Supporting information

Microwave-assisted ultra-stable to oxidation Fe_xNi_{1-x} nanoclusters in aqueous media

Antonio Santana-Otero[1,2], María Eugenia Fortes Brollo[3], María del Puerto Morales[4], Daniel Ortega[1,2,5]*

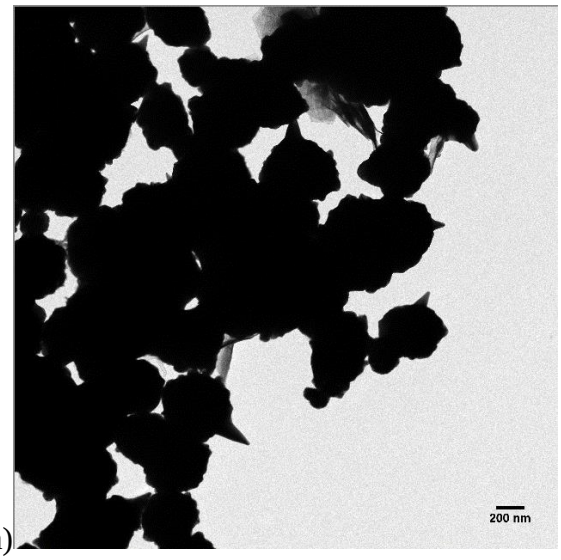
[1] Condensed Matter Physics department, Faculty of Sciences, Campus Universitario de Puerto Real, 11510 Puerto Real (Cádiz) Spain

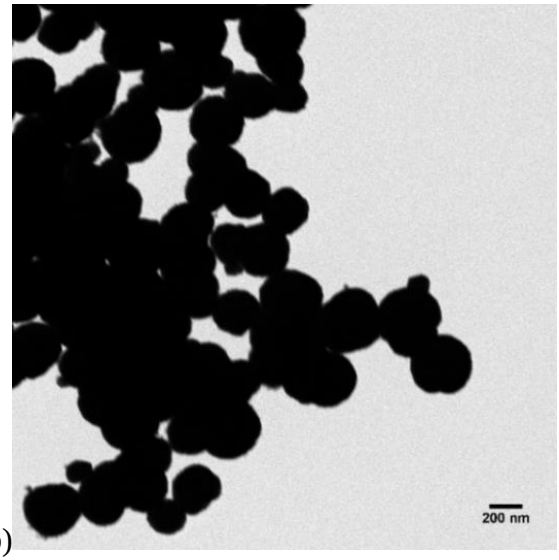
[2] Institute of Research and Innovation in Biomedical Sciences of Cádiz (INiBICA), University of Cádiz, 11009 Cádiz, Spain

[3] Department of Physics, Chalmers University, Fysikgränd 3, 412 96 Gothenburg, Sweden

[4] Institute of Materials Science of Madrid (ICMM-CSIC), Sor Juana Inés de la Cruz 3, 28049 Madrid, Spain

[5] IMDEA Nanoscience, Faraday 9, 28049 Madrid, Spain

a) 

b) 

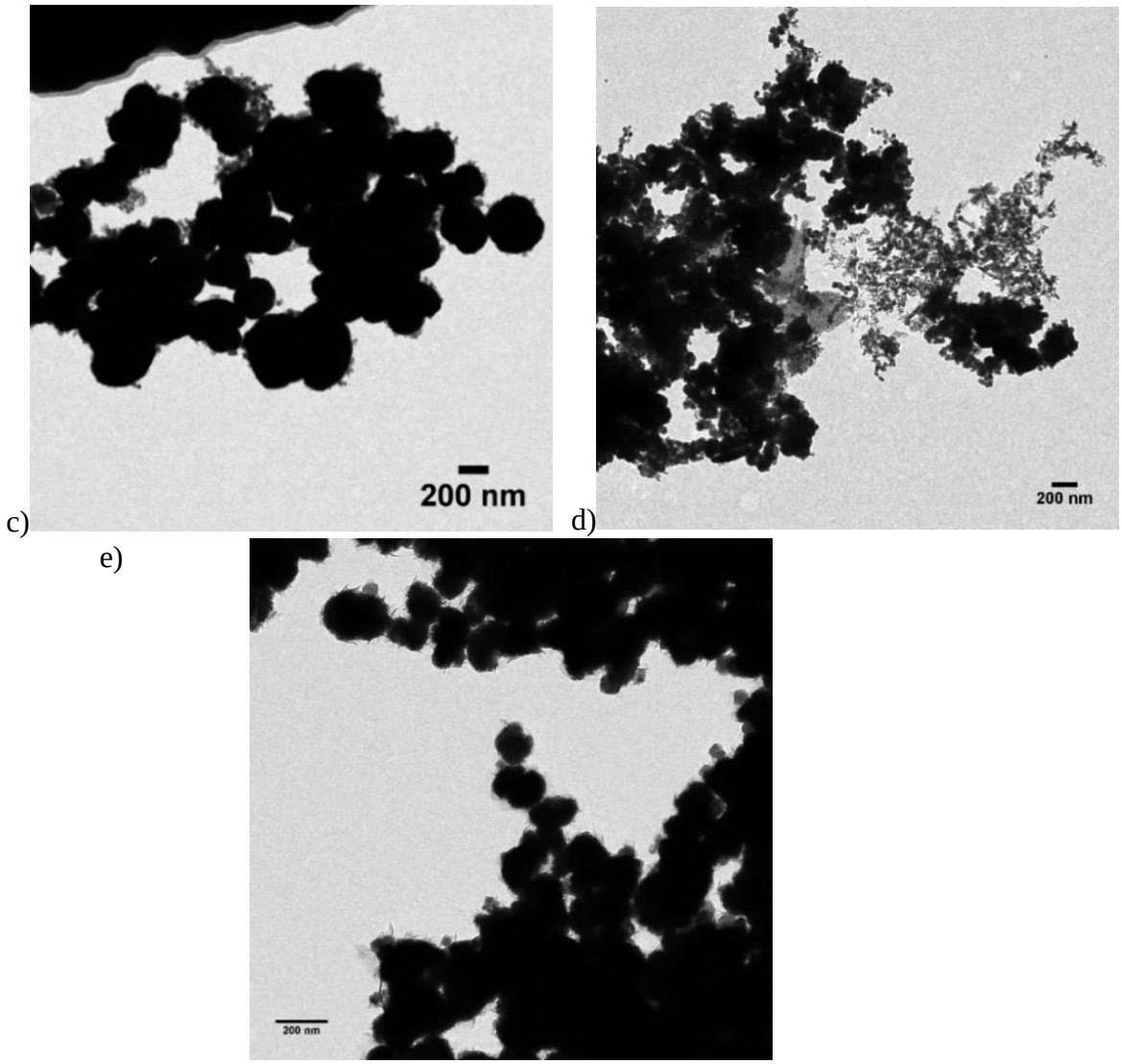


Figure S1 TEM images from FNNPS with (a) 0%, (b) 15%, (c) 25%, (d) 35%, and (e) 100% of Fe.

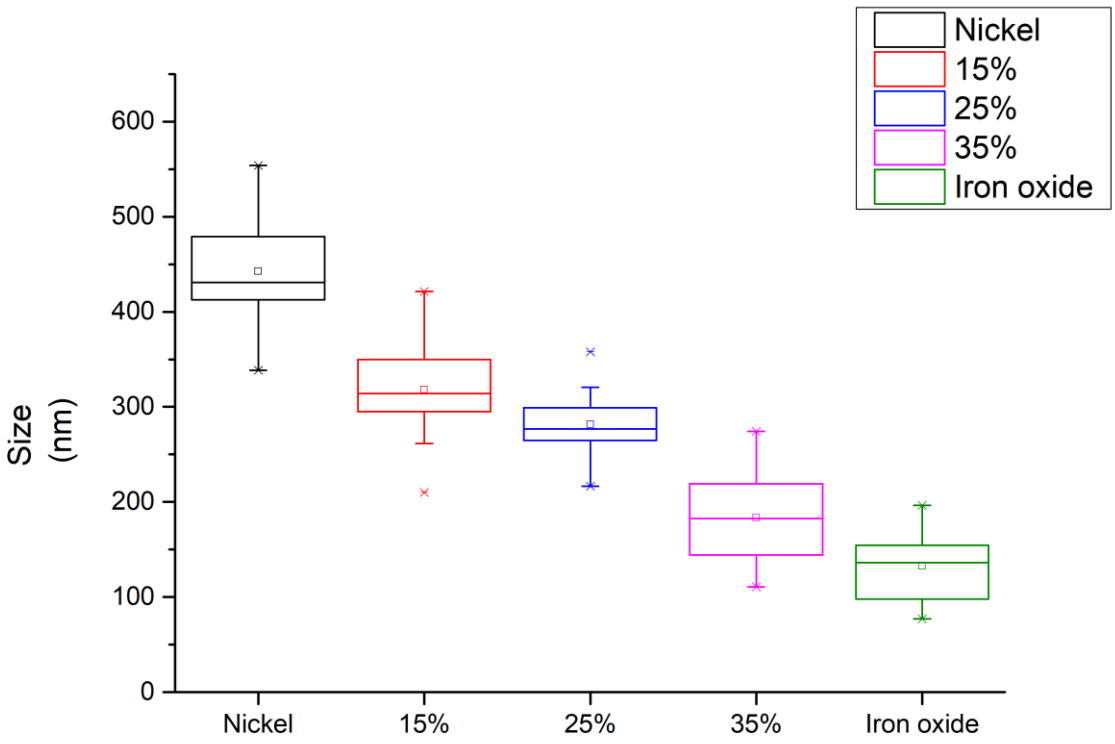


Figure S2 Size distribution of aggregates of FNNPS from TEM images.

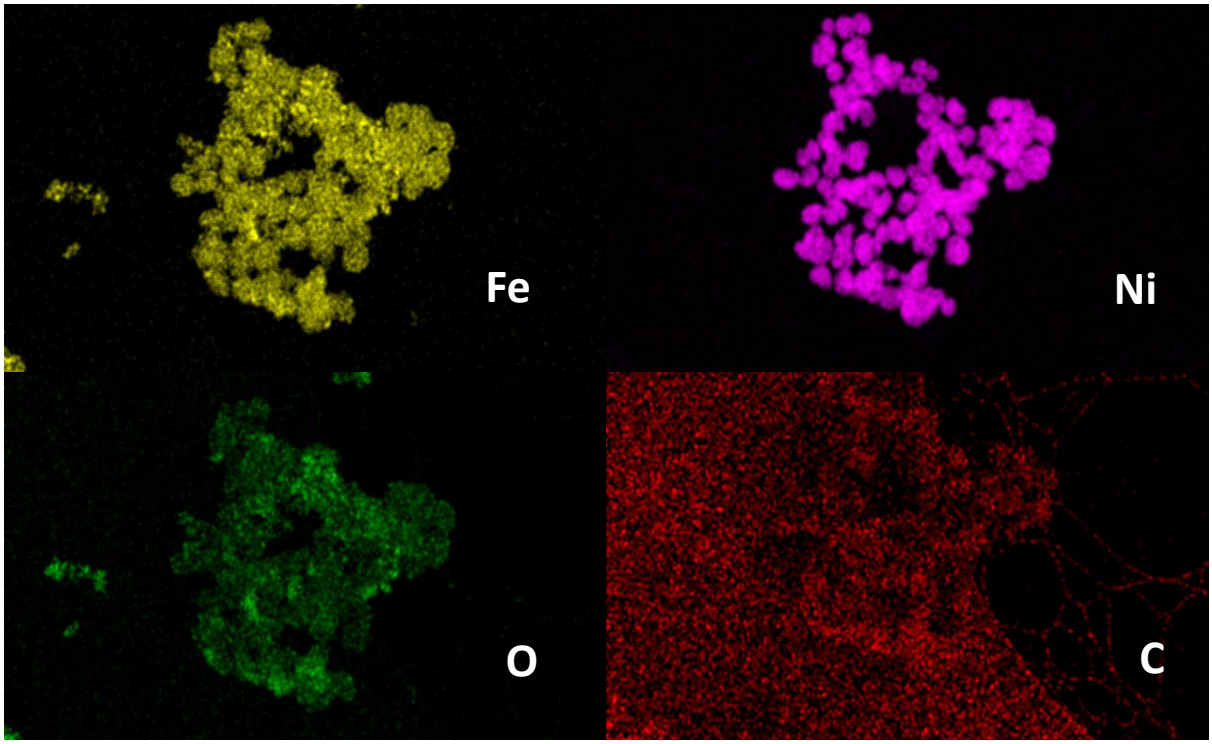


Figure S3 Individual Fe, Ni, C and O EDX chemical maps for sample $Fe_{25}Ni_{75}$.

Table S1 Parameters from the Rietveld analysis of Fe-Ni phases present in the studied sample set.

FeNi 0%																				
No.	Pos. [°2Th.]	FWHM Left [°2Th.]	Struct. B [°2Th.]	Area calc.	Assignment	h	k	l	Multiplicity	F obs.	F calc.	Area [cts*°2Th.]	Derivation	Backgr.[cts]	d-spacing [Å]	Height [cts]	Crystallite Size [Å]	Micro Strain [%]	K-A2 / K-A1 Ratio	Rel. Int. [%]	Integral Breadth [°2Th.]
1	44,45036	0,287227	0,2917	6649,555	global	1	1	1	8	68,19948	68,16285	6656,704	Pure K-Alpha1	1152,62	2,03649	18818,51	307,2775852	0,2478	0,5	100	0,353732
2	51,80224	0,382203	0,3932	2970,211	global	0	0	2	6	61,17652	62,57125	2839,273	Pure K-Alpha1	1135,25	1,76343	6277,566	234,5855301	0,3047	0,5	33,36	0,452289
3	76,32858	0,53272	0,5816	1443,931	global	0	2	2	12	46,50267	46,66282	1434,037	Pure K-Alpha1	1084,28	1,2466	2218,572	181,4508458	0,252	0,5	11,79	0,646378
4	92,87974	0,509126	0,5127	1573,037	global	1	1	3	24	37,4837	38,79097	1468,799	Pure K-Alpha1	1055,97	1,06299	2625,606	234,846711	0,2127	0,5	13,95	0,559413
5	98,38263	0,459016	0,4772	461,2541	global	2	2	2	8	35,3103	36,66243	427,8589	Pure K-Alpha1	1047,64	1,01771	868,708	266,0543111	0,1798	0,5	4,62	0,492523
FeNi 5%																				
No.	Pos. [°2Th.]	FWHM Left [°2Th.]	Struct. B [°2Th.]	Area calc.	Assignment	h	k	l	Multiplicity	F obs.	F calc.	Area [cts*°2Th.]	Derivation	Backgr.[cts]	d-spacing [Å]	Height [cts]	Crystallite Size [Å]	Micro Strain [%]	K-A2 / K-A1 Ratio	Rel. Int. [%]	Integral Breadth [°2Th.]
1	44,26837	0,361525	0,4396	5808,327	global	1	1	1	8	83,41291	84,76206	5624,897	Pure K-Alpha1	2530,55	2,04444	11175,26	203,76446	0,2217	0,5	100	0,503335
2	51,61706	0,498599	0,631	2238,045	global	0	0	2	6	71,95543	72,26607	2218,845	Pure K-Alpha1	2503,03	1,76932	3214,092	146,0646989	0,2686	0,5	28,76	0,690349
3	76,13093	0,772071	0,8851	962,3884	global	0	2	2	12	50,87789	50,68994	969,5382	Pure K-Alpha1	2391,22	1,24935	1022,375	119,0702827	0,3599	0,5	9,15	0,94832
4	92,67106	0,868612	0,918	955,4138	global	1	1	3	24	39,78119	40,23599	933,9374	Pure K-Alpha1	2298,37	1,06484	1013,746	130,9106456	0,3823	0,5	9,07	0,921273
5	98,16953	0,882453	0,905	290,123	global	2	2	2	8	38,50003	38,70378	287,0764	Pure K-Alpha1	2264,39	1,01935	315,9247	139,9872698	0,3422	0,5	2,83	0,908686
FeNi 10%																				
No.	Pos. [°2Th.]	FWHM Left [°2Th.]	Struct. B [°2Th.]	Area calc.	Assignment	h	k	l	Multiplicity	F obs.	F calc.	Area [cts*°2Th.]	Derivation	Backgr.[cts]	d-spacing [Å]	Height [cts]	Crystallite Size [Å]	Micro Strain [%]	K-A2 / K-A1 Ratio	Rel. Int. [%]	Integral Breadth [°2Th.]
1	44,19223	0,446111	0,5636	2111,82	global	1	1	1	8	51,76937	51,74858	2113,518	Pure K-Alpha1	1059,93	2,04779	3383,493	158,8904788	0,2717	0,5	100	0,624656
2	51,50977	0,597294	0,7825	634,3611	global	0	0	2	6	39,05611	38,94989	637,8257	Pure K-Alpha1	1089,85	1,77275	759,1015	117,7318238	0,3038	0,5	22,44	0,840238
3	75,90233	0,9852	1,3193	333,0883	global	0	2	2	12	30,16128	30,20999	332,0149	Pure K-Alpha1	1061,15	1,25254	241,6497	79,75814968	0,3248	0,5	7,14	1,373951
4	92,33542	1,229217	1,6634	304,3025	global	1	1	3	24	23,08347	23,05928	304,9413	Pure K-Alpha1	1041,32	1,06783	176,9411	72,02645918	0,2972	0,5	5,23	1,723406
5	97,79104	1,314106	1,7775	114,8506	global	2	2	2	8	24,77107	24,75823	114,9697	Pure K-Alpha1	1018,1	1,02228	62,4675	71,00320171	0,2901	0,5	1,85	1,840473
FeNi 15%																				
No.	Pos. [°2Th.]	FWHM Left [°2Th.]	Struct. B [°2Th.]	Area calc.	Assignment	h	k	l	Multiplicity	F obs.	F calc.	Area [cts*°2Th.]	Derivation	Backgr.[cts]	d-spacing [Å]	Height [cts]	Crystallite Size [Å]	Micro Strain [%]	K-A2 / K-A1 Ratio	Rel. Int. [%]	Integral Breadth [°2Th.]
1	44,1241	0,500892	0,6742	4762,546	global	1	1	1	8	23,33747	23,33407	4763,936	Pure K-Alpha1	1101,73	2,05079	6349,621	132,7930581	0,1936	0,5	100	0,750271
2	51,45003	0,688987	0,9598	1745,11	global	0	0	2	6	19,46468	19,39838	1757,06	Pure K-Alpha1	1086,9	1,77496	1691,471	95,95957456	0,213	0,5	26,64	1,038777
3	75,87524	1,160248	1,6513	1075,603	global	0	2	2	12	16,2584	16,2981	1070,369	Pure K-Alpha1	1040,21	1,25292	619,0898	63,71073244	0,2569	0,5	9,75	1,72894
4	92,33709	1,454044	2,0881	1208,904	global	1	1	3	24	13,80895	13,78948	1212,319	Pure K-Alpha1	1011,14	1,06781	555,2221	57,37782239	0,2231	0,5	8,74	2,183485
5	97,80422	1,556102	2,2273	430,5292	global	2	2	2	8	14,34839	14,37722	428,8041	Pure K-Alpha1	1001,92	1,02218	184,7639	56,67167943	0,2329	0,5	2,91	2,320822
FeNi 25%																				
No.	Pos. [°2Th.]	FWHM Left [°2Th.]	Struct. B [°2Th.]	Area calc.	Assignment	h	k	l	Multiplicity	F obs.	F calc.	Area [cts*°2Th.]	Derivation	Backgr.[cts]	d-spacing [Å]	Height [cts]	Crystallite Size [Å]	Micro Strain [%]	K-A2 / K-A1 Ratio	Rel. Int. [%]	Integral Breadth [°2Th.]
1	44,01104	1,046323	1,3384	1457,874	global	1	1	1	8	66,24718	66,1084	1464,001	Pure K-Alpha1	1062,09	2,0558	1055,792	66,86591274	0,7921	0,5	100	1,386639
2	51,32787	1,046323	1,3585	378,2289	global	0	0	2	6	46,66204	46,24244	385,1242	Pure K-Alpha1	1052,84	1,7786	273,9132	67,76206854	0,6394	0,5	25,94	1,406008
3	75,71768	1,046323	1,2847	271,7039	global	0	2	2	12	41,36942	41,95181	264,2125	Pure K-Alpha1	1052,63	1,25513	196,7678	81,80352557	0,4475	0,5	18,64	1,342763
4	92,14835	1,046323	1,2925	227,6511	global	1	1	3	24	30,41985	30,66784	223,9842	Pure K-Alpha1	1050,17	1,06951	164,8649	92,53817453	0,3251	0,5	15,62	1,358593
5	97,603	1,046323	1,2871	100,8209	global	2	2	2	8	35,37114	35,66938	99,142	Pure K-Alpha1	1042,93	1,02375	73,01448	97,87229995	0,2957	0,5	6,92	1,357841
FeNi 33%																				
No.	Pos. [°2Th.]	FWHM Left [°2Th.]	Struct. B [°2Th.]	Area calc.	Assignment	h	k	l	Multiplicity	F obs.	F calc.	Area [cts*°2Th.]	Derivation	Backgr.[cts]	d-spacing [Å]	Height [cts]	Crystallite Size [Å]	Micro Strain [%]	K-A2 / K-A1 Ratio	Rel. Int. [%]	Integral Breadth [°2Th.]
1	44,11382	1,504027	1,2999	1175,407	global	1	1	1	8	14,0954	14,09696	1175,147	Pure K-Alpha1	1113,48	2,05125	903(218)	68,87131711	1,3997	0,5	100	1,301273
2	51,41678	1,623471	1,1954	256,6882	global	0	0	2	6	9,059109	9,046399	257,41	Pure K-Alpha1	1104,75	1,77574	215(78)	77,03624675	1,0832	0,5	23,81	1,196922
3	75,75263	2,040321	0,8727	90,5262	global	0	2	2	12	5,682905	5,752055	88,3627	Pure K-Alpha1	1098,23	1,25464	101(45)	120,4513759	0,4895	0,5	11,18	0,875321
4	92,13585	2,367048	0,7775	49,1506	global	1	1	3	24	3,423882	3,388496	50,1825	Pure K-Alpha1	1065,88	1,06962	64(20)	153,816138	0,3268	0,5	7,11	0,781292
5	97,57156	2,490571	0,705	17,112	global	2	2	2	8	3,4416	3,496175	16,582	Pure K-Alpha1	1022,95	1,02399	23(5)	178,6269108	0,2694	0,5	2,59	0,709707
FeNi 35%																				
No.	Pos. [°2Th.]	FWHM Left [°2Th.]	Struct. B [°2Th.]	Area calc.	Assignment	h	k	l	Multiplicity	F obs.	F calc.	Area [cts*°2Th.]	Derivation	Backgr.[cts]	d-spacing [Å]	Height [cts]	Crystallite Size [Å]	Micro Strain [%]	K-A2 / K-A1 Ratio	Rel. Int. [%]	Integral Breadth [°2Th.]
1	44,142	1,514252	1,9592	1883,258	global	1	1	1	8	54,46395	54,4303	1885,588	Pure K-Alpha1	1102,92	2,05	942,4011	45,69964739	1,1543	0,5	100	2,000834
2	51,43948	1,514252	2,0092	270,6721	global	0	0	2	6	28,68529	28,33603	277,3856	Pure K-Alpha1	1089,17	1,77501	135,447	45,83810212	0,9066	0,5	14,37	2,047927
3	75,75399	1,514252	1,8877	282,6553	global	0	2	2	12	30,56051	31,00682	274,5768	Pure K-Alpha1	1066,3	1,25462	141,4435	55,68622457	0,6501	0,5	15,01	1,941248
4	92,1185	1,514252	1,91	180,6963	global	1	1	3	24	19,68677	19,82858	178,1209	Pure K-Alpha1	1060,48	1,06977	90,42222	62,60379925	0,4697	0,5	9,59	1,96988
5	97,54674	1,514252	1,9467	130,0345	global	2	2	2	8	29,48929	29,41954	130,6518	Pure K-Alpha1	1052,75	1,02419	65,07055	64,67398206	0,4056	0,5	6,9	2,007848

Table S2 Lattice parameter (in Å) of each of the phases present in FNNPS samples.

Name	Iron-Nickel phase	Iron oxide phase	Nickel phase
0% FNNPS	N/A	N/A	3,52442
5% FNNPS	3,52570	N/A	N/A
15% FNNPS	3,53500	N/A	N/A
25% FNNPS	3,54049	--*	N/A
33% FNNPS	3,54411	--*	N/A
35% FNNPS	3,54634	--*	N/A
50% FNNPS	3,55056	8,390079	N/A
70% FNNPS	3,55681	8,396987	N/A
90% FNNPS	N/A	8,394861	N/A
100% FNNPS (IONPS)	N/A	8,394224	N/A

** Some iron oxide peaks are not intense enough to be included in the Rietveld refinement.*