95.74(2)	202264
Corundum	Al_2O_3	rhombohedral	R-3cH (167)	a=b=4.757(1) c=12.9877(1)	92628
Boehmite	AlO(OH)	orthorombic	Cmcm (63)	a= 2.8681(1) b=12.2256(8) c= 3.6941(2)	93733

Iridium	Ir	cubic	Fm-3m (225)	a=b=c=3.8390	640729
Iridium Oxide	IrO_2	tetragonal	P42/mnm (136)	a=b= 4.4983 c= 3.1544	640888

*ICSD- Inorganic Crystal Structure Database

2. Results of Rietveld refinement

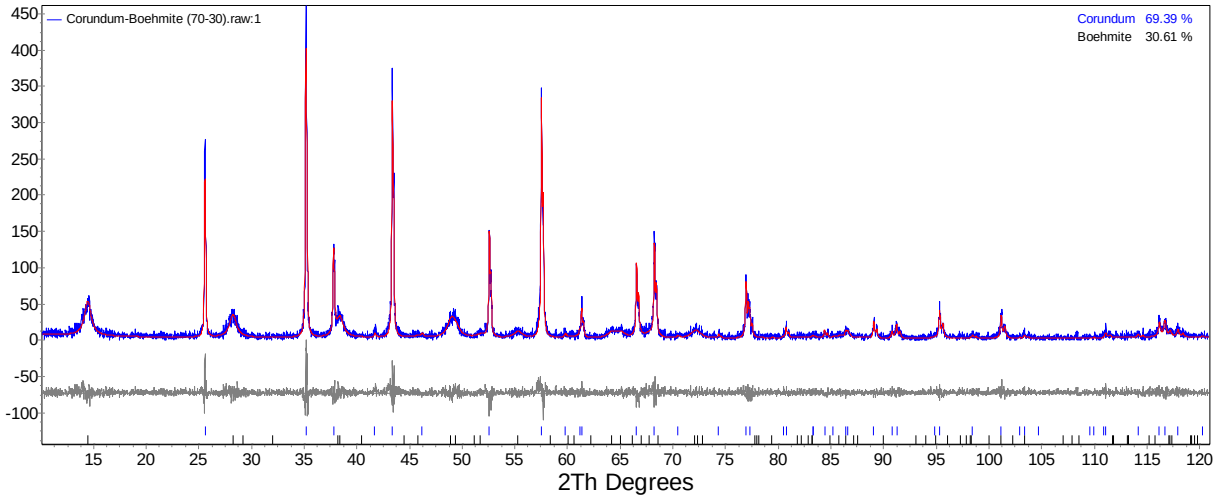

Fig.S1. Graphical representation of Rietveld refinement results of Corundum-Boehmite (70:30) mixture (the refined Bragg peak positions are shown by vertical bars)

Table S1. Results of Rietveld refinement of Corundum-Boehmite (70:30) artificial mixture.

Parameters	Angular range at Rietveld refinement, 2θ°						
	10-70	10-75	10-80	10-90	10-100	10-110	10-120
R_{wp}*	27.37	27.87	28.42	29.29	30.29	31.21	31.95
Corundum,wt % 4.0	68.6 4.0	68.3 3.3	69.7 2.9	71.9 1.2	69.4 1.0	69.2 1.0	69.39 0.98
a=b, Å** e.s.d.***	4.75766 0.00022	4.75763 0.00022	4.75761 0.00021	4.75753 0.00020	4.75733 0.00017	4.75733 0.00016	4.75729 0.00014
c, Å e.s.d.	12.98597 0.00086	12.98586 0.00084	12.98556 0.00062	12.98530 0.00058	12.98469 0.00055	12.98471 0.00050	12.98434 0.00047
d, nm** e.s.d.	74.5 1.8	74.5 1.8	75.2 1.8	74.3 1.7	73.9 1.7	73.5 1.6	72.7 1.6
Boehmite,wt% e.s.d.	31.4 4.0	31.7 3.3	30.3 2.9	28.7 4.2	30.6 1.0	30.8 1.0	30.61 0.98
a, Å e.s.d.	2.8692 0.0017	2.8690 0.0017	2.8687 0.0017	2.8687 0.0015	2.8684 0.0014	2.8684 0.0015	2.8680 0.0014
b, Å e.s.d.	12.2314 0.0080	12.2285 0.0079	12.2291 0.0079	12.2257 0.0072	12.2228 0.0071	12.2228 0.0071	12.2211 0.0069
c, Å e.s.d.	3.6934 0.0021	3.6921 0.0021	3.6921 0.0019	3.6912 0.0018	3.6911 0.0018	3.6910 0.0018	3.6901 0.0018
d, nm e.s.d.	10.74 0.34	10.80 0.34	10.78 0.34	10.68 0.32	10.71 0.32	10.70 0.32	10.74 0.33

* R_{wp} is residual-weighted parameter characterizing the refinement quality. $R_{wp}=\{\Sigma w_i[y_{i(obs)}-y_{i(calc)}]^2/\Sigma w_i[y_{i(obs)}]^2\}^{1/2}$ (where $y_{i(obs)}$ is the observed intensity at step I, $y_{i(calc)}$ is the calculated intensity, and w_i is the weight).

**a, b, c and d are calculated unit cell parameters an crystallite size correspondingly

***The Rietveld e.s.d. (estimated standard deviation) calculated by the TOPAS software.

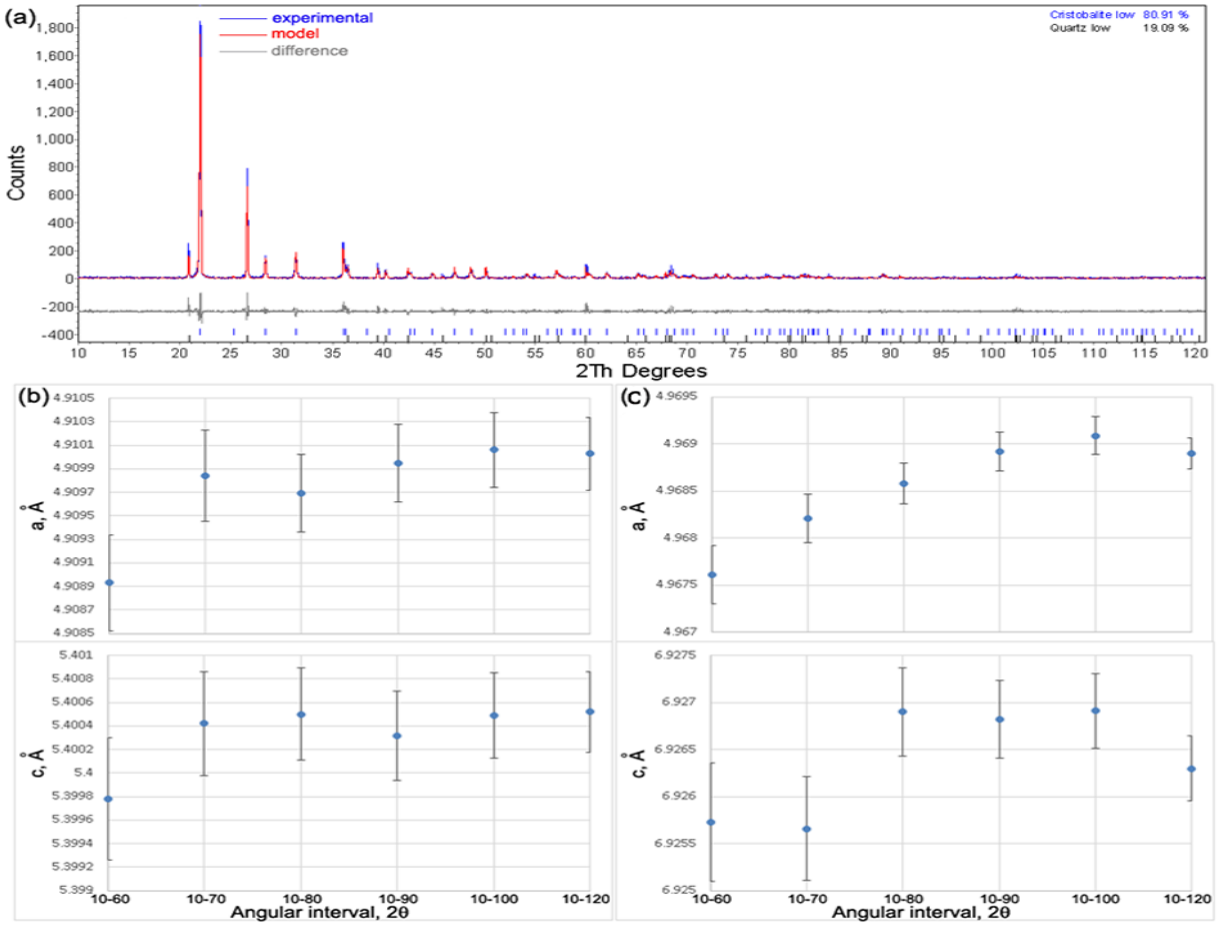

Fig.S2. Graphical representation of Rietveld refinement results of Cristobalite-Quartz (4:1) mixture (the refined Bragg peak positions are shown by vertical bars) (a) and the values of unit cell parameters of Cristobalite (b) and Quartz (c) calculated by Rietveld refinement for different angular intervals.

Table S2.Results of Rietveld refinement of Cristobalite-Quartz (80:20) artificial mixtures

Parameter	Angular range at Rietveld refinement, 2θ°				
	10-70	10-80	10-90	10-100	10-120
R_{wp}	30.81	31.8	32.74	33.46	35.22
Cristobalite, wt %	79.8	80.2	80.5	80.56	80.75
e.s.d.	1.2	1.1	1.0	0.95	0.84
a=*b*, Å	4.96821	4.96858	4.96892	4.96909	4.96890
e.s.d.	0.00026	0.00022	0.00021	0.00020	0.00017
c, Å	6.92873	6.92690	6.92682	6.92691	6.92630
e.s.d.	0.00055	0.00047	0.00041	0.00040	0.00035
d, nm	94.4	92.4	90.4	89.7	88.2
e.s.d.	3.3	3.1	3.0	2.4	2.7
Quartz, wt %	20.2	19.8	19.5	19.44	19.25
e.s.d.	1.2	1.1	1.0	0.95	0.84
a=*b*, Å	4.90984	4.90969	4.90995	4.91006	4.91003
e.s.d.	0.00022	0.00019	0.00018	0.00017	0.00016
c, Å	5.40042	5.40050	5.40032	5.40049	5.40052
e.s.d.	0.00044	0.00039	0.00038	0.00036	0.00034
d, nm	260	250	260	264	260
e.s.d.	150	81	150	94	140

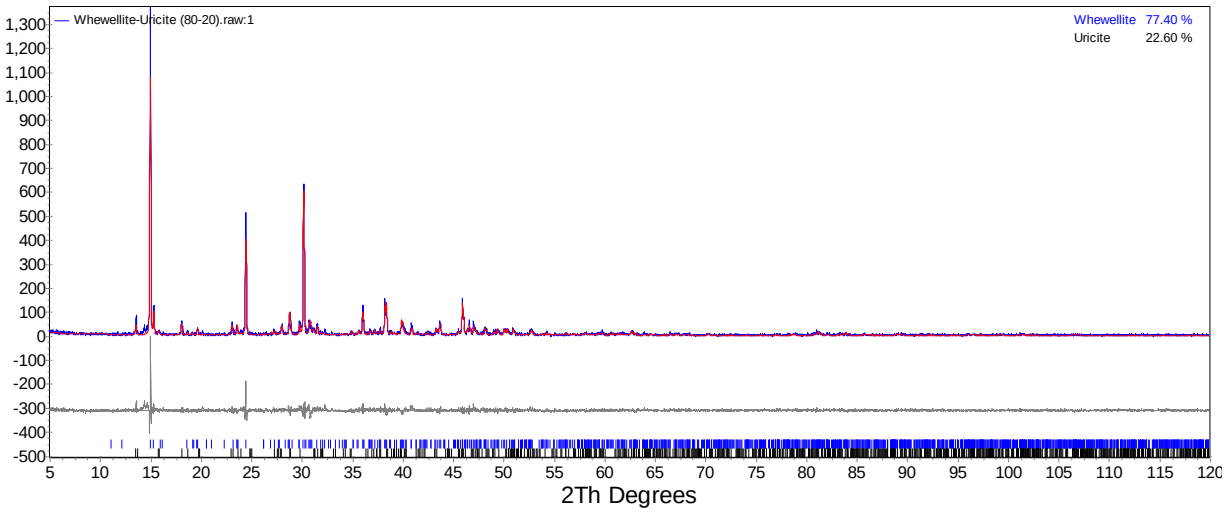

Fig.S3. Graphical representation of Rietveld refinement results for artificial Whewellite-Uricite (80:20) mixtures (the refined Bragg peak positions are shown by vertical bars).

Table S3.Results of Rietveld refinement of Whewellite-Uricite (80:20) artificial mixtures.

Parameters	Angular range at Rietveld refinement, 2θ°					
	5-60	5-70	5-80	5-90	5-100	5-120
R_{wp}	29.06	30.00	31.07	31.77	32.46	33.95
Whewellite, wt %	79.3	79.7	78.0	78.3	78.0	77.4
e.s.d.	3.6	4.0	3.7	4.0	4.9	4.1
a, Å	6.29063	6.29046	6.29052	6.28990	6.28976	6.29011
e.s.d.	0.00041	0.00039	0.00033	0.00033	0.00032	0.00031
b, Å	14.59050	14.58998	14.58972	14.58865	14.58829	14.58862
e.s.d.	0.00096	0.00092	0.00089	0.00082	0.00079	0.00076
c, Å	10.11627	10.11625	10.11600	10.11571	10.11534	10.11554
e.s.d.	0.00072	0.00067	0.00060	0.00058	0.00056	0.00053
β, °	109.4647	109.4642	109.4670	109.4616	109.4621	109.4656
e.s.d.	0.0059	0.0055	0.0055	0.0049	0.0046	0.0044
d, nm	207	208	206	207	207	200
e.s.d.	15	15	14	15	14	13
Uricite, wt %	20.7	20.3	22.0	21.7	22.0	22.6
e.s.d.	3.6	4.0	3.7	4.0	4.9	4.1
a, Å	14.4499	14.4491	14.44819	14.4464	14.4458	14.4467
e.s.d.	0.0047	0.0047	0.00026	0.0043	0.0042	0.0039
b, Å	7.4395	7.4388	7.4392	7.4382	7.4379	7.4386
e.s.d.	0.0020	0.0020	0.0022	0.0019	0.0019	0.0022
c, Å	6.2011	6.2007	6.2001	6.2012	6.2015	6.2022
e.s.d.	0.0033	0.0032	0.0032	0.0030	0.0029	0.0022
β, °	65.095	65.100	65.102	65.100	65.099	65.097
e.s.d.	0.0031	0.0031	0.0030	0.0029	0.0028	0.0020
d, nm	73.8	78.3	79.0	78.7	78.5	75.9
e.s.d.	8.7	9.5	3.7	9.3	9.01	8.5

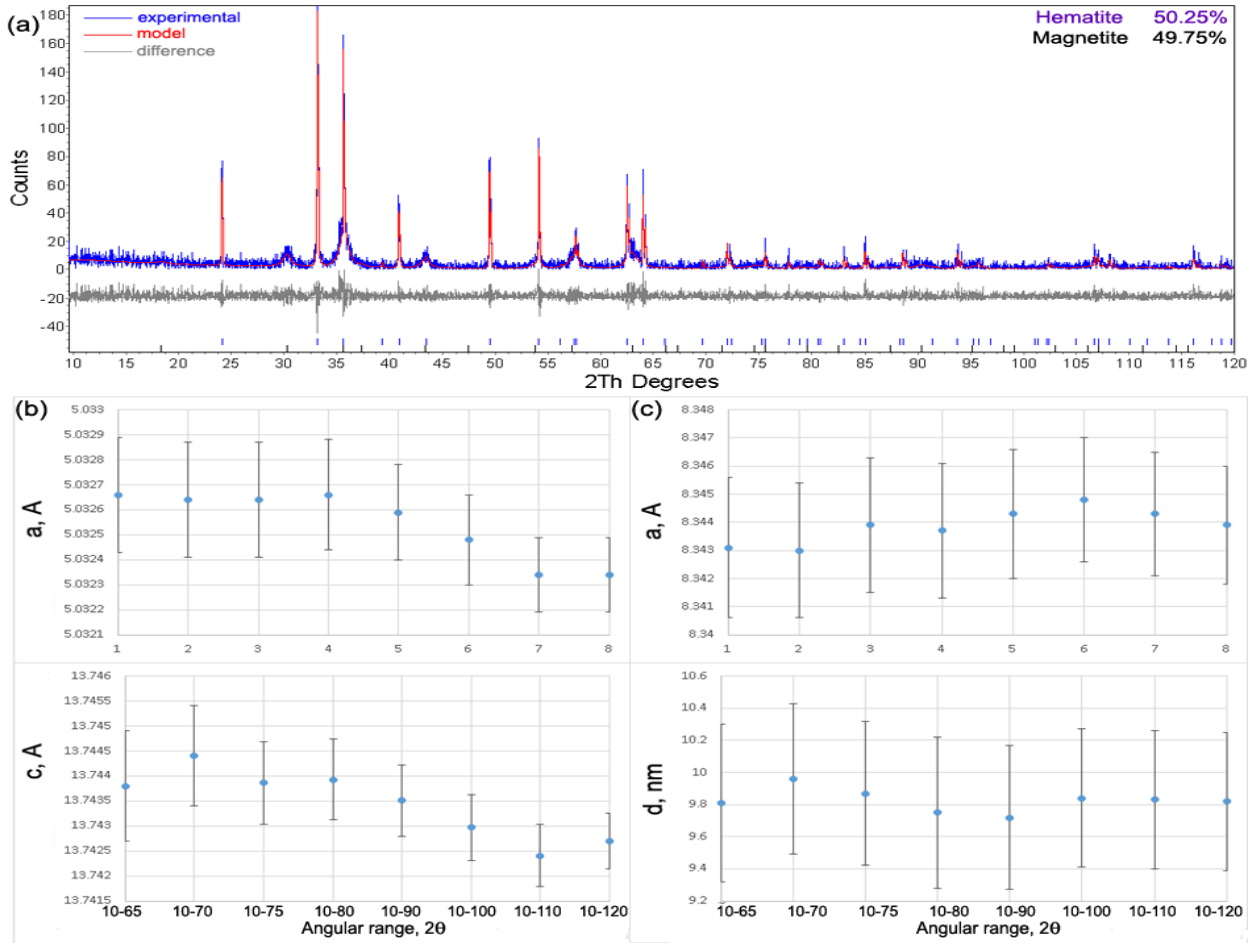

Fig.S4. Graphical representation of Rietveld refinement results for Hematite-Magnetite (1:1) artificial mixture (the refined Bragg peak positions are shown by vertical bars) (a), the values of unit cell parameters of Hematite (b) and the values of unit cell parameters and crystallite size of Magnetite (c) calculated by Rietveld refinement for different angular intervals.

Table S4.Results of Rietveld refinement of Hematite-Magnetite (1:1) mixture.

Parameter	Angular range at Rietveld refinement, 2θ°							
	10-65	10-70	10-75	10-80	10-90	10-100	10-110	10-120
R_{wp}	39.60	40.53	41.25	41.86	42.81	43.87	44.57	45.51
Hematite,%	49.35	49.56	48.51	49.20	51.10	49.56	51.44	50.25
e.s.d.	0.72	0.71	0.71	0.66	0.54	0.54	0.52	0.48
a=*b*, Å	5.03266	5.03264	5.03264	5.03266	5.03259	5.03248	5.03234	5.03234
e.s.d.	0.00023	0.00023	0.00023	0.00022	0.00019	0.00018	0.00015	0.00015
c, Å	13.74380	13.74440	13.74386	13.74393	13.74351	13.74297	13.74241	13.74270
e.s.d.	0.00110	0.00100	0.00082	0.00081	0.00072	0.00066	0.00062	0.00055
d, nm	245	245	246	239	236	236	234	232
e.s.d.	23	22	36	21	19	19	18	18
Magnetite,%	50.65	50.44	51.49	50.80	48.90	50.44	48.57	49.75
e.s.d.	0.72	0.71	0.71	0.66	0.54	0.54	0.49	0.48
a=*b*=*c*, Å	8.3431	8.3430	8.3439	8.3437	8.3443	8.3448	8.3443	8.3439
e.s.d.	0.0025	0.0024	0.0024	0.0024	0.0023	0.0022	0.0022	0.0021
d, nm	9.81	9.96	9.87	9.75	9.72	9.84	9.83	9.82
e.s.d.	0.49	0.47	0.45	0.47	0.45	0.43	0.43	0.43

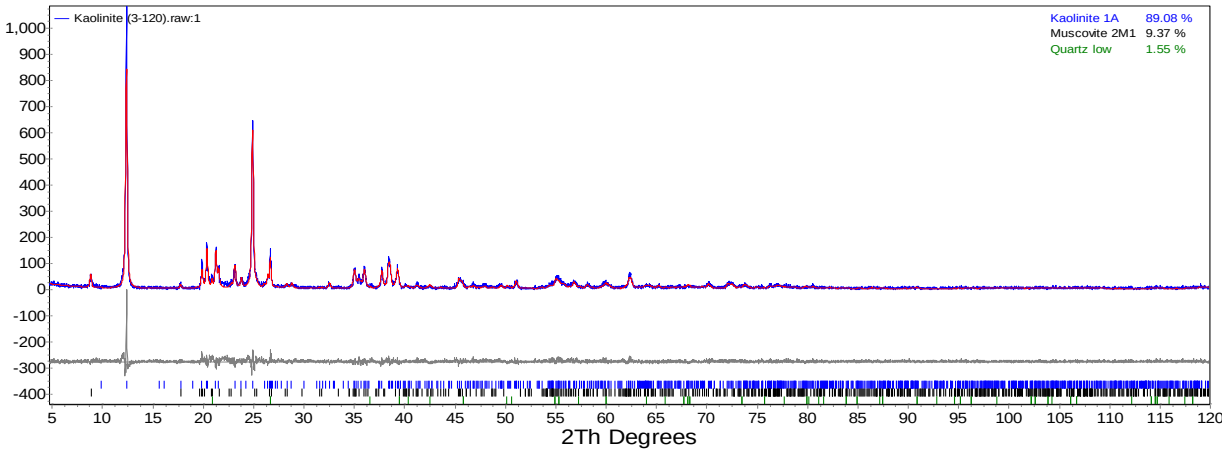

Fig.S5. Graphical representation of Rietveld refinement results of Kaolinite (the refined Bragg peak positions are shown by vertical bars)

Table S5.Results of Rietveld refinement of Kaolinite.

Parameters	Angular range at Rietveld refinement, 2θ°					
	5-60	5-65	5-75	5-90	5-100	5-120
R_{wp}	24.95	25.5	26.03	27.55	28.35	29.71
Kaolinite, wt % e.s.d.	84.6 5.5	88.6 2.4	87.6 2.1	91.3 2.9	89.5 2.7	89.1 1.9
a, Å e.s.d.	5.15182 0.00094	5.15236 0.00075	5.15282 0.00068	5.1522 0.00068	5.15197 0.00067	5.15222 0.00063
b, Å e.s.d.	8.9385 0.0017	8.9402 0.0016	8.94 0.0014	8.9392 0.0013	8.939 0.0012	8.9405 0.0012
c, Å e.s.d.	7.3959 0.001	7.39637 0.00099	7.39664 0.00084	7.39622 0.00077	7.39795 0.00068	7.39795 0.00038
α, ° e.s.d.	91.689 0.013	91.696 0.012	91.697 0.011	91.712 0.01	91.712 0.0098	91.721 0.0098
β, ° e.s.d.	104.832 0.014	104.815 0.012	104.811 0.01	104.825 0.0097	104.839 0.0093	104.834 0.0090
λ, ° e.s.d.	89.786 0.013	89.794 0.011	89.794 0.011	89.786 0.01	89.784 0.01	89.7908 0.0098
d, nm e.s.d.	68 1.8	66.1 1.7	65.3 1.6	66.5 1.6	66.3 1.6	64.6 1.5
Muscovite, wt % e.s.d.	11.7 5.5	9.4 2.5	10.7 2.2	7.1 2.9	8.9 2.7	9.4 2
a, Å e.s.d.	5.1904 0.0024	5.1969 0.0011	5.1966 0.0011	5.1897 0.0019	5.189 0.0019	5.1727 0.0021
b, Å e.s.d.	8.9506 0.0071	9.0032 0.0026	9.0016 0.0028	8.9549 0.0051	8.9549 0.0052	9.0433 0.0051
c, Å e.s.d.	20.0825 0.0079	20.0863 0.0064	20.0913 0.0058	20.092 0.0066	20.0816 0.0064	20.1231 0.0061
β, ° e.s.d.	95.989 0.052	95.61 0.049	95.562 0.044	95.887 0.054	95.966 0.053	96.809 0.058
d, nm e.s.d.	83 12	87 18	76 13	78 11	79 11	89 9.4
Quartz, wt % e.s.d.	3.64 0.76	2 1.6	1.7 1.4	1.6 1.4	1.6 1.3	1.5 1.3
a=*b*, Å e.s.d.	4.9058 0.0021	4.9103 0.0012	4.9092 0.0012	4.91141 0.00098	4.91097 0.0009	4.91067 0.00079
c, Å e.s.d.	5.413 0.58	5.4099 0.0025	5.4118 0.0022	5.4053 0.0019	5.4071 0.0017	5.4076 0.0015

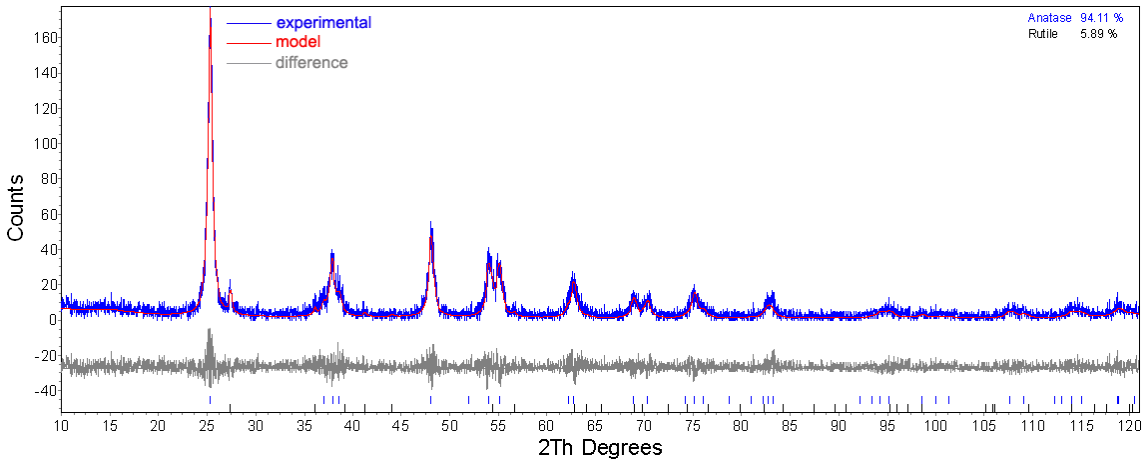

Fig.S6. Graphical representation of Rietveld refinement results for P90 Degussa specimen (the refined Bragg peak positions are shown by vertical bars).

Table S6.Results of Rietveld refinement of P90 Degussa sample.

Parameter	Angular range at Rietveld refinement, 2θ°							
	10-60	10-65	10-72	10-80	10-90	10-100	10-110	10-120
R_{wp},%	34.27	34.9	35.73	36.76	37.98	39.08	39.94	40.55
Anatase, wt %	94.83	94.75	94.94	94.63	94.61	94.56	94.55	94.51
e.s.d.	0.48	0.58	0.60	0.57	0.53	0.51	0.50	0.49
a=*b*, Å	3.7855	3.7850	3.7838	3.7833	3.7831	3.7834	3.7834	3.78331
e.s.d.	0.0031	0.0027	0.0022	0.0017	0.0014	0.0014	0.0012	0.00094
c=Å	9.4963	9.4960	9.4939	9.4922	9.4921	9.4930	9.4927	9.4932
e.s.d.	0.0081	0.0071	0.0056	0.0045	0.0039	0.0038	0.0033	0.0024
d, nm	16.59	16.43	16.60	16.59	16.60	16.62	16.64	16.62
e.s.d.	0.43	0.41	0.39	0.39	0.39	0.37	0.35	0.34
Rutile, wt %	5.17	5.25	5.06	5.37	5.39	5.44	5.45	5.49
e.s.d.	0.48	0.58	0.60	0.57	0.53	0.51	0.50	0.49
a=*b*, Å	4.5942	4.5932	4.5913	4.5907	4.5904	4.5921	4.5921	4.5923
e.s.d.	0.0045	0.0043	0.0036	0.0032	0.0029	0.0028	0.0026	0.0025
c,Å	2.9585	2.9594	2.9562	2.9567	2.9553	2.9550	2.9552	2.9556
e.s.d.	0.0047	0.0045	0.0042	0.0040	0.0036	0.0036	0.0035	0.0034
d, nm	35.5	30.3	31.1	31.2	32.8	31.8	31.8	31.8
e.s.d.	7.5	5.7	6.0	5.9	.2	5.7	5.7	5.5

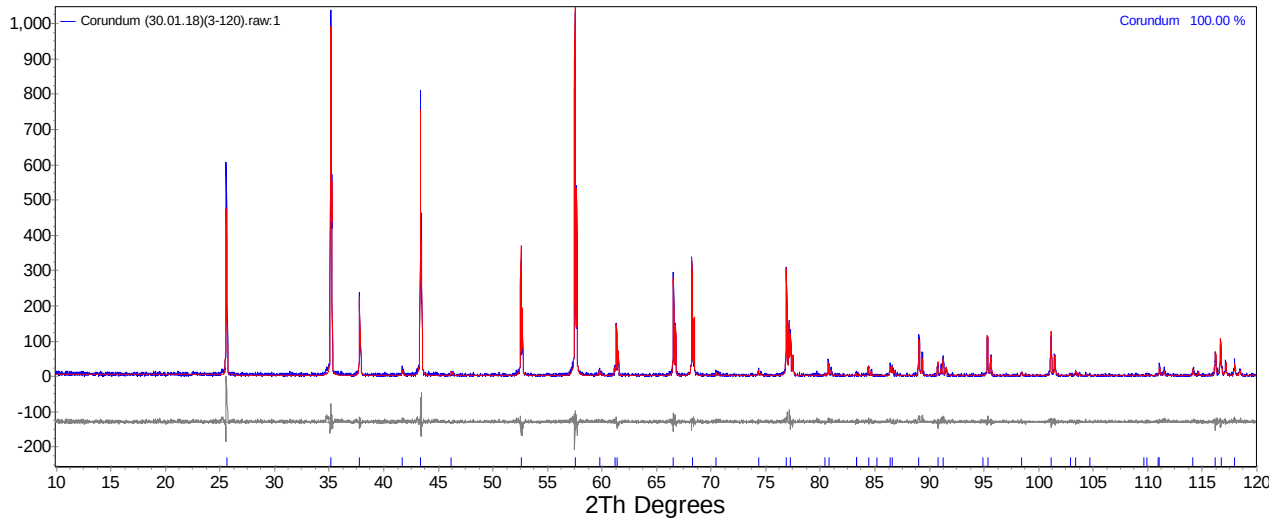

Fig.S7. Graphical representation of Rietveld refinement results of Corundum (Al_2O_3) (the refined Bragg peak positions are shown by vertical bars).

Table S7.Results of Rietveld refinement of Corundum (Al_2O_3).

Parameters	Angular range at Rietveld refinement, 2θ°							
	10-65	10-70	10-75	10-80	10-90	10-100	10-110	10-120
R_{wp}	29.04	28.58	28.95	29.51	30.16	31.04	31.85	32.25
a=*b*, Å e.s.d.	4.75710 0.00012	4.756529 0.000072	4.756494 0.000071	4.756358 0.000069	4.756363 0.000043	4.756349 0.000054	4.756354 0.000052	4.756273 0.000056
c, Å e.s.d.	12.98848 0.00032	12.98771 0.00030	12.98784 0.00029	12.98666 0.00018	12.98664 0.00016	12.98652 0.00015	12.98655 0.00014	12.98653 0.00013
d, nm e.s.d.	1108 83	1229 80	1137 80	1118 75	1141 78	1145 76	1125 72	1111 68

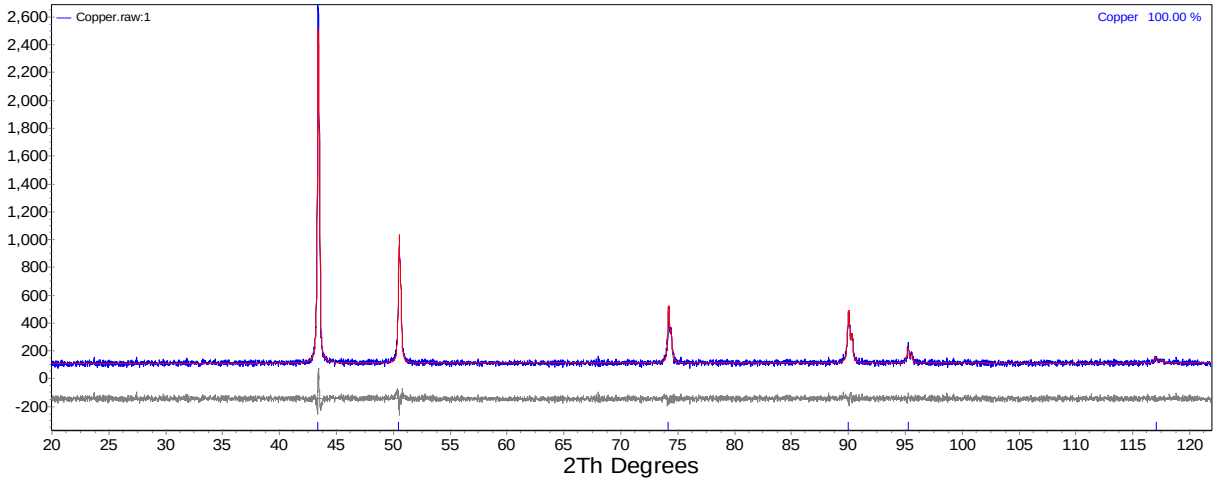

Fig.S8. Graphical representation of Rietveld refinement results of Copper (the refined Bragg peak positions are shown by vertical bars).

Table S8. Results of Rietveld refinement of copper.

Parameters	Angular range at Rietveld refinement, 2θ°			
	20-80	20-92	20-100	20-120
R_{wp}	9.25	9.23	9.25	9.31
a, Å e.s.d.	3.61314 0.00018	3.61287 0.00010	3.612832 0.000093	3.612861 0.000090
d, nm e.s.d.	93.2 2.4	87.9 2.1	87.9 2.1	87.1 2.0

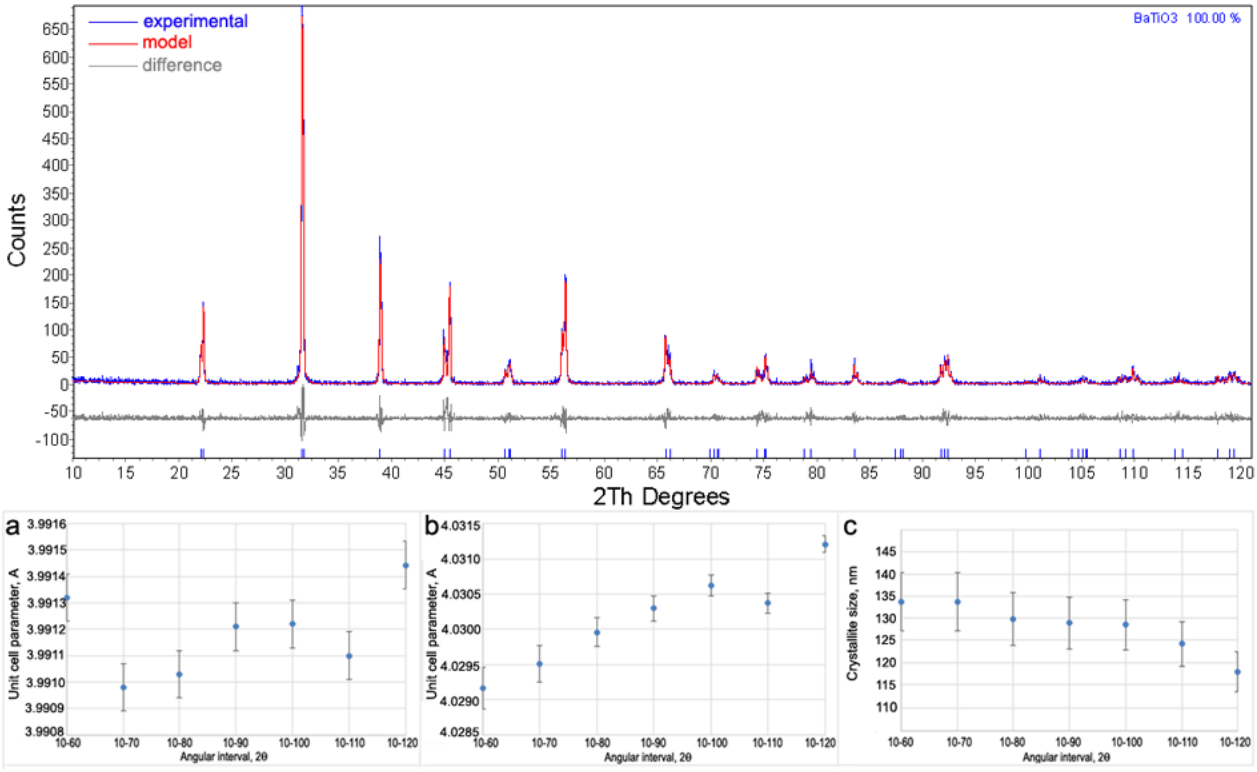

Fig.S9. Graphical representation of Rietveld refinement results of $BaTiO_3$ (the refined Bragg peak positions are shown by vertical bars) (top) and the values of unit cell parameters a=b (a), c (b), and crystallite size (c) of $BaTiO_3$ calculated by Rietveld refinement.

Table S9.Results of Rietveld refinement of $BaTiO_3$.

Parameter	Angular range at Rietveld refinement, 2θ°						
	10-60	10-70	10-80	10-90	10-100	10-110	10-120
R_{wp},%	32.2	34.3	35.04	35.95	36.45	37.28	38.09
a=*b*, Å e.s.d.	3.99132 0.00026	3.99098 0.00022	3.99103 0.00017	3.99121 0.00015	3.99122 0.00013	3.9911 0.00011	3.99144 0.00009
c, Å e.s.d.	4.02916 0.0003	4.02951 0.00026	4.02996 0.0002	4.0303 0.00018	4.03062 0.00015	4.03037 0.00014	4.03121 0.00012
d, nm e.s.d.	133.8 6.6	133.7 6.6	129.9 6	128.9 5.8	128.5 5.6	124.2 5.1	117.9 4.5

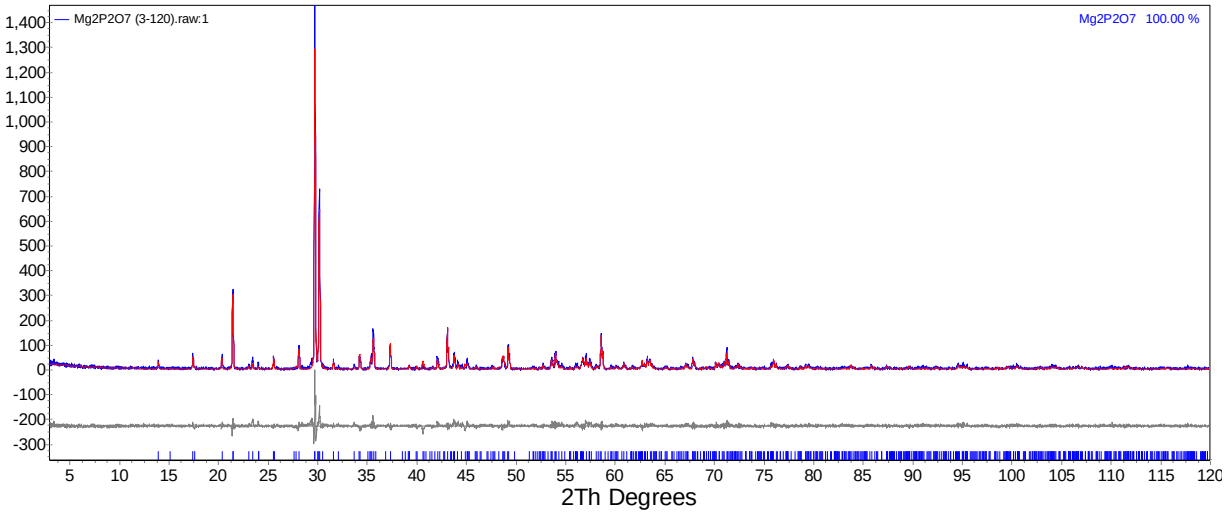

Fig.S10. Graphical representation of Rietveld refinement results for $Mg_2P_2O_7$ (the refined Bragg peak positions are shown by vertical bars).

Table S10. Results of Rietveld refinement of $Mg_2P_2O_7$.

Parameters	Angular range at Rietveld refinement, 2θ°					
	5-65	5-70	5-80	5-90	5-100	5-120
R_{wp}	29.06	33.36	33.69	34.39	35.23	36.1
a, Å	6.94286	6.94258	6.94228	6.94248	6.94229	6.94200
e.s.d.	0.00044	0.00040	0.00038	0.00036	0.00035	0.00032
b, Å	8.28742	8.28648	8.28657	8.28651	8.28638	8.28606
e.s.d.	0.00052	0.00042	0.00040	0.00039	0.00038	0.00036
c, Å	9.04355	9.04282	9.04267	9.04268	9.04262	9.04225
e.s.d.	0.00059	0.00058	0.00049	0.00046	0.00045	0.00041
β, °	113.840	113.843	113.842	113.843	113.842	113.842
e.s.d.	0.003	0.003	0.002	0.002	0.002	0.002
d, nm	172	169	177	173	173	173
e.s.d.	43	44	41	36	36	34

1. **Graphical representation of differences of parameters calculated at different angular ranges.**

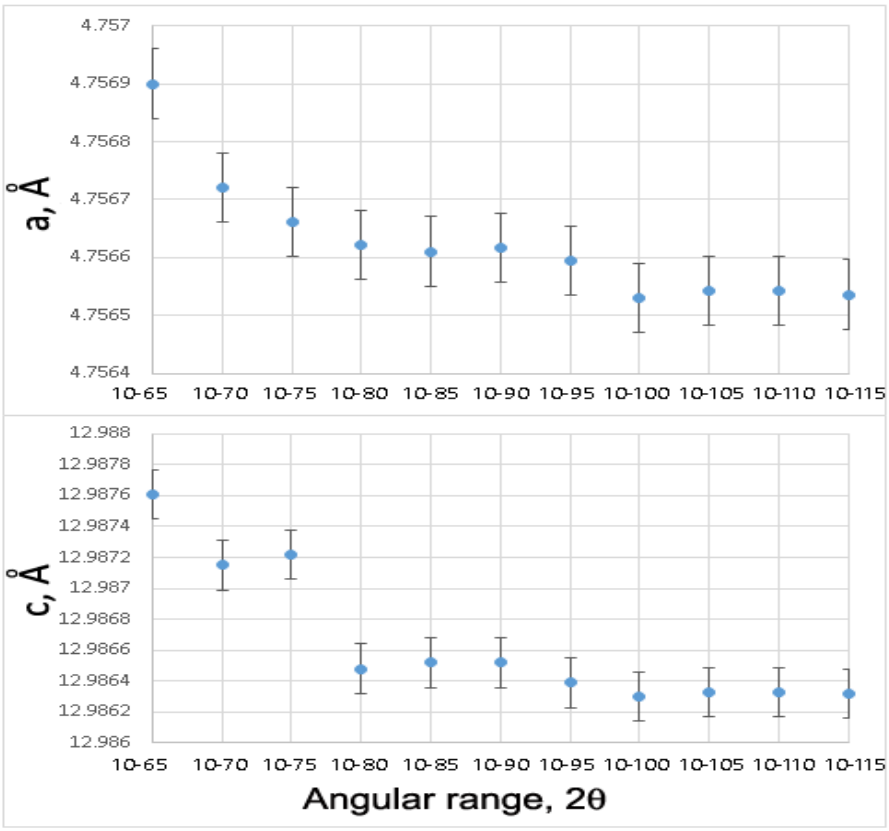

Fig.S11. The values of unit cell parameters of Corundum calculated by Rietveld refinement for different angular intervals.

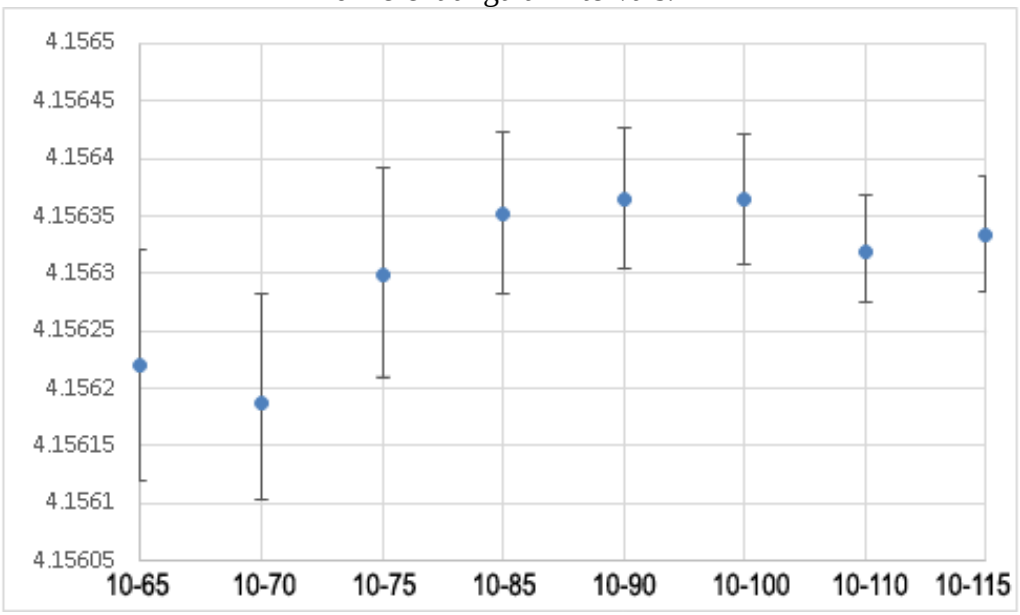

Fig.S12. The values of unit cell parameter of LaB_6 calculated by Rietveld refinement for different angular intervals.

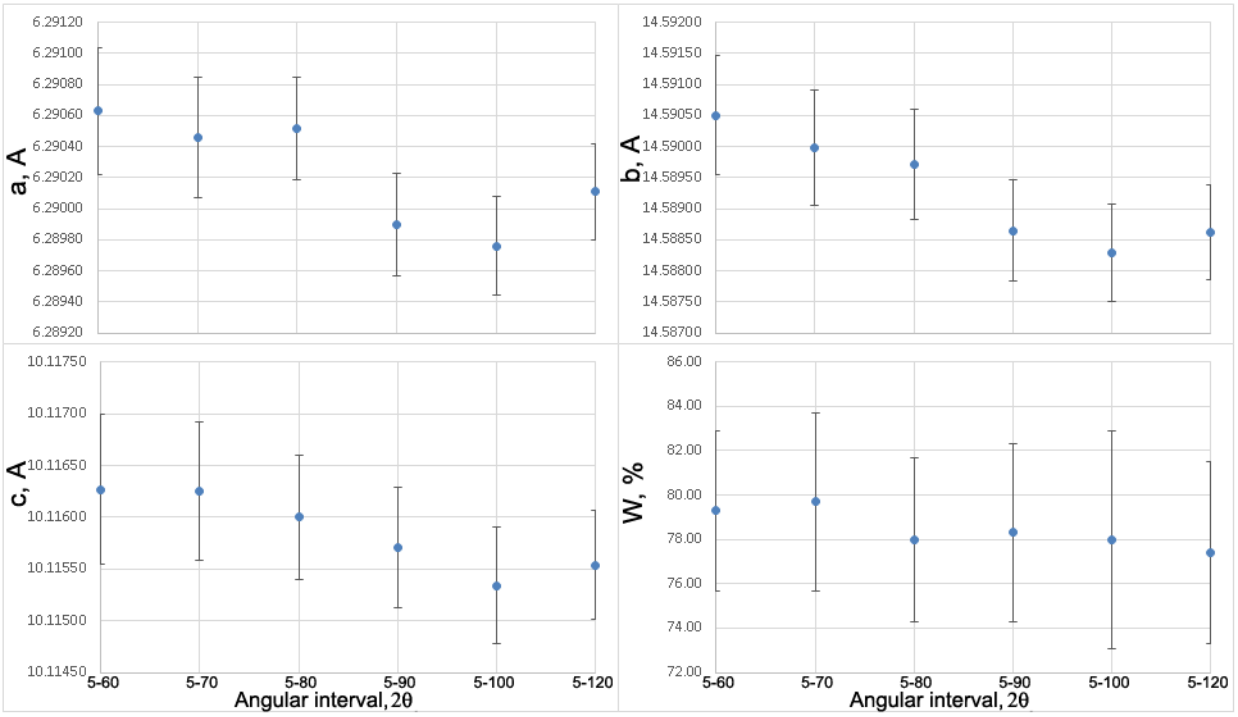

Fig.S13. The values of unit cell parameters and percentage of Whewellite calculated by Rietveld refinement for different angular intervals.

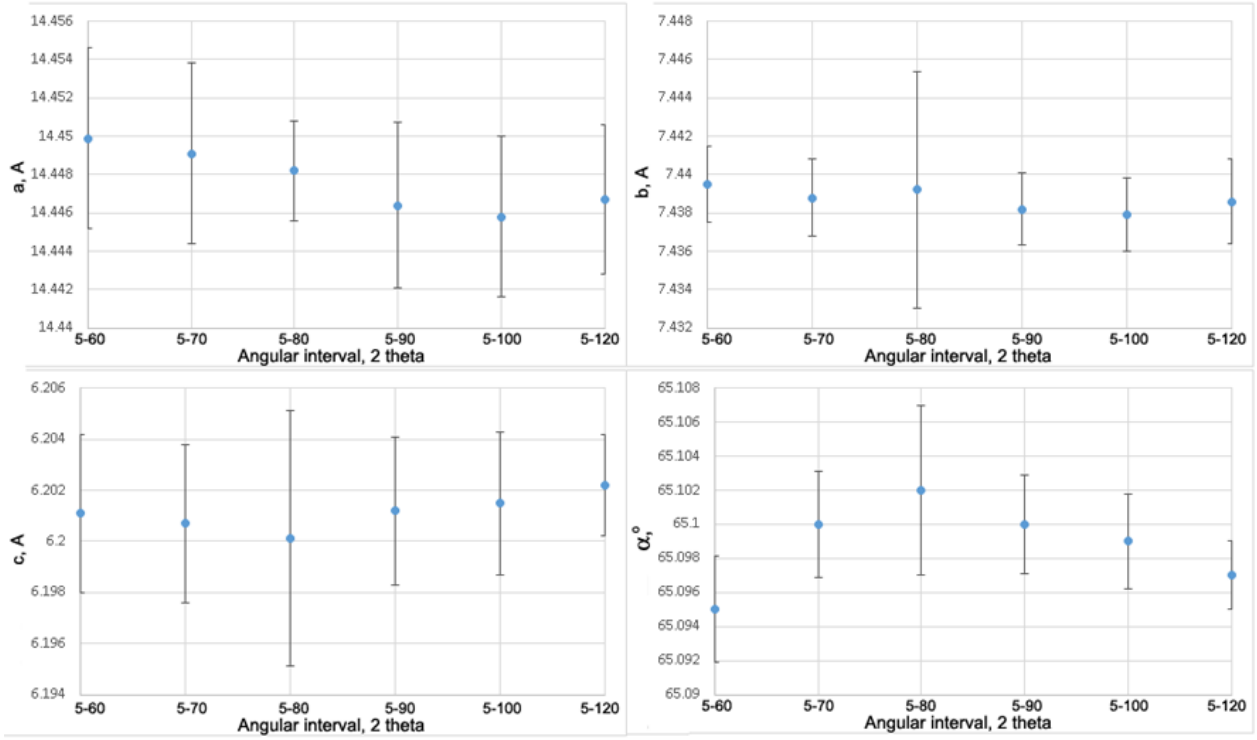

Fig.S14. The values of unit cell parameters and percentage of Uricite calculated by Rietveld refinement for different angular intervals.

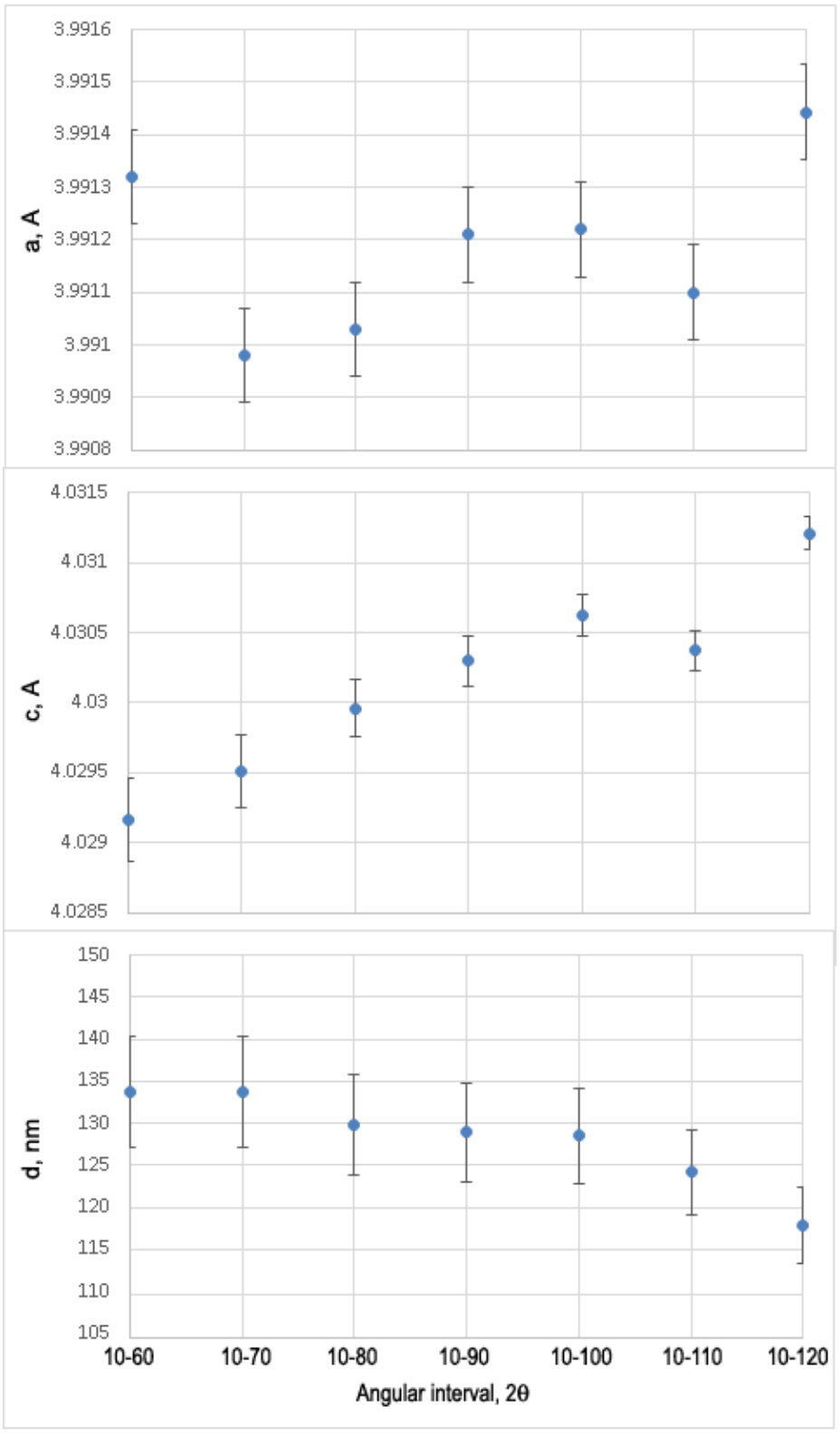

Fig.S15. The values unit cell parameters and crystallite size of $BaTiO_3$ calculated by Rietveld refinement for different angular intervals.

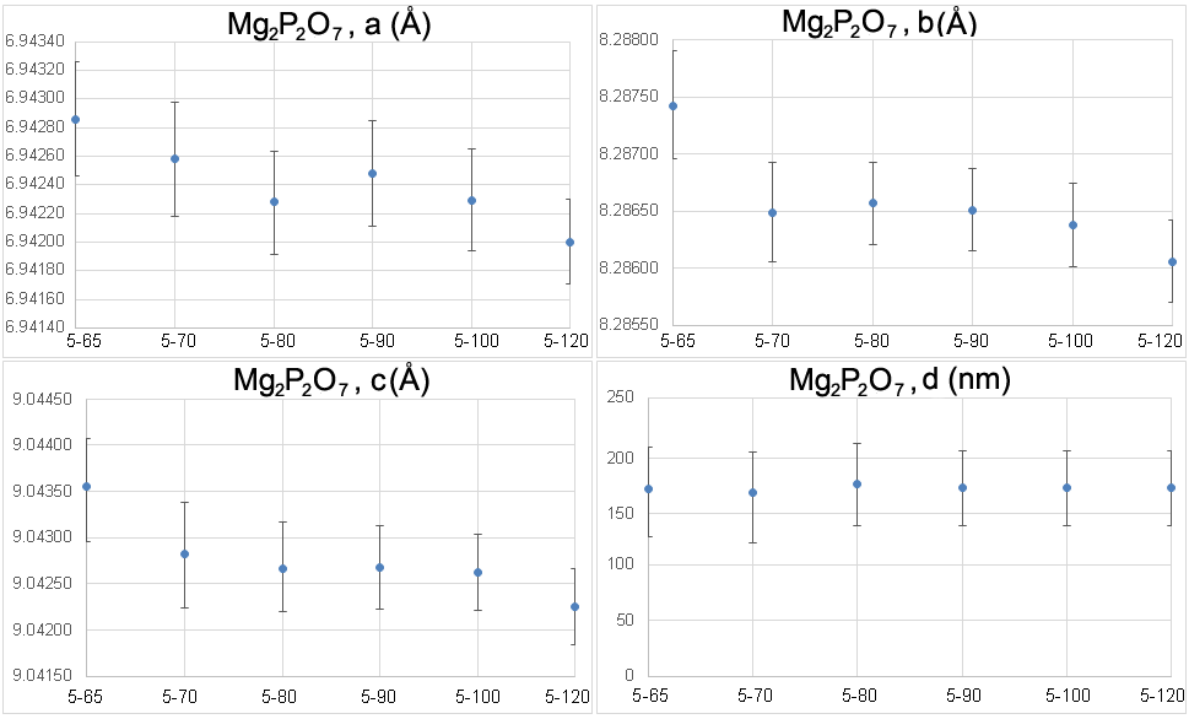

Fig.S16. The values of unit cell parameters and crystallite sizes of $Mg_2P_2O_7$ calculated by Rietveld refinement for different angular intervals.

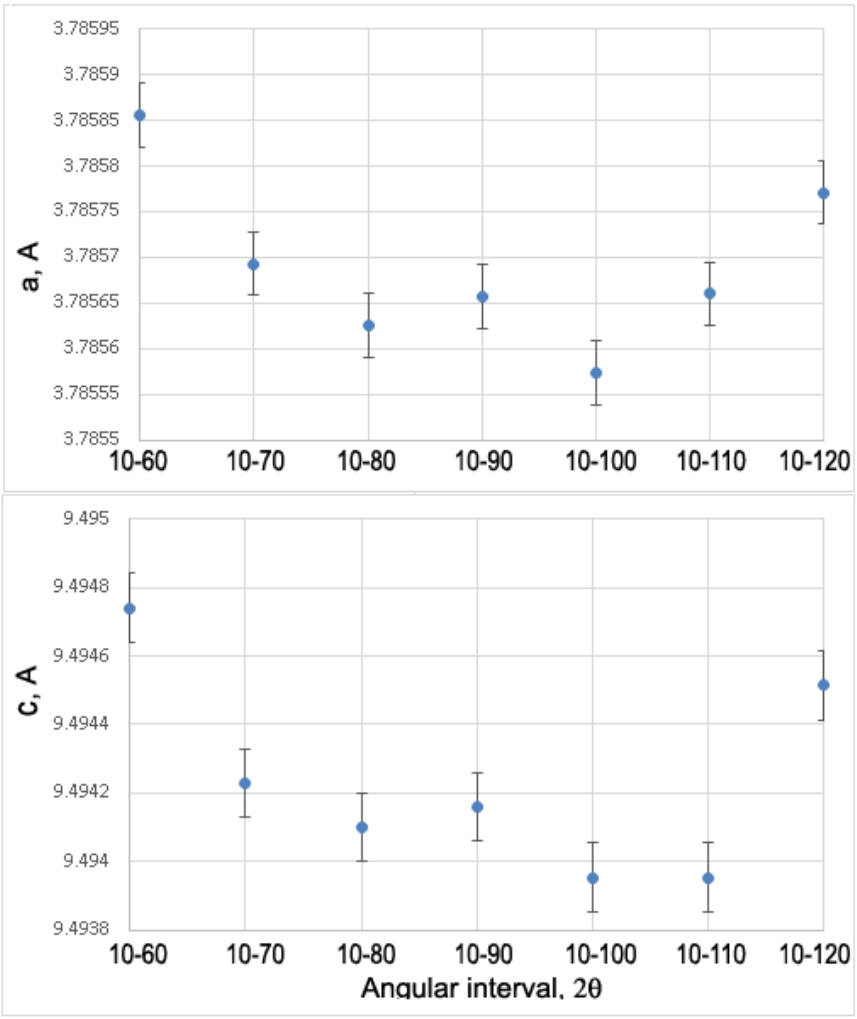

Fig.S17. The values unit cell parameters and crystallite size of Anatase (model, ICSD-154604) calculated by Rietveld refinement for different angular intervals.

2. Results of the reproducibility test

Table S11. Means and Standard deviations calculated for $BaTiO_3$ sample at reproducibility test.

Parameter	Mean and Standard deviations for different angular intervals						
	10-60	10-70	10-80	10-90	10-100	10-110	10-120
a=*b*, Å	3.9916 0.00062	3.9917 0.00054	3.9915 0.00040	3.9916 0.00028	3.9916 0.00028	3.9915 0.00028	3.9917 0.00021
c, Å	4.02937 0.00087	4.0298 0.00071	4.0299 0.00054	4.0300 0.00041	4.0302 0.00040	4.0302 0.00038	4.0307 0.00037
d, nm	136.5 11.3	128.2 13.32	127.9 9.97	128.2 9.73	126.1 9.00	122.7 8.34	116.4 7.11

Table S12. Means and Standard deviations calculated for Corundum sample at reproducibility test.

Parameter	Mean and Standard deviations for different angular intervals									
	10-70	10-75	10-80	10-85	10-90	10-95	10-100	10-105	10-110	10-115
a=*b*, Å	4.75718 0.00049	4.75716 0.00050	4.75711 0.00049	4.75712 0.00050	4.75707 0.000470	4.75706 0.00047	4.75702 0.00047	4.75683 0.00050	4.75700 0.00046	4.75699 0.00047
c, Å	12.98921 0.00140	12.98919 0.00140	12.98842 0.00150	12.98841 0.00149	12.98831 0.00147	12.98819 0.00140	12.98813 0.00140	12.98773 0.00148	12.98808 0.00135	12.98802 0.00134

3. Comparison of Ritveld refinement results at “Zero error” and “Specimen displacement” as refined parameters.

Table S13. Rietveld refinement results of Hematite-Magnetite (1:1) artificial mixture obtained at "Zero error"(a) and "Specimen displacement"(b) corrections.

(a)

Parameters	Angular range at Rietveld refinement, 2θ°							
	10-65	10-70	10-75	10-80	10-90	10-100	10-110	10-120
R_{wp}	39.75	40.77	41.54	42.00	43.03	44.06	44.81	45.64
Hematite, wt%	50.31	50.45	50.79	51.01	51.03	51.46	51.66	51.41
e.s.d.	0.66	0.63	0.60	0.58	0.56	0.54	0.51	0.50
a=*b*, Å	5.03263	5.03265	5.03264	5.03261	5.03254	5.03240	5.03232	5.03226
e.s.d.	0.00031	0.00032	0.00030	0.00029	0.00025	0.00024	0.00020	0.00020
c, Å	13.7442	13.7446	13.7440	13.74410	13.74359	13.74296	13.74253	13.74263
e.s.d.	0.0012	0.0012	0.0010	0.00093	0.00087	0.00079	0.00072	0.00066
d, nm	234	254	250	233	221	219	219	231
e.s.d.	43	57	55	60	42	47	47	43
Magnetite, wt%	49.69	49.55	48.21	48.99	48.97	48.54	48.34	48.59
e.s.d.	0.66	0.63	0.60	0.58	0.56	0.54	0.51	0.50
a=*b*=*c*, Å	8.3450	8.3449	8.3453	8.3450	8.3451	8.3459	8.3451	8.3453
e.s.d.	0.0024	0.0024	0.0024	0.0024	0.0023	0.0022	0.0021	0.0021
d, nm	9.63	9.79	9.74	9.74	9.52	9.72	9.78	9.75
e.s.d.	0.47	0.45	0.44	0.43	0.41	0.41	0.41	0.40

(b)

Parameters	Angular range at Rietveld refinement, 2θ°							
	10-65	10-70	10-75	10-80	10-90	10-100	10-110	10-120
R_{wp}	39.94	40.79	41.51	42.05	42.95	43.98	44.66	45.63
Hematite, wt%	51.05	50.78	51.03	51.08	51.10	51.44	51.44	51.49
e.s.d.	0.59	0.61	0.59	0.57	0.54	0.52	0.52	0.48
a=*b*, Å	5.03249	5.03249	5.03250	5.03249	5.03243	5.03233	5.03219	5.03220
e.s.d.	0.00030	0.00028	0.00029	0.00027	0.00024	0.00023	0.00020	0.00019
c, Å	13.7438	13.7442	13.74361	13.74363	13.74318	13.74272	13.74207	13.74242
e.s.d.	0.0011	0.0011	0.00091	0.00091	0.00082	0.00076	0.00072	0.00061
d, nm	241	251	245	244	243	243	242	237
e.s.d.	49	38	36	36	34	33	33	30
Magnetite, wt%	48.95	49.22	48.97	48.92	48.90	48.56	48.57	48.51
e.s.d.	0.59	0.61	0.59	0.57	0.54	0.52	0.49	0.48
a=*b*=*c*, Å	8.3459	8.3445	8.3449	8.3449	8.3450	8.3458	8.3458	8.3452
e.s.d.	0.0024	0.0024	0.0024	0.0030	0.0030	0.0021	0.0021	0.0021
d, nm	10.20	9.84	9.75	9.74	9.65	9.74	9.75	9.73
e.s.d.	0.50	0.45	0.44	0.43	0.42	0.41	0.40	0.40

4. Results of Rietveld refinement of simulated XRD patterns

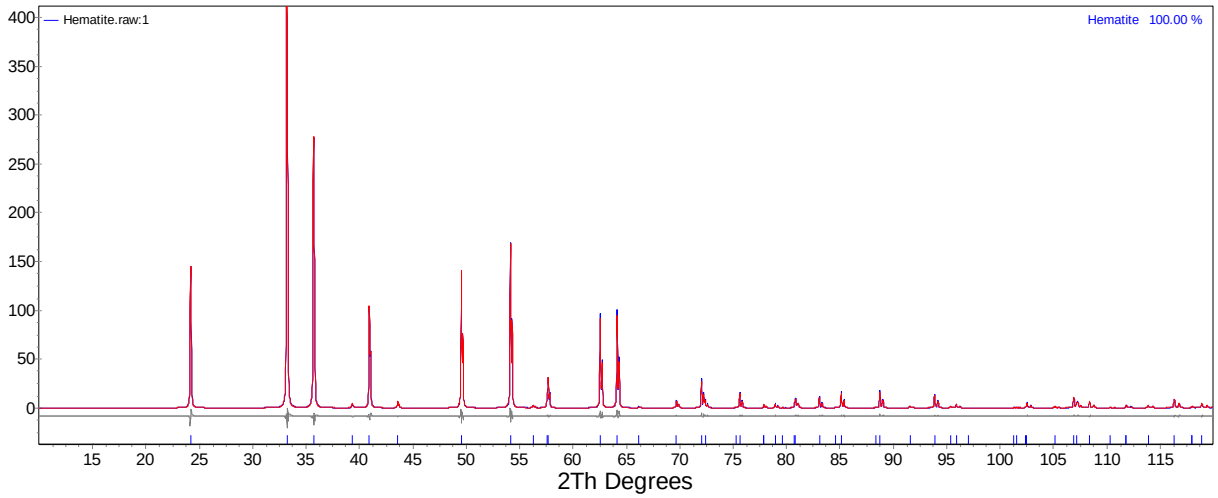

Fig.S18. Graphical representation of Rietveld refinement results of Hematite (model, ICSD-415251) (the refined Bragg peak positions are shown by vertical bars).

Table S14. Results of Rietveld refinement of Hematite (model, ICSD-415251).

Parameters	Angular range at Rietveld refinement, 2θ°					
	10-60	10-70	10-80	10-90	10-100	10-120
R_{wp}	5.30	6.26	6.38	6.62	6.7	7.44
a=*b*, Å e.s.d.	5.027477 0.000035	5.026995 0.000030	5.026874 0.000025	5.026742 0.000021	5.026694 0.000019	5.026530 0.000015
c, Å e.s.d.	13.738871 0.000097	13.73847 0.00010	13.737782 0.000079	13.737255 0.000068	13.737038 0.000059	13.736544 0.000049
d, nm e.s.d.	173.3 4.1	141.0 2.5	144.8 2.2	139.2 1.7	136.7 1.6	131.1 1.5

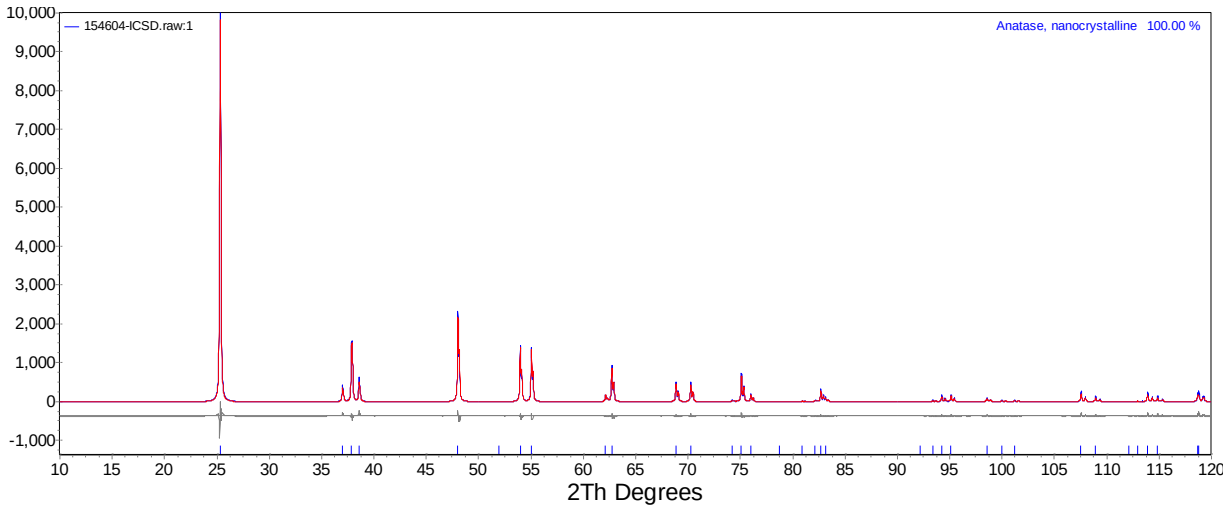

Fig.S19. Graphical representation of Rietveld refinement results of Anatase (model, ICSD-154604) (the refined Bragg peak positions are shown by vertical bars).

Table S15. Results of Rietveld refinement of Anatase (model, ICSD-154604).

Parameters	Angular range at Rietveld refinement, 2θ°						
	10-60	10-70	10-80	10-90	10-100	10-110	10-120
R_{wp}	7.35	8.22	8.53	8.58	9.13	9.86	10.93
a=*b*, Å	3.787397	3.786945	3.786943	3.786856	3.786713	3.786490	3.786525
e.s.d.	0.000059	0.000050	0.000038	0.000034	0.000031	0.000023	0.000023
c, Å	9.49860	9.49752	9.49754	9.497391	9.4969973	9.496434	9.496552
e.s.d.	0.00016	0.00012	0.00010	0.000093	0.000084	0.000070	0.000063
d, nm	103.0	105.3	108.3	109.1	108.6	111.2	113.5
e.s.d.	1.5	1.4	1.4	1.2	1.1	1.2	1.2

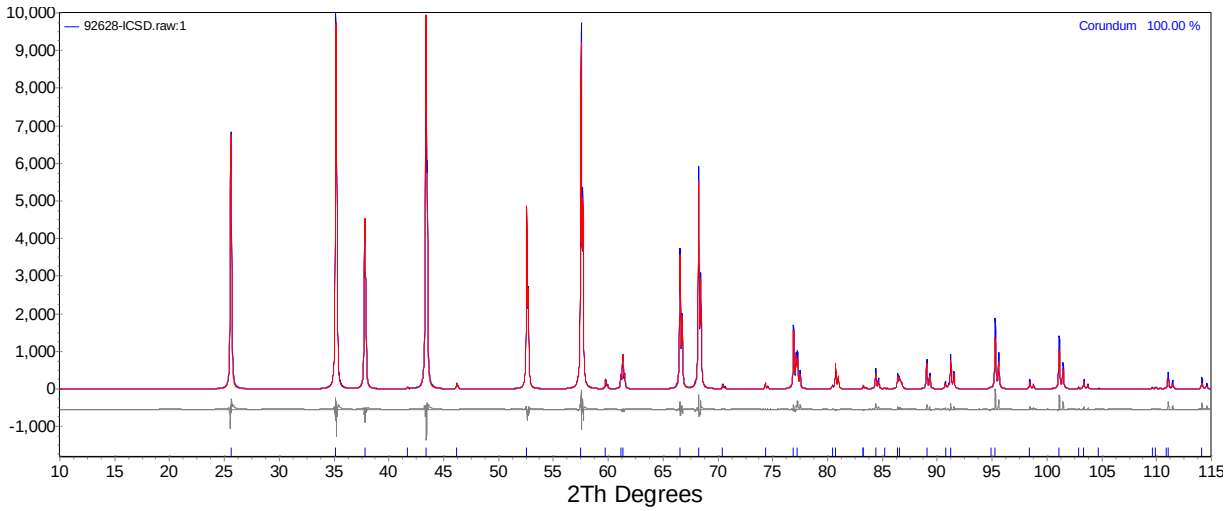

Fig.S20. Graphical representation of Rietveld refinement results of Corundum (model, ICSD-92628) (the refined Bragg peak positions are shown by vertical bars)

Table S16. Results of Rietveld refinement of Corundum (model, ICSD-92628).

Parameters	Angular range at Rietveld refinement, 2θ°					
	10-65	10-70	10-80	10-90	10-100	10-115
R_{wp}	11.38	11.50	11.68	12.36	13.09	13.56
a=*b*, Å	4.758445	4.757920	4.757845	4.757770	4.757639	4.757590
e.s.d.	0.000086	0.000052	0.000049	0.000044	0.000039	0.000035
c, Å	12.99096	12.99091	12.99005	12.98970	12.98942	12.98914
e.s.d.	0.000021	0.00021	0.00015	0.00014	0.00013	0.00011
d, nm	147.4	143.7	150.7	137.4	141.9	143.6
e.s.d.	4.2	3.7	2.7	1.9	1.6	1.7

5. Example of the application of One-way ANOVA with post-hoc Tukey HSD Test Calculator for Rietveld refinement results obtained at estimation of the reproducibility ($BaTiO_3$ sample, *a* unit cell parameter, seven angular intervals, six repeated measurements) (http://astatsa.com/OneWay_Anova_with_TukeyHSD/_result/)

Your input data on k=7 independent treatments:

Treatment →	A	B	C	D	E	F	G
Input Data →	3.99273	3.99256	3.99221	3.99202	3.99206	3.99186	3.99201
	3.99138	3.99134	3.99132	3.99147	3.99148	3.99144	3.991607
	3.99107	3.99098	3.99103	3.99121	3.99122	3.99110	3.991443
	3.99132	3.99186	3.99139	3.99149	3.99146	3.99123	3.991507
	3.99187	3.99187	3.9916	3.99159	3.99156	3.99151	3.991749
	3.9912	3.99157	3.99162	3.99177	3.99167	3.99169	3.991782

Descriptive statistics of your k=7 independent treatments:

Treatment→	A	B	C	D	E	F	G	Pooled Total
observations N	6	6	6	6	6	6	6	42
$\sum xi$	23.9496	23.9502	23.9492	23.9495	23.9494	23.9488	23.9501	167.6468
mean $\bar{x}$	3.9916	3.9917	3.9915	3.9916	3.9916	3.9915	3.9917	3.9916
$\sum x^2_i$	95.5970	95.6019	95.5938	95.5968	95.5960	95.5911	95.6012	669.1778
Sample variance s^2	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
sample std. dev. *s*	0.0006	0.0005	0.0004	0.0003	0.0003	0.0003	0.0002	0.0004
std. dev. of mean $SE_{\bar{x}}$	0.0003	0.0002	0.0002	0.0001	0.0001	0.0001	0.0001	0.0001

One-way ANOVA of your k=7 independent treatments:

Source	Sum of squares SS	Degrees of freedom *v*	Mean square MS	F statistic	p-value
Treatment	0.0000	6	0.0000	0.2398	0.9602
Error	0.0000	35	0.0000		
Total	0.0000	41			

Conclusion from Anova:

The p-value corresponing to the F-statistic of one-way ANOVA is higher than 0.05, suggesting that the treatments are not significantly different for that level of significance. The Tukey HSD test, as well as other multiple comparison tests like Scheffe or Bonferroni, might not narrow down which of the pairs of treatments are significantly different. Even though your data does not suggest the presence of significatly different treatment pairs in one-way ANOVA, we proceed witht he multiple conparison tests. In some instances, a Bonferroni test of a small set of pairs might show significance, even though 1-way ANOVA suggests that there is too much noise and randomness in your data.

Tukey HSD results:

treatments pair	Tukey HSD Q statistic	Tukey HSD p-value	Tukey HSD inferfence
A vs B	0.6250	0.8999947	insignificant
A vs C	0.4098	0.8999947	insignificant
A vs D	0.0205	0.8999947	insignificant
A vs E	0.1229	0.8999947	insignificant
A vs F	0.7582	0.8999947	insignificant
A vs G	0.5410	0.8999947	insignificant
B vs C	1.0348	0.8999947	insignificant
B vs D	0.6455	0.8999947	insignificant
B vs E	0.7479	0.8999947	insignificant
B vs F	1.3831	0.8999947	insignificant
B vs G	0.0840	0.8999947	insignificant
C vs D	0.3893	0.8999947	insignificant
C vs E	0.2869	0.8999947	insignificant
C vs F	0.3483	0.8999947	insignificant
C vs G	0.9508	0.8999947	insignificant
D vs E	0.1025	0.8999947	insignificant
D vs F	0.7377	0.8999947	insignificant
D vs G	0.5615	0.8999947	insignificant
E vs F	0.6352	0.8999947	insignificant
E vs G	0.6639	0.8999947	insignificant
F vs G	1.2991	0.8999947	insignificant

Scheffé multiple comparison:

Treatments pair	Scheffé *T*-statistic	Scheffé p-value	Scheffé inferfence
A vs B	0.4419	0.9998322	insignificant
A vs C	0.2898	0.9999860	insignificant
A vs D	0.0145	1.0000000	insignificant
A vs E	0.0869	1.0000000	insignificant
A vs F	0.5361	0.9994860	insignificant
A vs G	0.3825	0.9999279	insignificant
B vs C	0.7317	0.9970132	insignificant
B vs D	0.4564	0.9997975	insignificant
B vs E	0.5289	0.9995247	insignificant
B vs F	0.9780	0.9857432	insignificant
B vs G	0.0594	1.0000000	insignificant
C vs D	0.2753	0.9999897	insignificant
C vs E	0.2029	0.9999983	insignificant
C vs F	0.2463	0.9999947	insignificant
C vs G	0.6723	0.9981376	insignificant
D vs E	0.0724	1.0000000	insignificant
D vs F	0.5216	0.9995610	insignificant
D vs G	0.3970	0.9999103	insignificant
E vs F	0.4492	0.9998155	insignificant
E vs G	0.4695	0.9997614	insignificant
F vs G	0.9186	0.9897379	insignificant

Bonferroni and Holm results: only pairs relative to A simultaineously compared:

Treatments pair	Bonferroni and Holm *T*-statistic	Bonferroni p-value	Bonferroni inferfence	Holm p-value	Holm inferfence
A vs B	0.4419	3.9675739	insignificant	3.3063116	insignificant
A vs C	0.2898	4.6421358	insignificant	2.3210679	insignificant
A vs D	0.0145	5.9311312	insignificant	0.9885219	insignificant
A vs E	0.0869	5.5873071	insignificant	1.8624357	insignificant
A vs F	0.5361	3.5716606	insignificant	3.5716606	insignificant
A vs G	0.3825	4.2263398	insignificant	2.8175599	insignificant