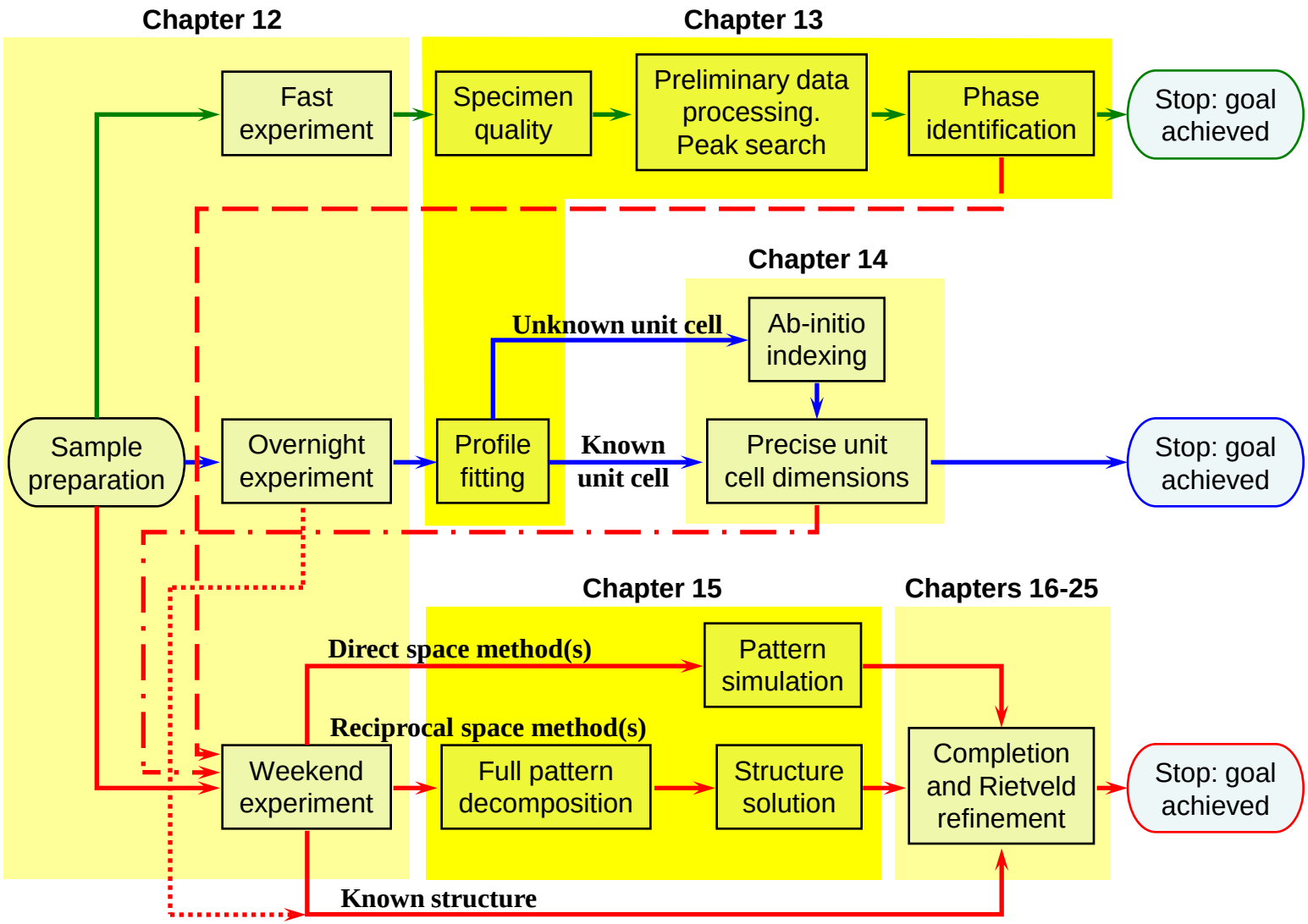
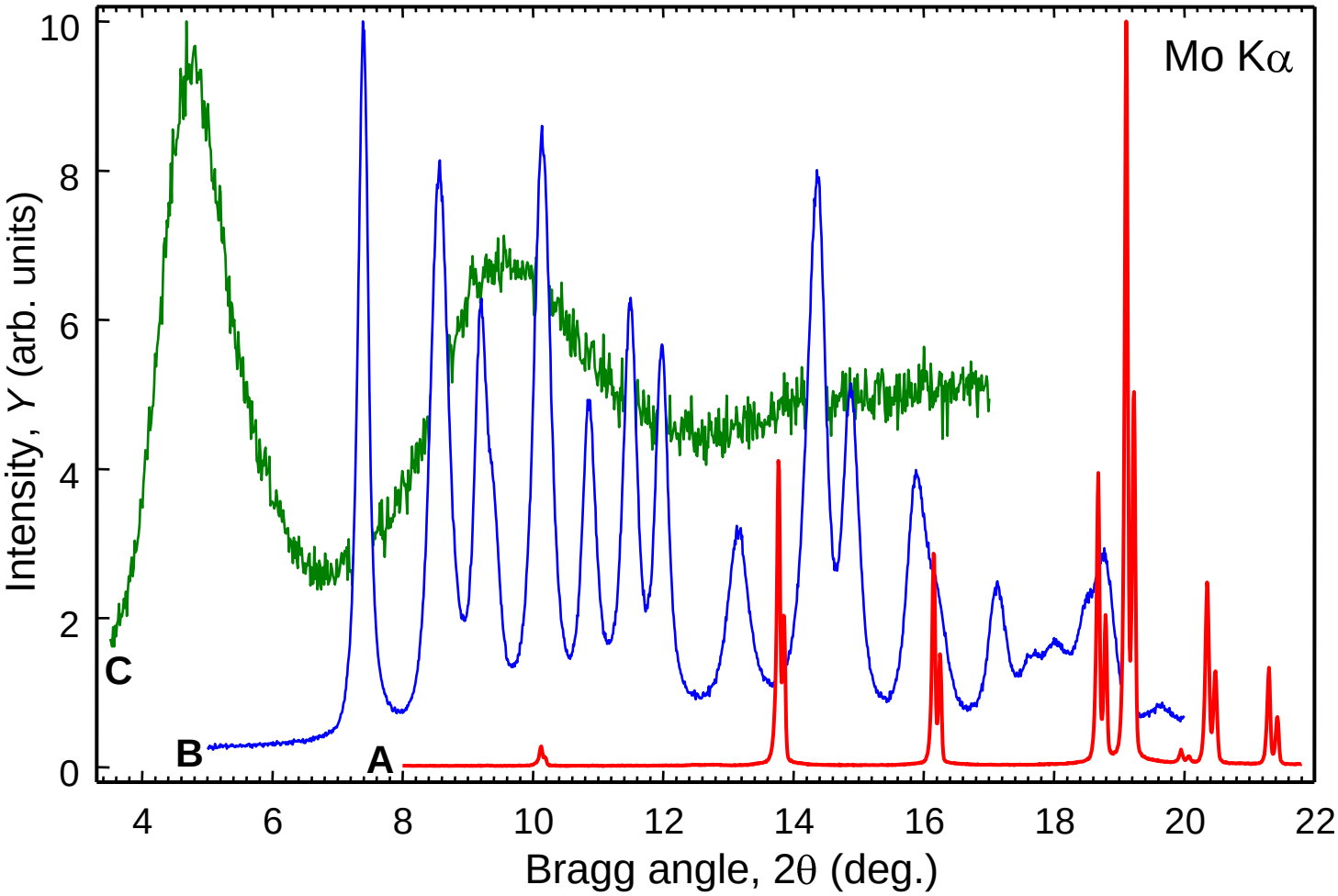
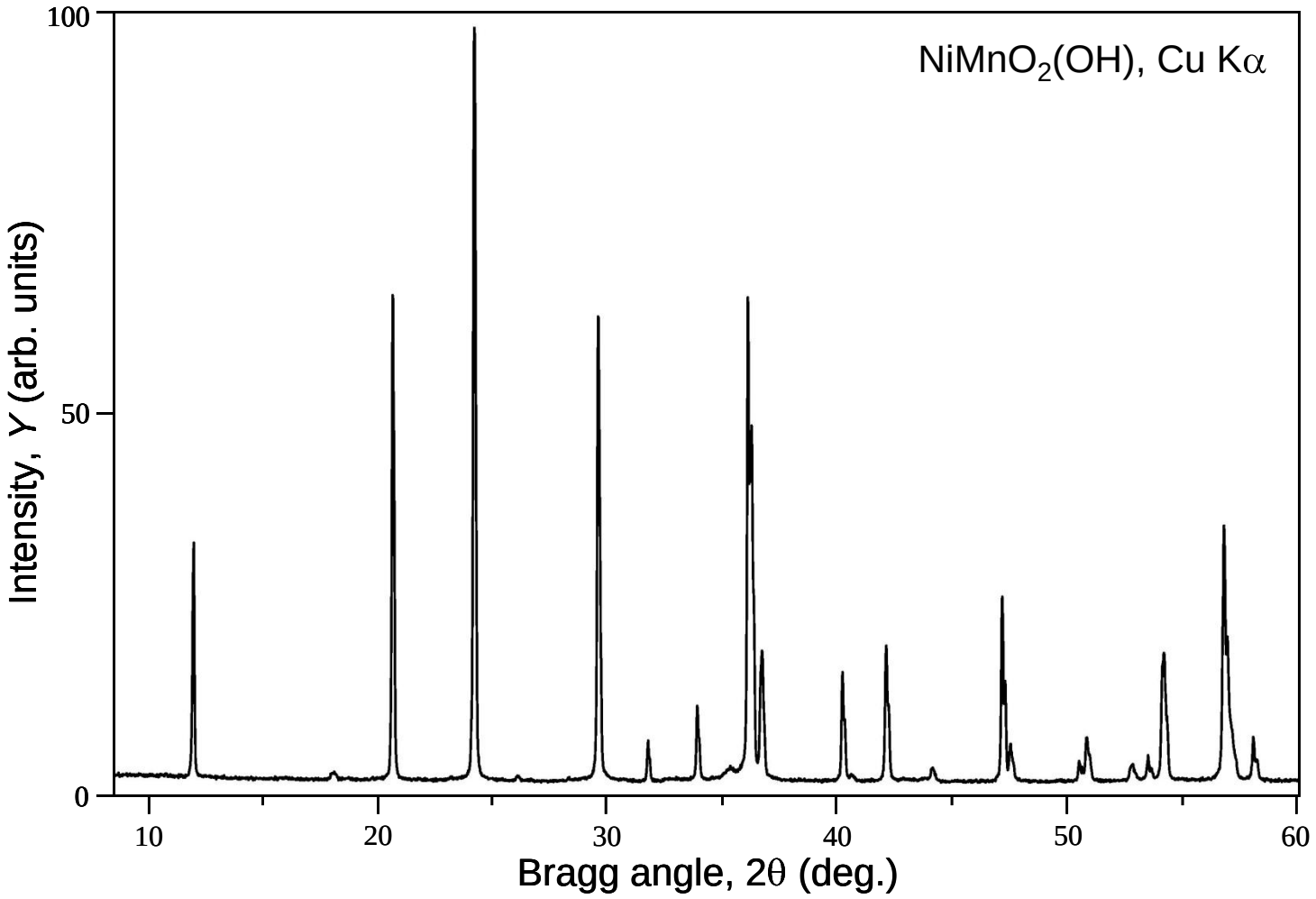
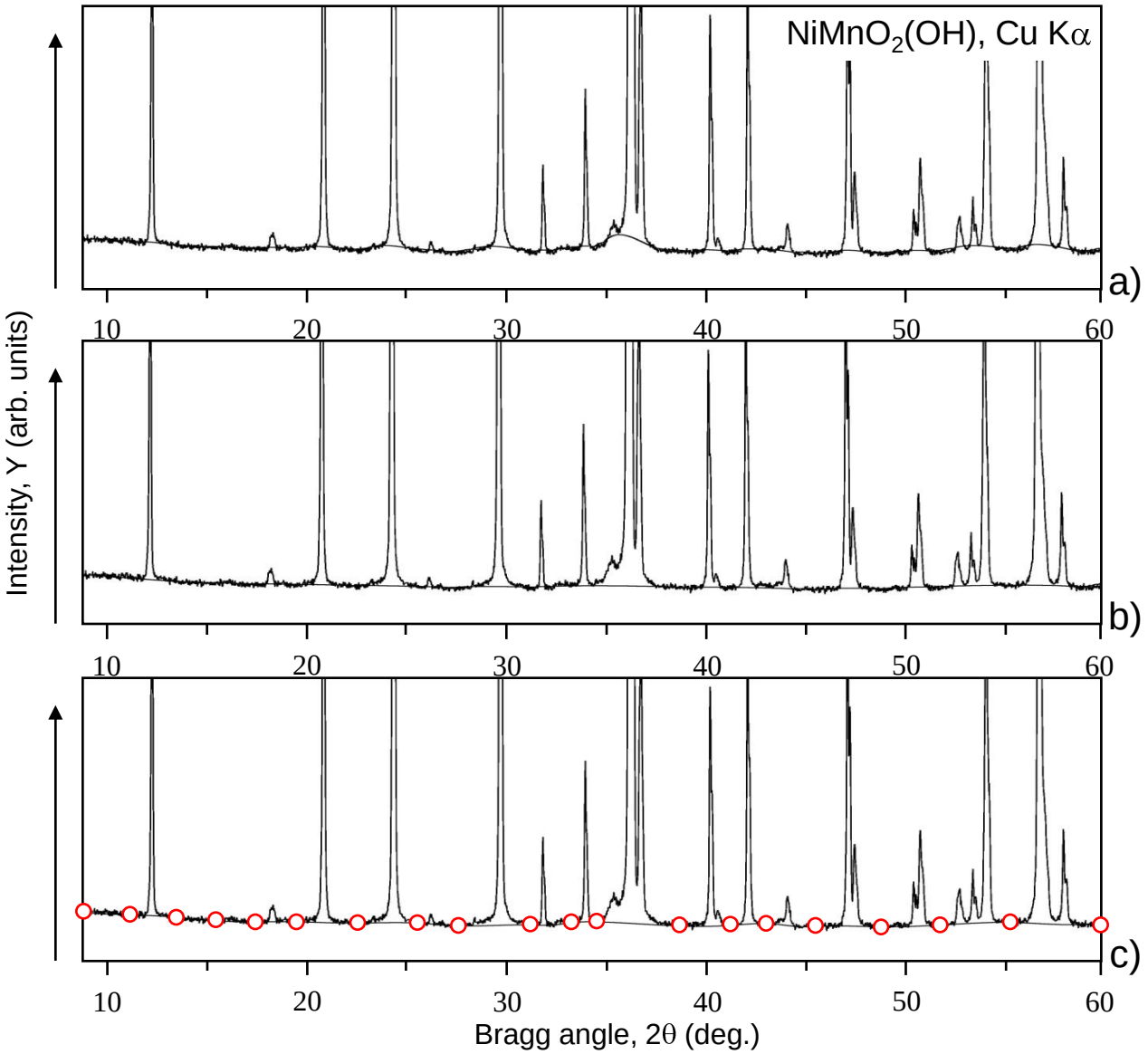
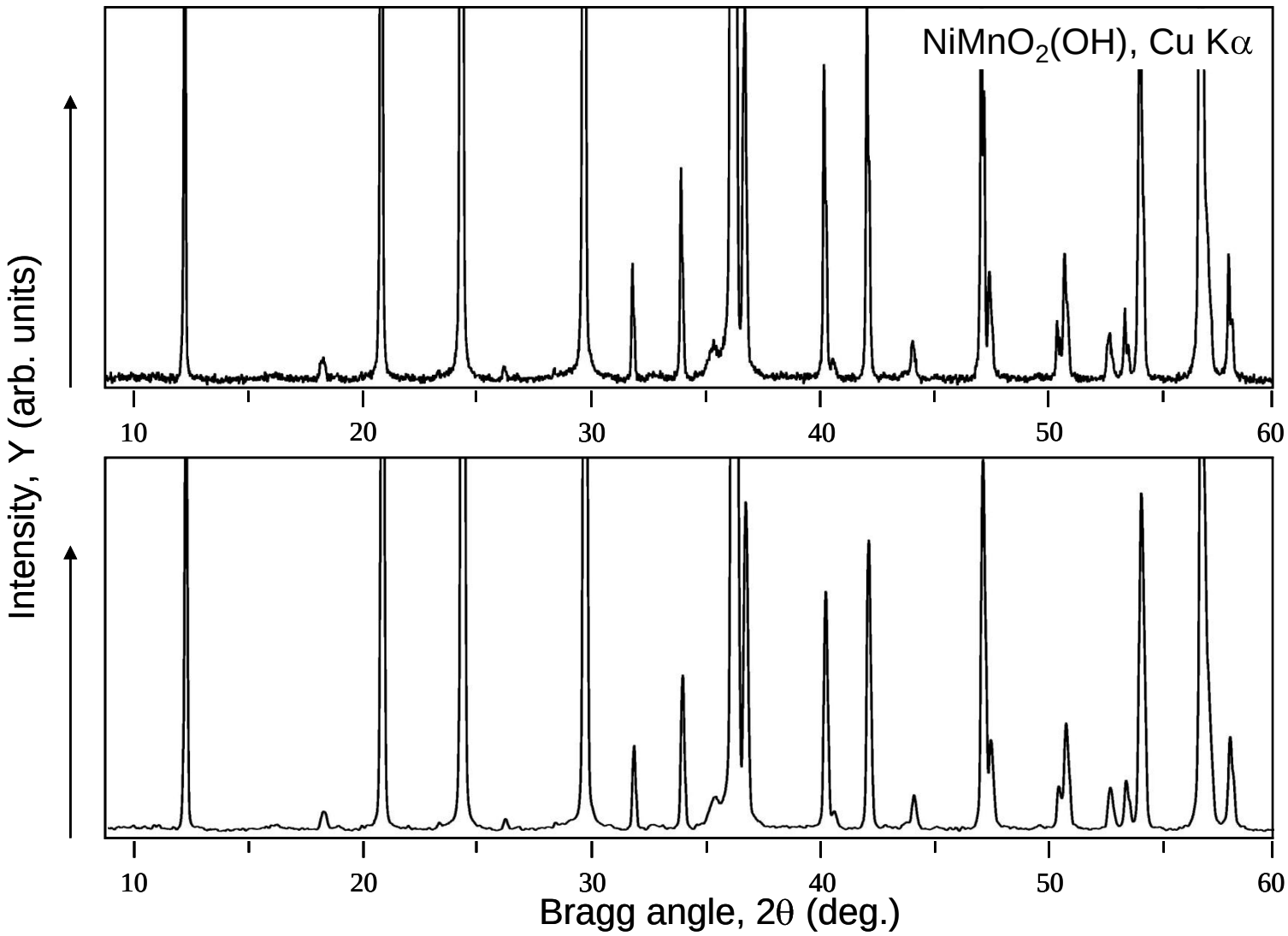
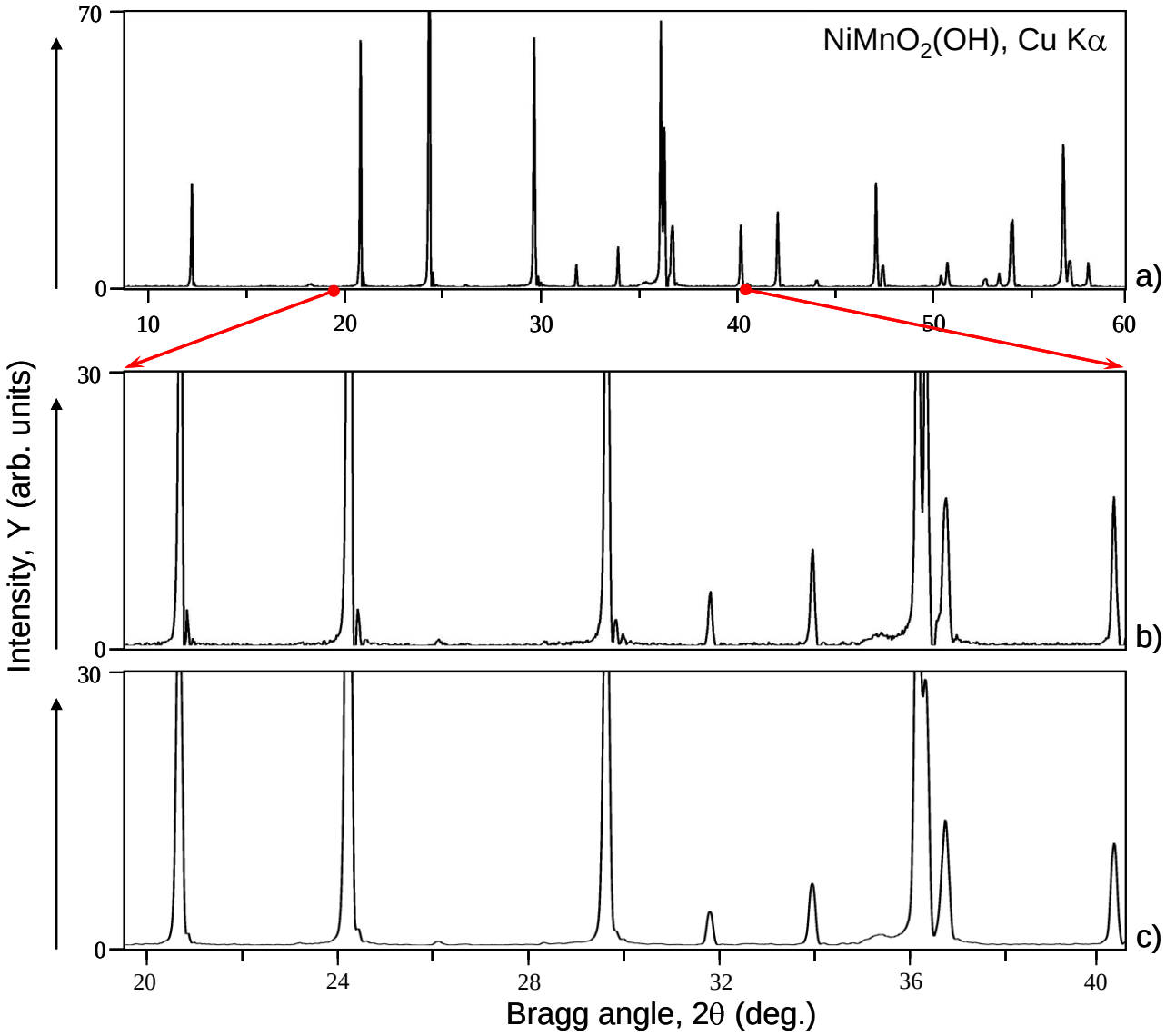

Chapter 12
Chapter 13
Fast experiment
Specimen quality
Preliminary data processing. Peak search
Phase identification
Stop: goal achieved
Chapter 14
Unknown unit cell
Ab-initio indexing
Sample preparation
Overnight experiment
Profile fitting
Known unit cell
Precise unit cell dimensions
Stop: goal achieved
Chapter 15
Chapters 16-25
Direct space method(s)
Pattern simulation
Reciprocal space method(s)
Weekend experiment
Full pattern decomposition
Structure solution
Completion and Rietveld refinement
Stop: goal achieved
Known structure

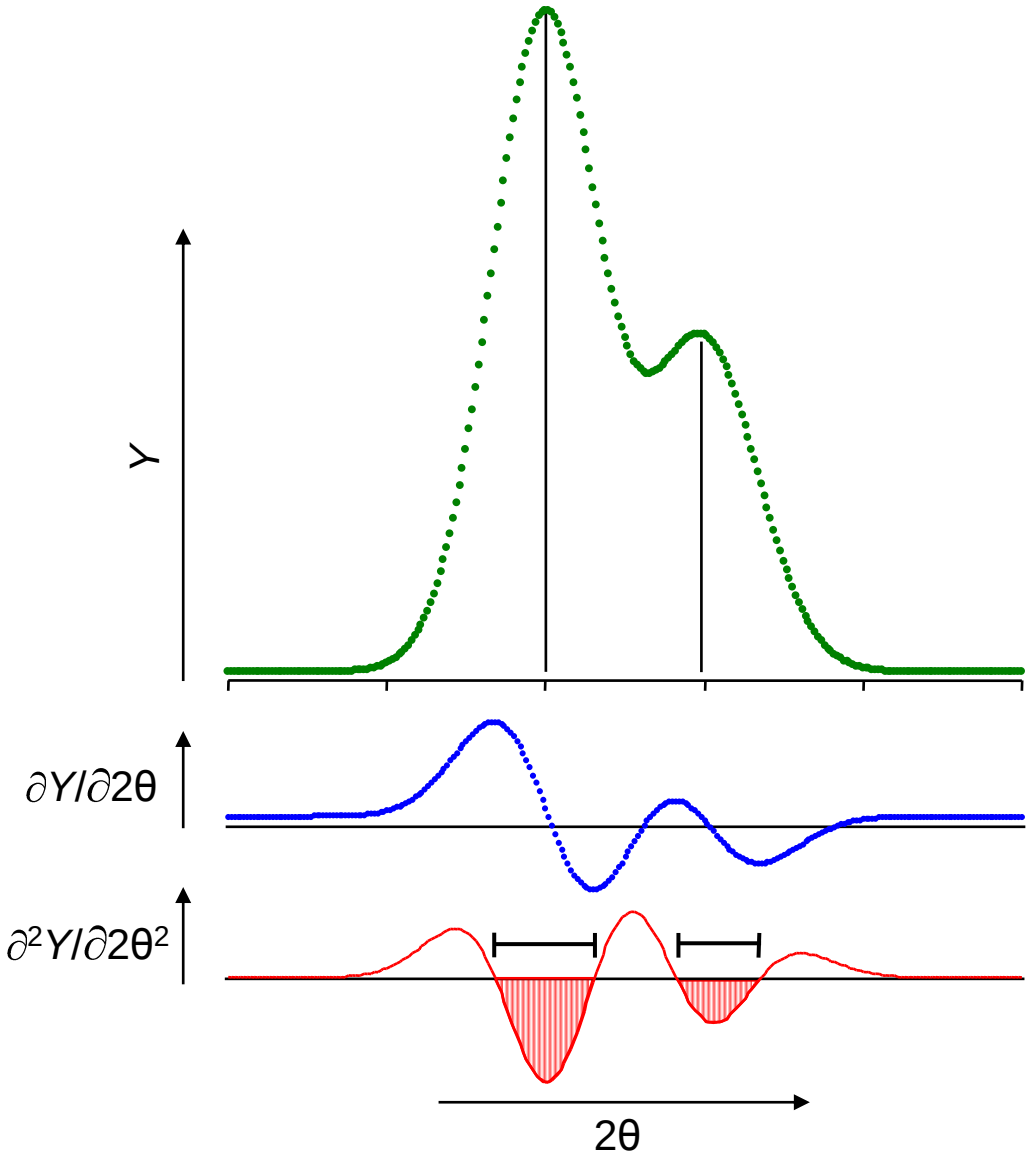

Mo Kα
Intensity, Y (arb. units)
Bragg angle, 2θ (deg.)
C
B
A
0
2
4
6
8
10
4
6
8
10
12
14
16
18
20
22

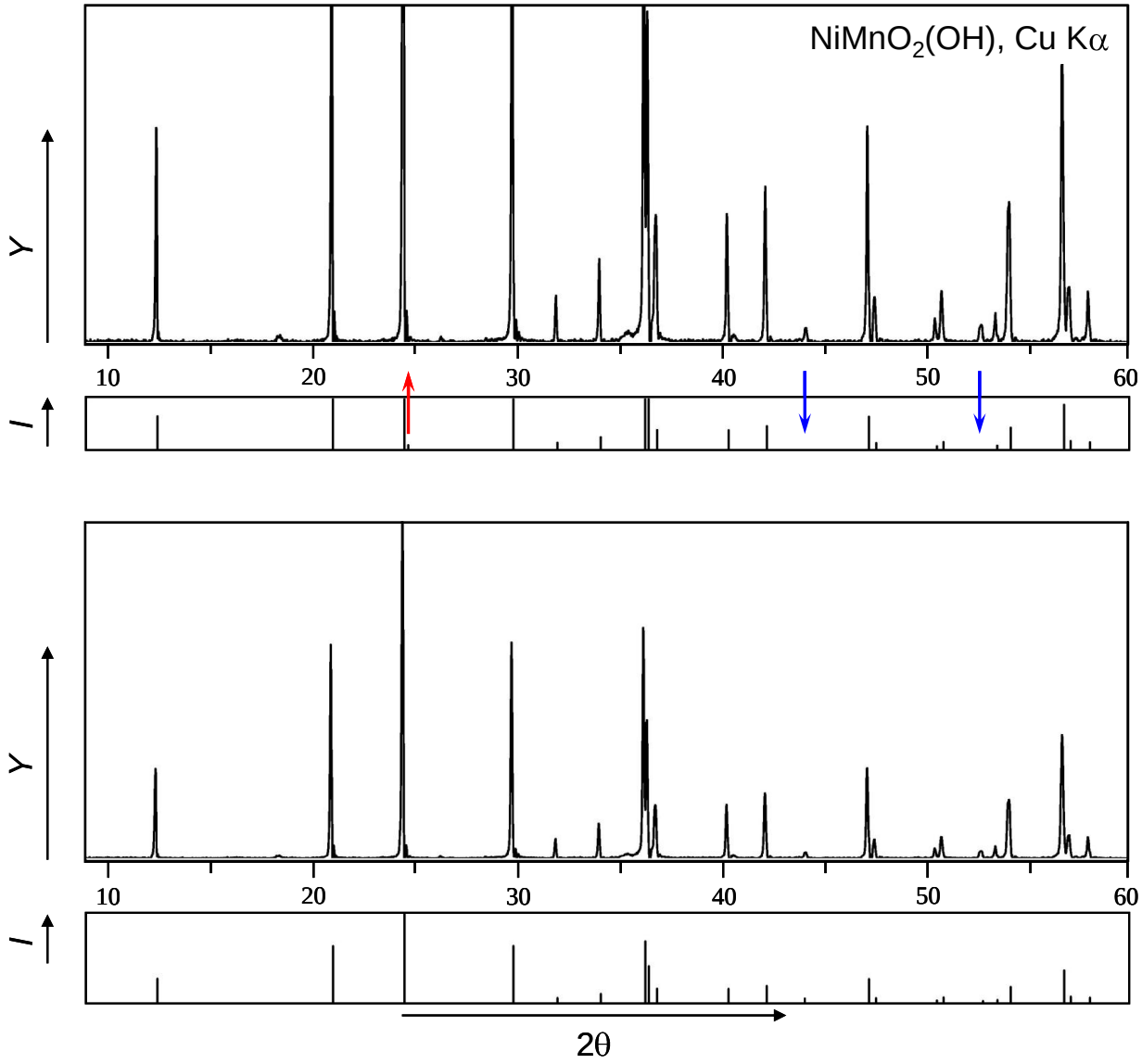

NiMnO2(OH), Cu Kα
Intensity, Y (arb. units)
100
50
0
10
20
30
40
50
60
Bragg angle, 2θ (deg.)

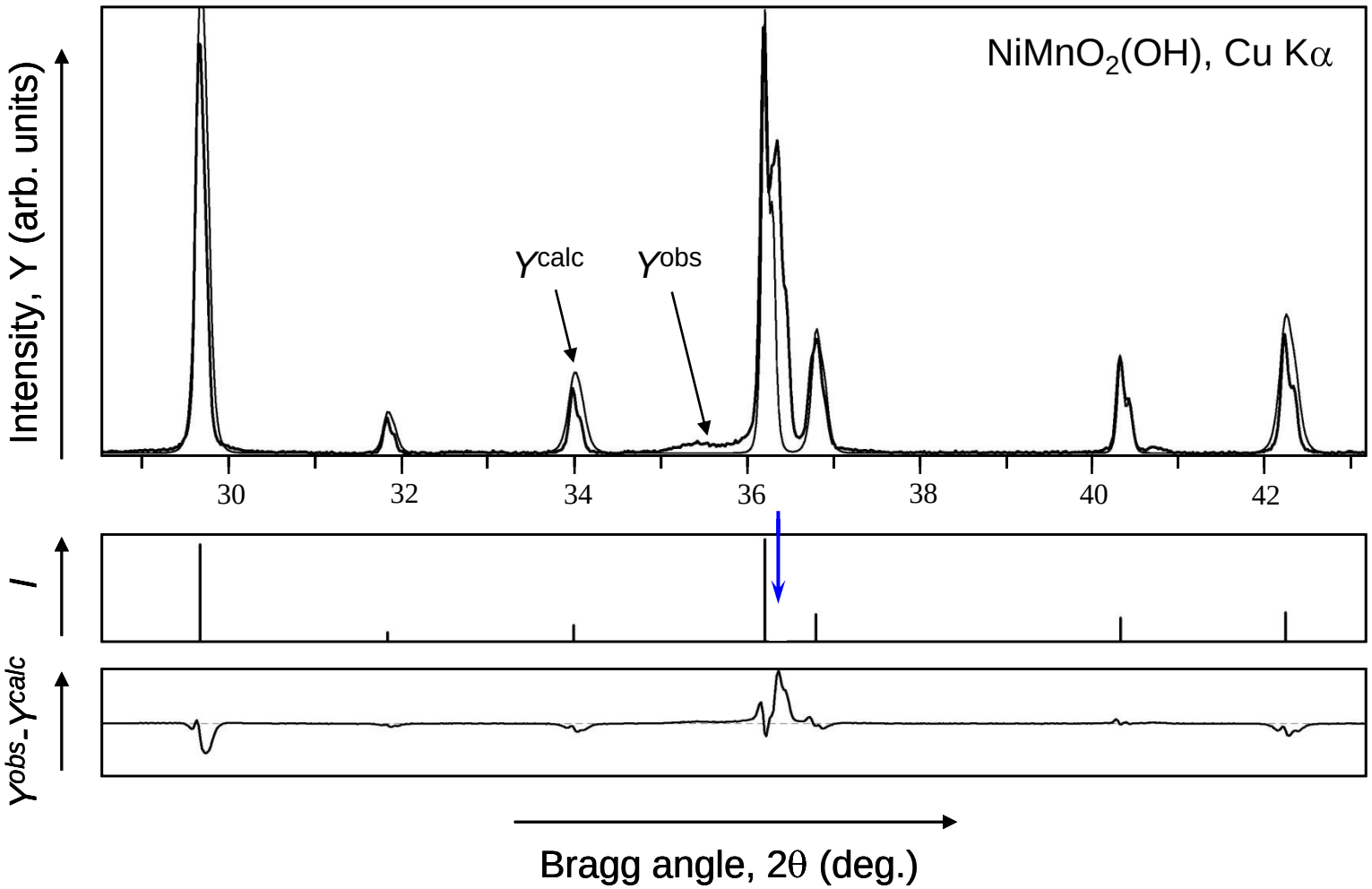

NiMnO2(OH), Cu Kα
a)
b)
c)
10
20
30
40
50
60
Intensity, Y (arb. units)
Bragg angle, 2θ (deg.)

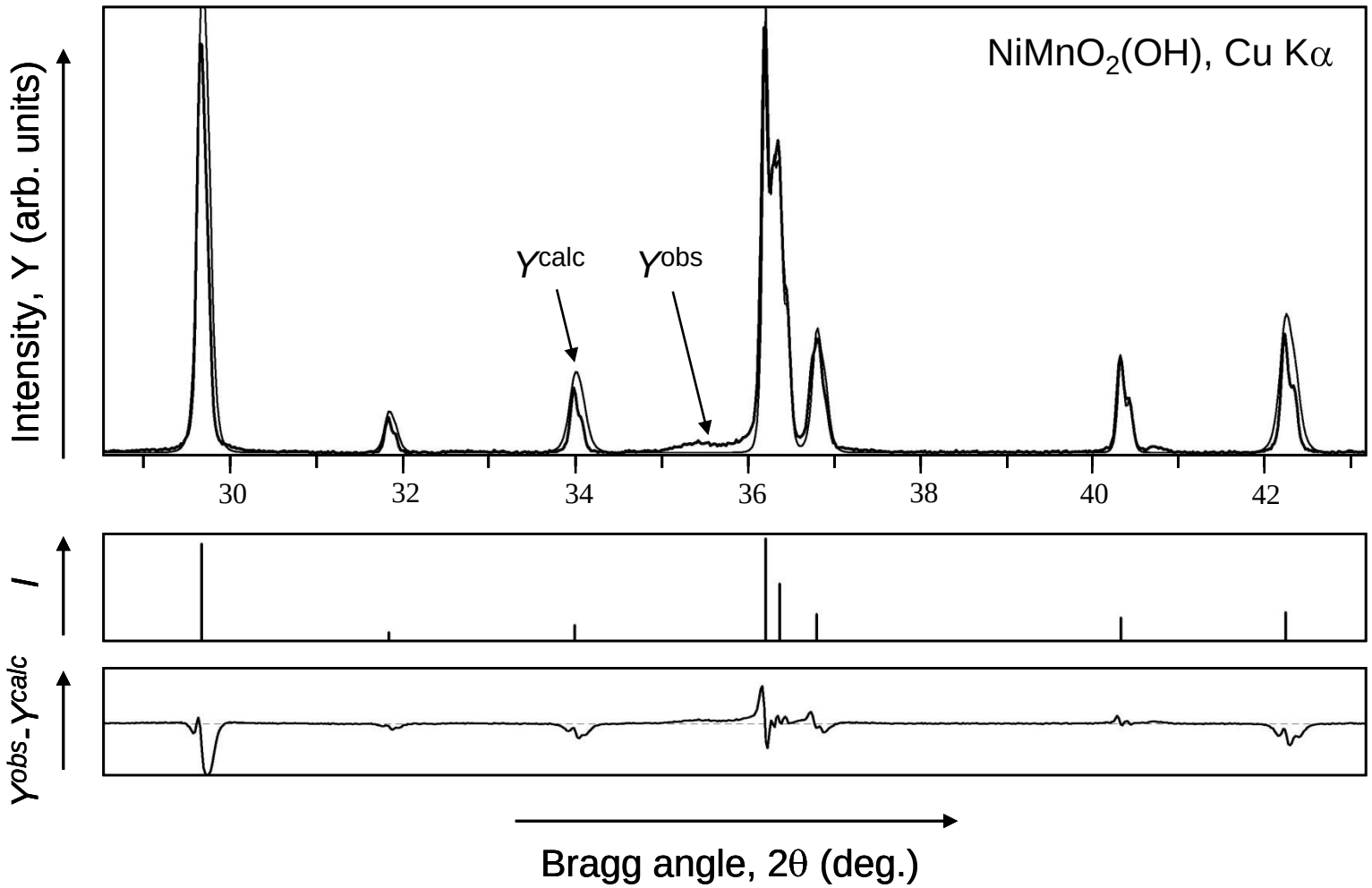

NiMnO2(OH), Cu Kα
Intensity, Y (arb. units)
10
20
30
40
50
60
Bragg angle, 2θ (deg.)

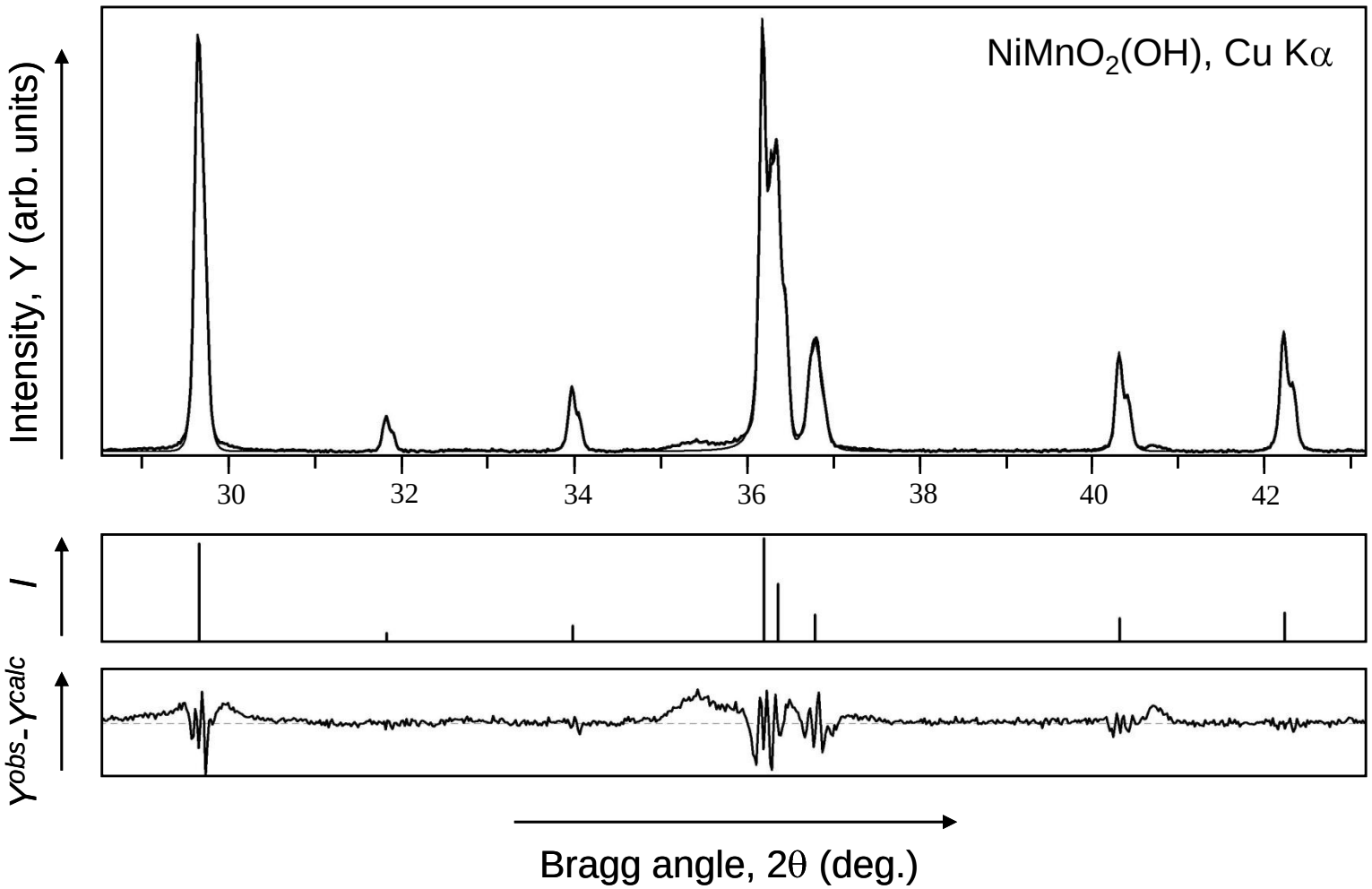

NiMnO2(OH), Cu Kα
a)
b)
c)
Intensity, Y (arb. units)
Bragg angle, 2θ (deg.)
70
0
30
0
30
0
10
20
30
40
50
60
20
24
28
32
36
40

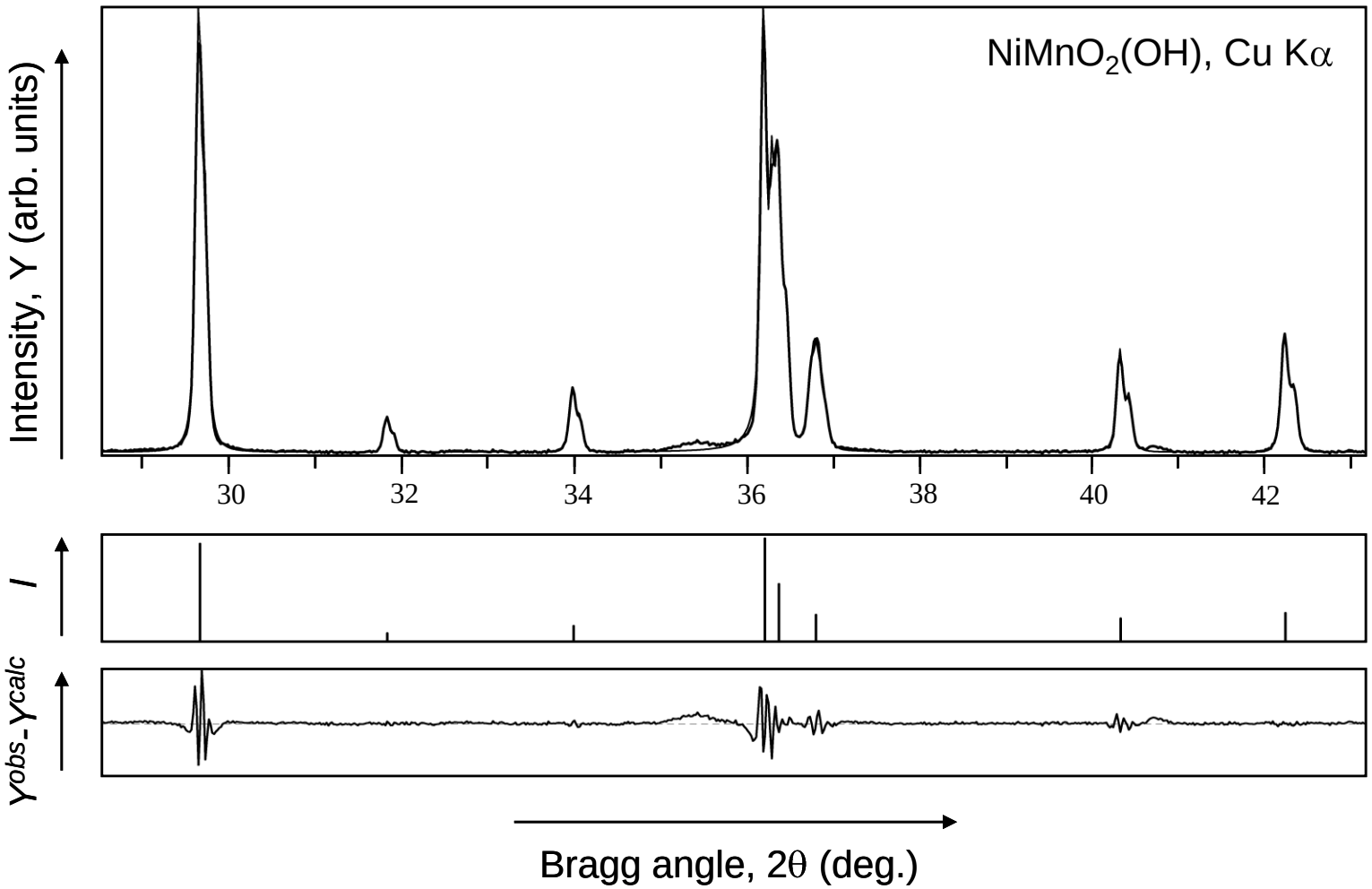

Y
∂Y/∂2θ
∂²Y/∂2θ²
2θ

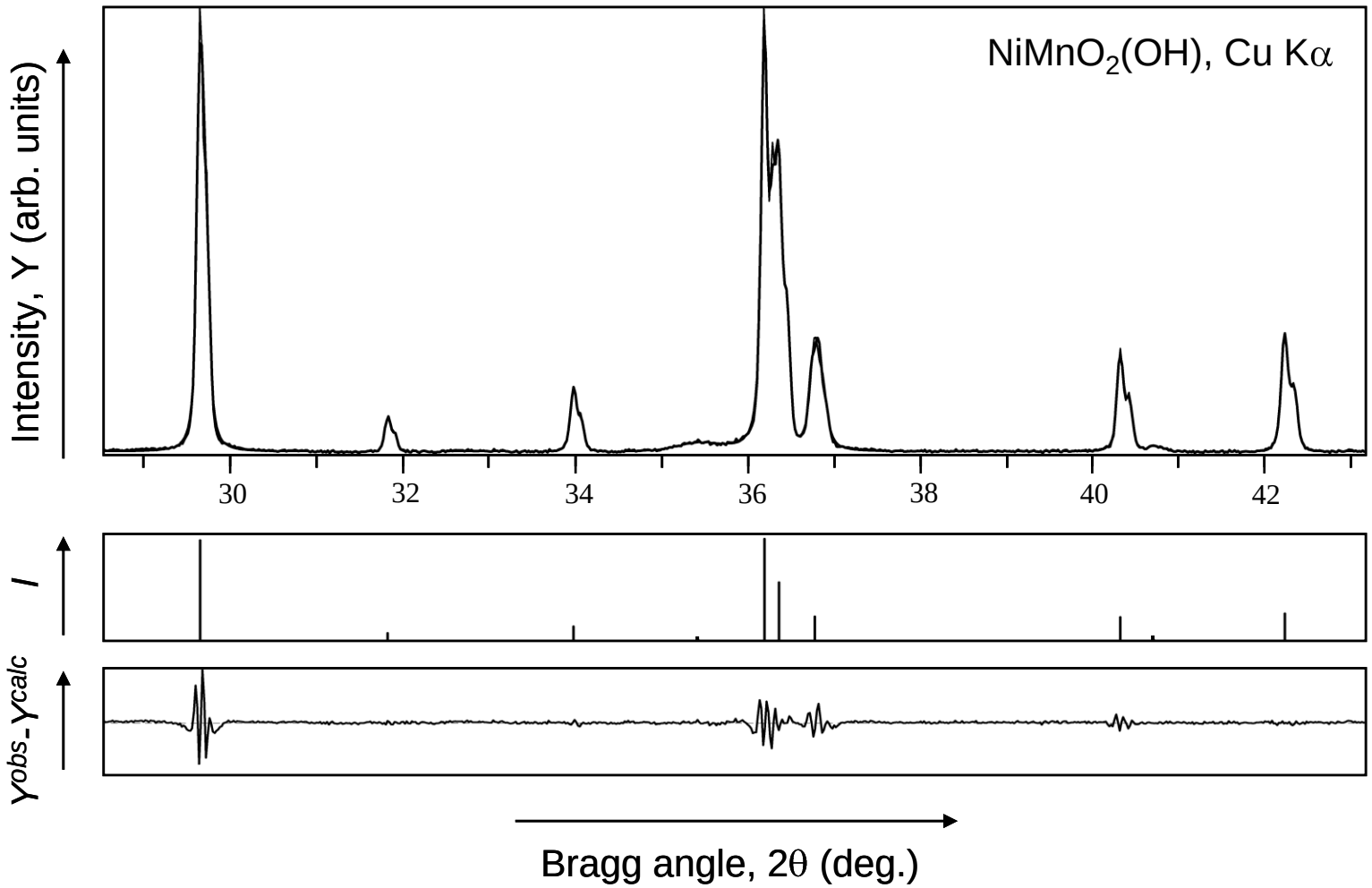

NiMnO2(OH), Cu Kα
Y
I
10
20
30
40
50
60
2θ

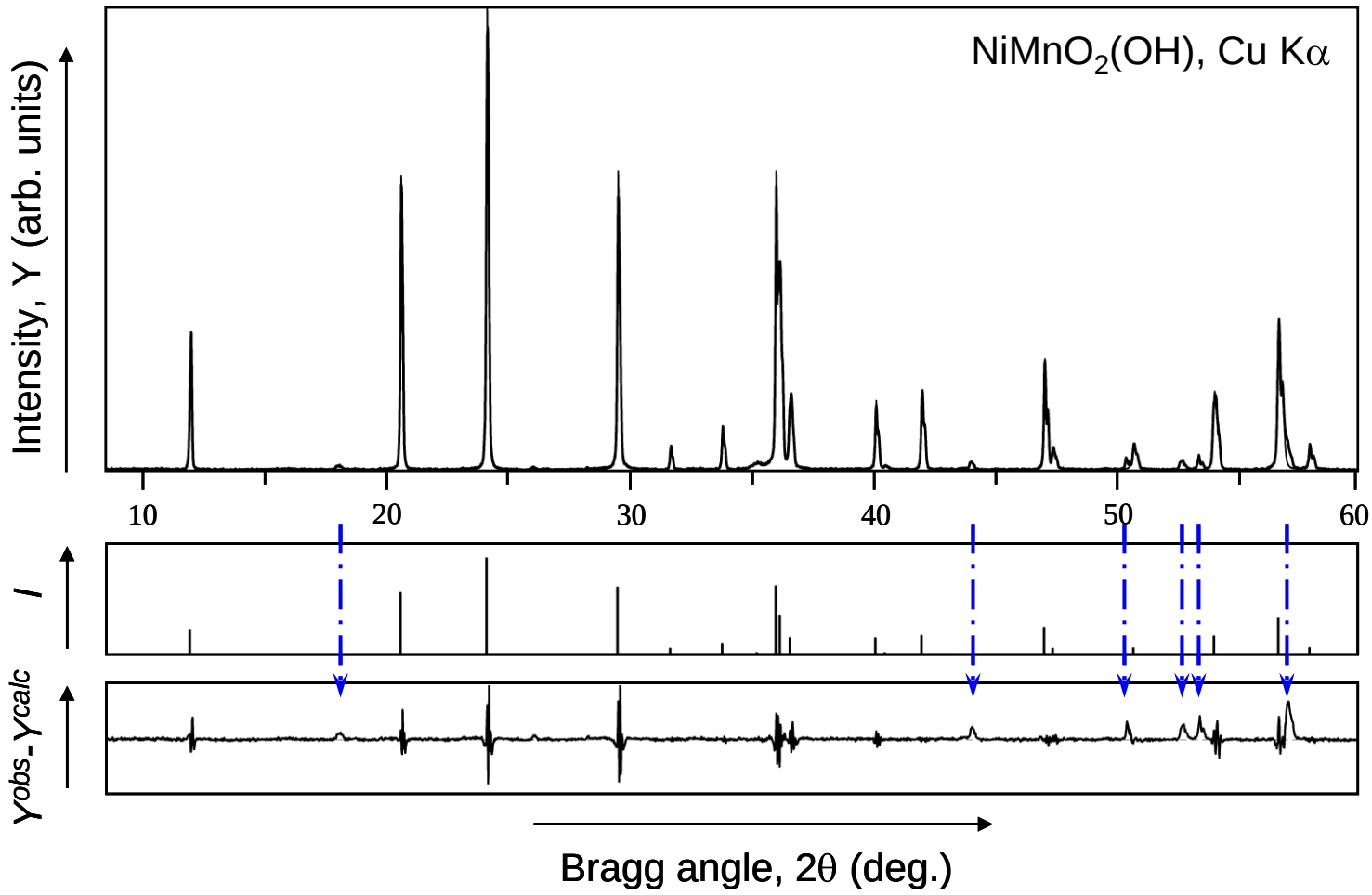

NiMnO2(OH), Cu Kα
Intensity, Y (arb. units)
Ycalc
Yobs
30
32
34
36
38
40
42
I
Yobs-Ycalc
Bragg angle, 2θ (deg.)

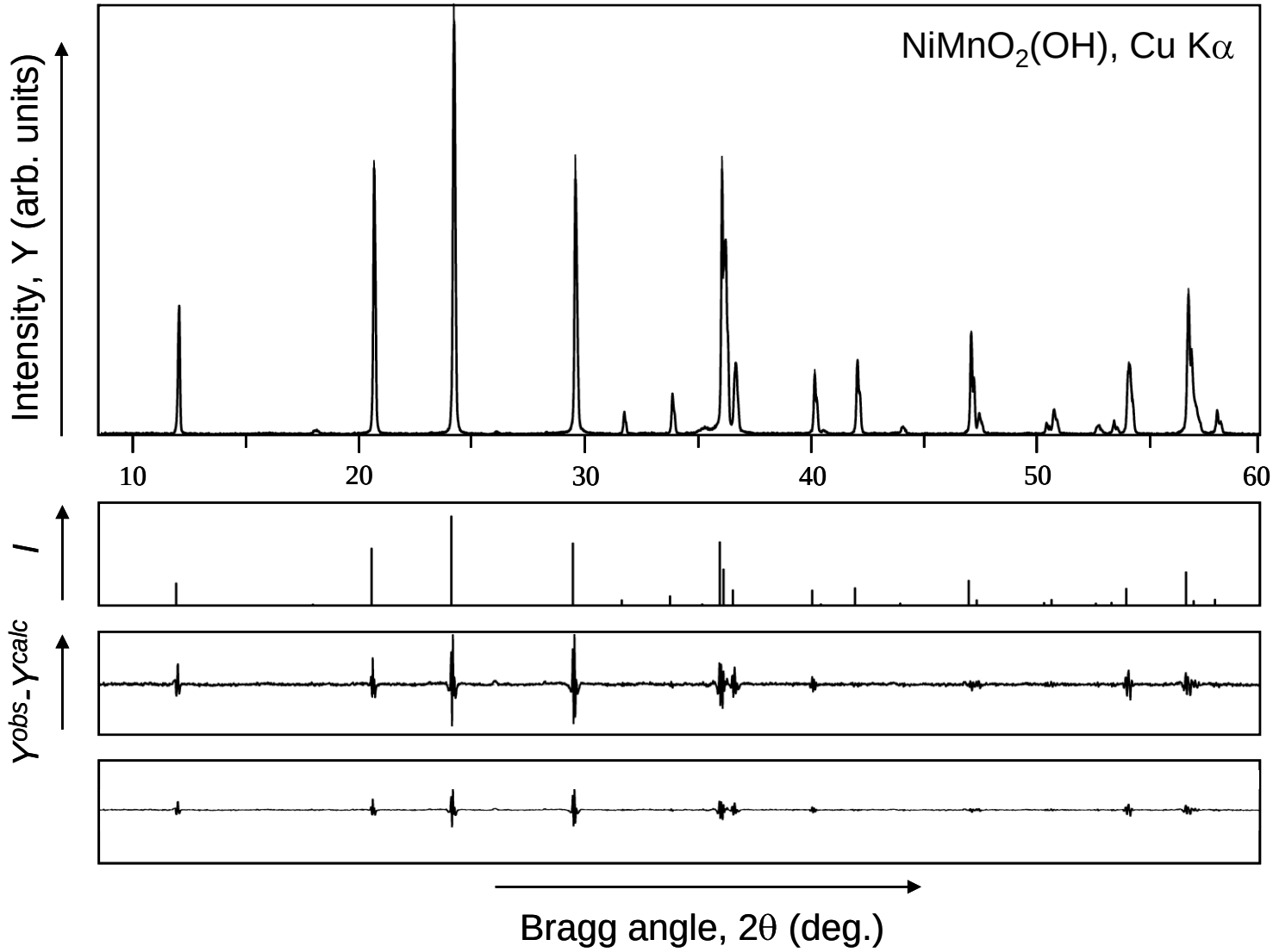

NiMnO2(OH), Cu Kα
Intensity, Y (arb. units)
Ycalc
Yobs
30
32
34
36
38
40
42
I
Yobs-Ycalc
Bragg angle, 2θ (deg.)

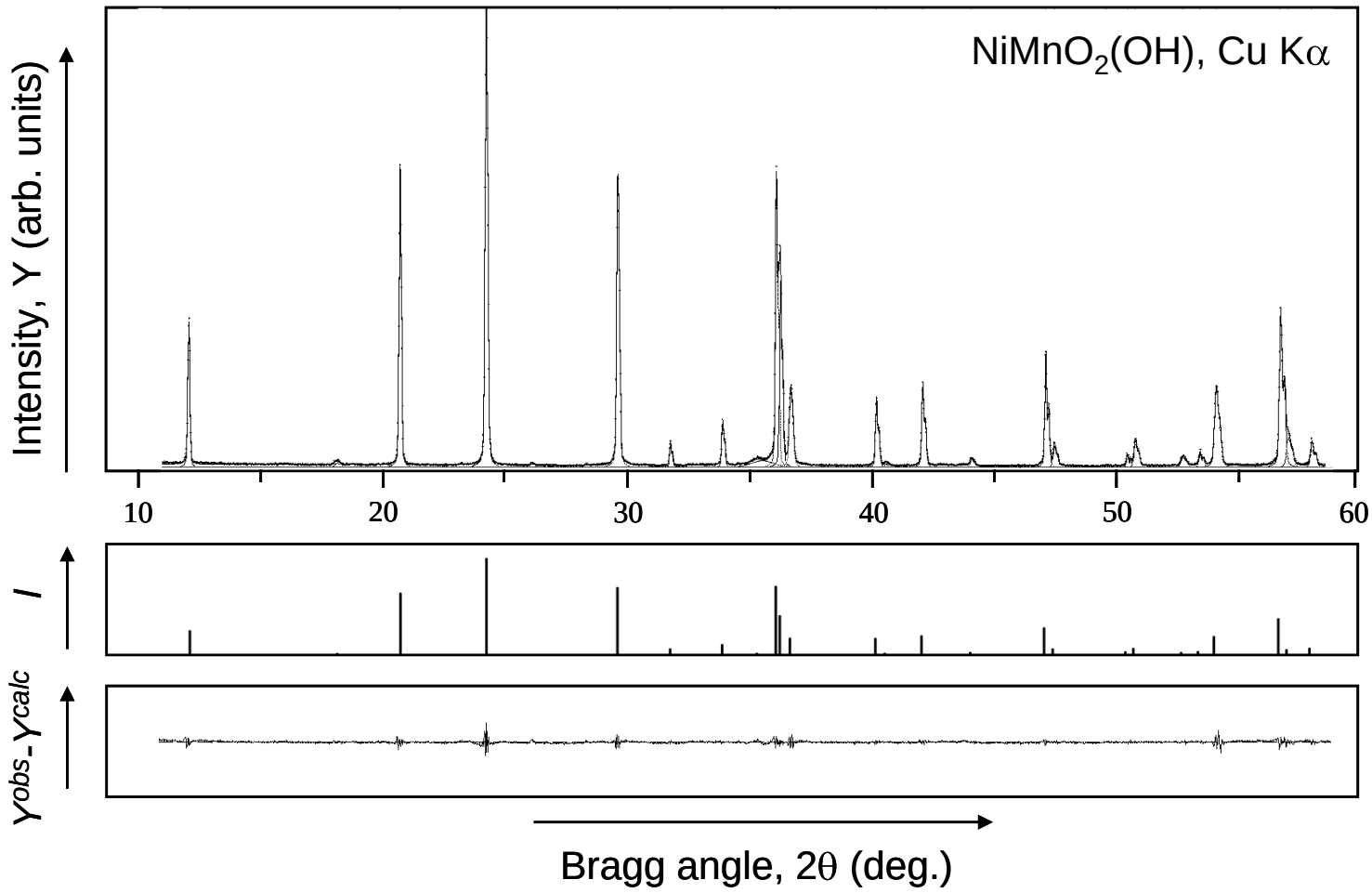

NiMnO2(OH), Cu Kα
Intensity, Y (arb. units)
30
32
34
36
38
40
42
I
Yobs-Ycalc
Bragg angle, 2θ (deg.)

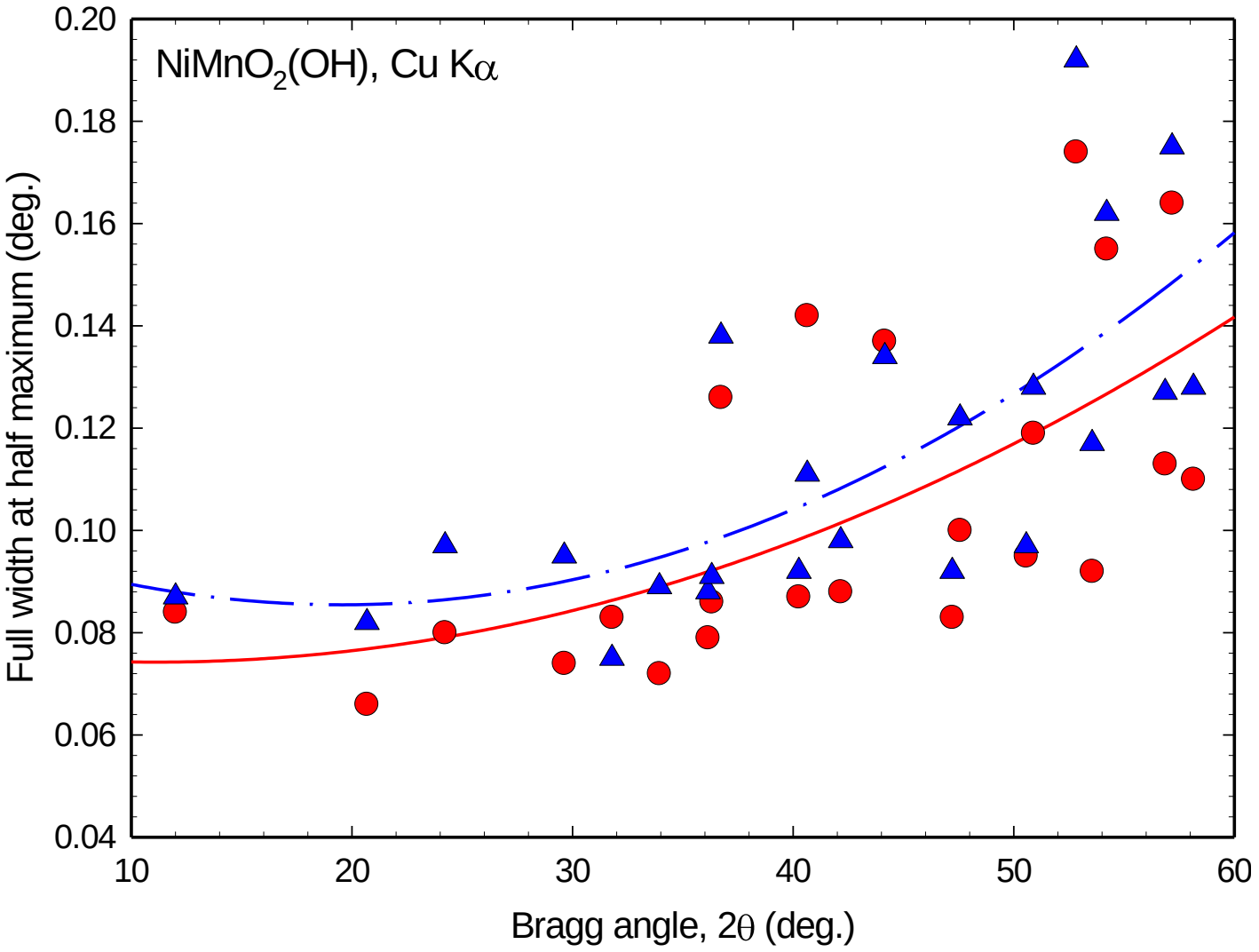

NiMnO2(OH), Cu Kα
Intensity, Y (arb. units)
30
32
34
36
38
40
42
I
Yobs-Ycalc
Bragg angle, 2θ (deg.)

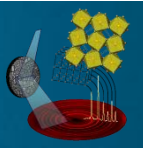

NiMnO2(OH), Cu Kα
Intensity, Y (arb. units)
30
32
34
36
38
40
42
I
Yobs-Ycalc
Bragg angle, 2θ (deg.)


NiMnO2(OH), Cu Kα
Intensity, Y (arb. units)
10
20
30
40
50
60
I
Yobs-Ycalc
Bragg angle, 2θ (deg.)


NiMnO2(OH), Cu Kα
Intensity, Y (arb. units)
10
20
30
40
50
60
I
Yobs-Ycalc
Bragg angle, 2θ (deg.)


NiMnO2(OH), Cu Kα
Intensity, Y (arb. units)
10
20
30
40
50
60
I
Yobs-Ycalc
Bragg angle, 2θ (deg.)


NiMnO2(OH), Cu Kα
Full width at half maximum (deg.)
Bragg angle, 2θ (deg.)
0.20
0.18
0.16
0.14
0.12
0.10
0.08
0.06
0.04
10
20
30
40
50
60



1 48-1152 Quality: Indexed

2 Li0.6 V1.67 O3.67 ! H2 O
Lithium Vanadium Oxide Hydrate

3 Rad:CuKa1 Lambda:1.54056 Filter: d sp:Diffractometer
Cutoff: Int:Diffractometer I/Icor:
Ref:Whittingham, M., SUNY at Binghamton, MaterialsResearch Center, NY, USA.Chyrayil, T., Zavalij, P., Whittingham, M., (1

4 Sys:Tetragonal S.G.:I4/mmm
a:3.7047±0.0003 b: c:15.804±0.002
α: β: γ: Z:2 mp
Ref2
Dx:2.53 Dm:2.541 SS/FOM: F30=46.5(0.0161,40) Volume[CD]:216.91

5 εα: ηωβ: εγ: Sign: 2V:
Ref3

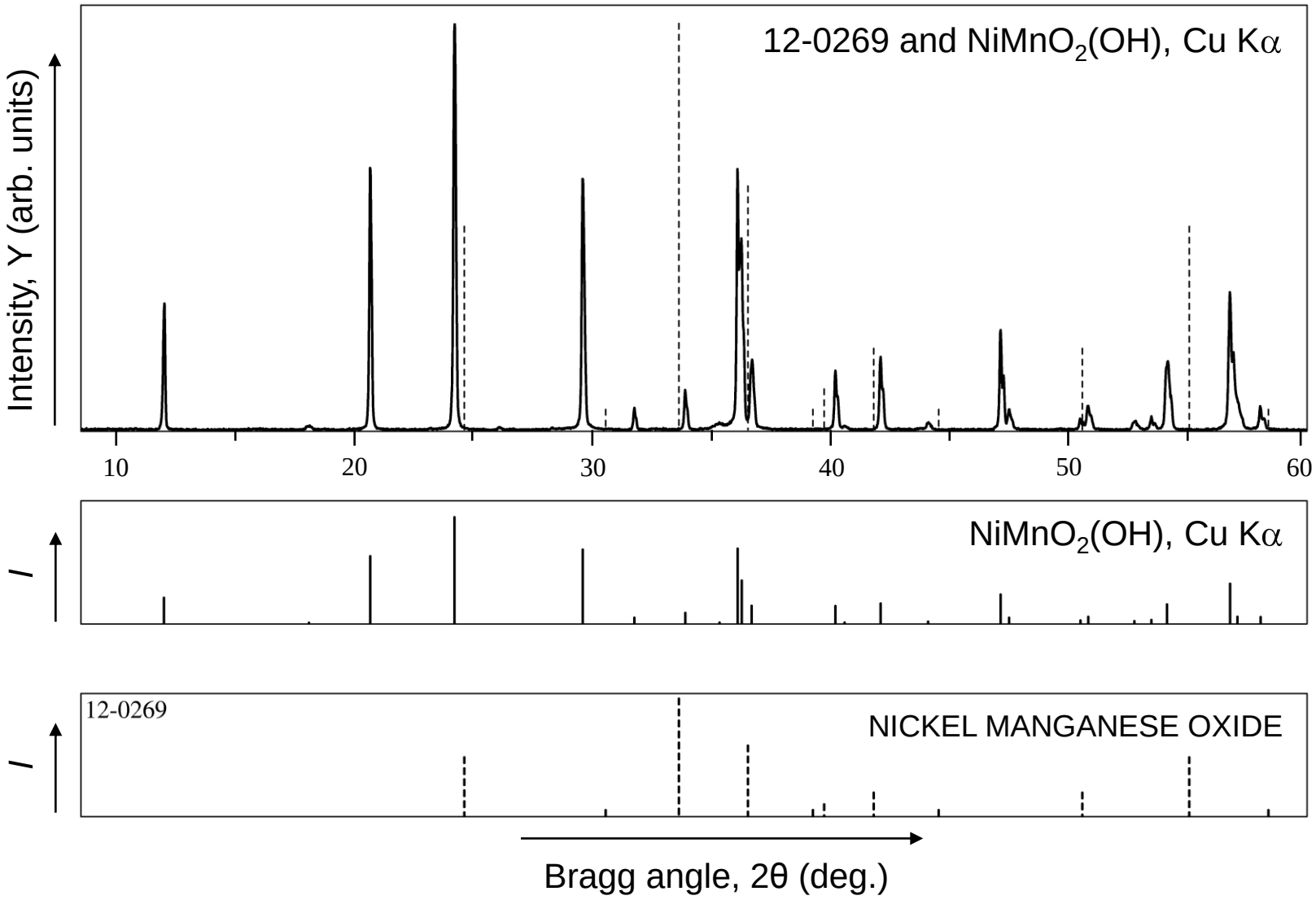
6 Color:

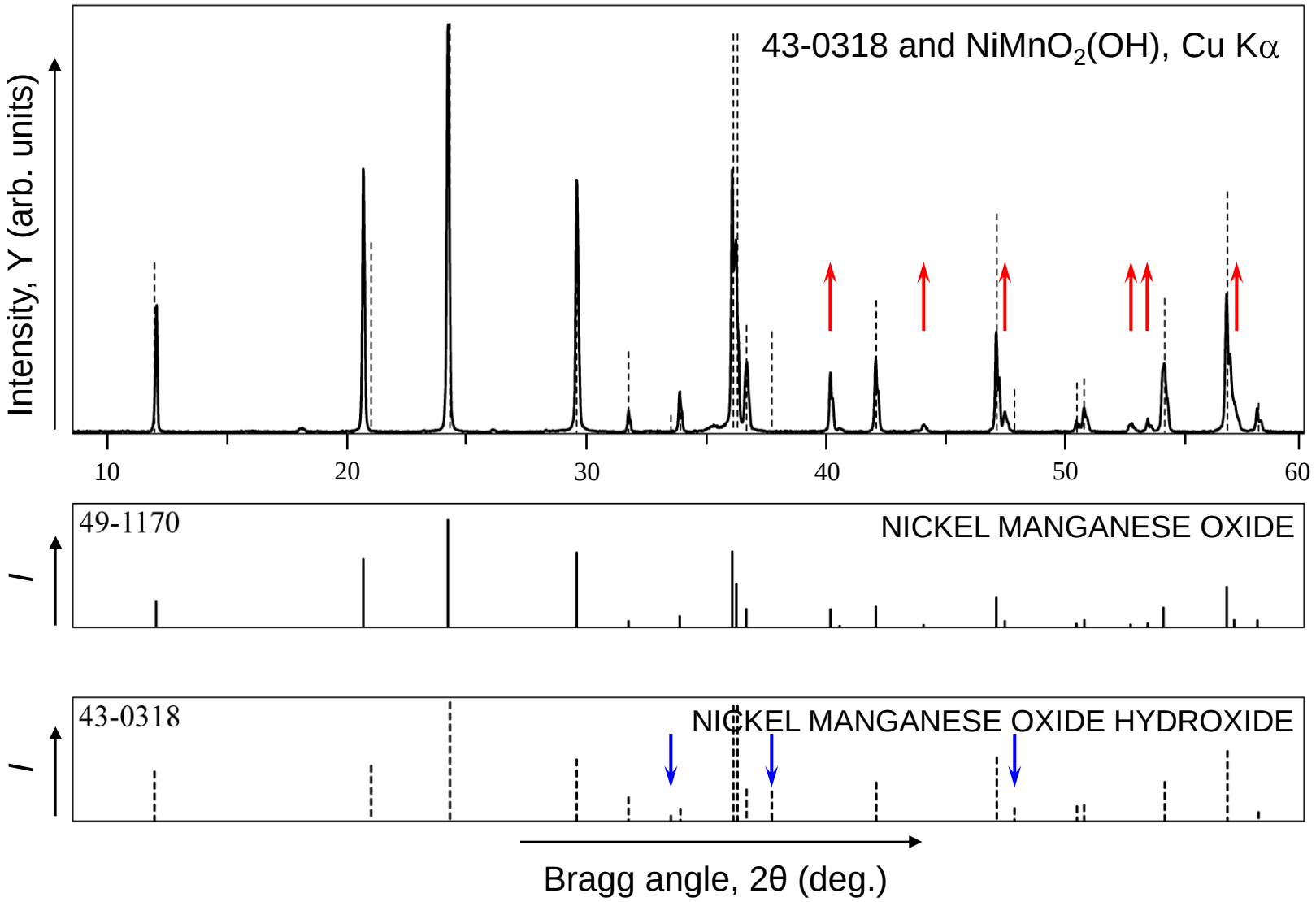
7 Prepared by hydrothermal treatment of tetramethylammonium hydroxide, vanadium pentoxide and \Li O H\ acidified to pH 2-5 for 3 days at 200 C. Pattern taken at 23(1) C.

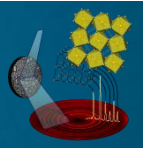
32 reflections in pattern.

8

2θ	Int.	h k l	2θ	Int.	h k l	2θ	Int.	h k l	2θ	Int.	h k l
11.2026	100	0 0 2	50.5721	8	0 2 2	72.0262	4	2 2 0	83.7228	1	0 1 13
22.4967	19	0 0 4	54.6668	3	0 2 4	73.1843	2	2 2 2	84.1343	1	0 3 5
24.6618	9	0 1 1	55.7443	2	1 2 1	76.5173	1	2 2 4			
29.4652	50	0 1 3	58.0669	3	0 1 9	77.4598	1	0 3 1			
33.9955	1	0 0 6	58.3367	13	1 2 3	79.4091	2	1 2 9			
34.2095	14	1 1 0	58.3367	13	0 0 10	79.6864	4	0 3 3			
36.0710	1	1 1 2	58.4543	4	1 1 8	79.6864	4	0 2 10			
37.3772	4	0 1 5	63.3383	3	1 2 5	81.7407	2	1 1 12			
47.1058	19	0 1 7	69.4008	10	1 1 10	82.1813	2	1 3 0			
49.1443	16	0 2 0	70.4377	7	1 2 7	83.3159	1	1 3 2			


12-0269 and NiMnO2(OH), Cu Kα
Intensity, Y (arb. units)
10
20
30
40
50
60
NiMnO2(OH), Cu Kα
I
12-0269
NICKEL MANGANESE OXIDE
I
Bragg angle, 2θ (deg.)


43-0318 and NiMnO2(OH), Cu Kα
Intensity, Y (arb. units)
10
20
30
40
50
60
49-1170
NICKEL MANGANESE OXIDE
I
43-0318
NICKEL MANGANESE OXIDE HYDROXIDE
I
Bragg angle, 2θ (deg.)



43-0318 Quality: Doubtful quality

Ni Mn O2 (O H)
Nickel Manganese Oxide Hydroxide

Rad:FeKa	Lambda:1.9373	Filter:	d sp:Diffractometer
Cutoff:	Int:Diffractometer	I/Icor:	
Ref:Yamamoto, N., 33 48, (1986)			

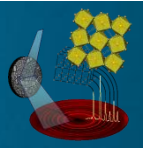
Sys:		S.G.:		
a:	b:	c:		
α:	β:	γ:	Z:	mp
Ref2				
Dx:	Dm:	SS/FOM:	Volume[CD]:0	
εα:	ηωβ:	εγ:	Sign:	2V:
Ref3				

Color:

Prepared by hydrothermal treatment at 200-320 C and 100 MPa of an equimolar mixture of \Ni (O H)2\ and $GG-MnOOH in a 1N \N H4 O H\ solution. Chemical analysis (wt.%): Ni 36.0, Mn 34.0, H 0.58, O 29.4. O assigned because unindexed.

21 reflections in pattern.

2 θ	Int.	h k l	2 θ	Int.	h k l	2 θ	Int.	h k l	2 θ	Int.	h k l
11.9241	41.3		37.8012	24.5		63.4445	12				
21.0048	46.2		42.1740	32.1							
24.3059	99.9		47.2279	53.3							
29.6253	51.6		47.9692	10.3							
31.7969	19.6		50.5841	12							
33.5754	4		50.8863	13							
33.9689	10		54.2674	32.6							
36.1914	97.3		56.8980	58.7							
36.3582	97.3		58.1954	7							
36.7434	26.1		61.6166	18.5							



49-1170 Quality: Quality Data

Ni Mn O3
Nickel Manganese Oxide

Rad:CuKa1 Lambda:1.54056 Filter: d sp:Diffractometer
Cutoff: Int:Diffractometer I/Icor:
Ref:Whittingham, S., SUNY at Binghamton, MaterialsResearch Center, NY, USA., (1997)

Sys:Orthorhombic S.G.:A21am
a:2.8609±0.0001 b:14.6482±0.0005 c:5.2703±0.0002
α: β: γ: Z:4 mp
Ref2
Dx:4.861 Dm:4.861 SS/FOM: F30=103.1(0.0081,36) Volume[CD]:220.86

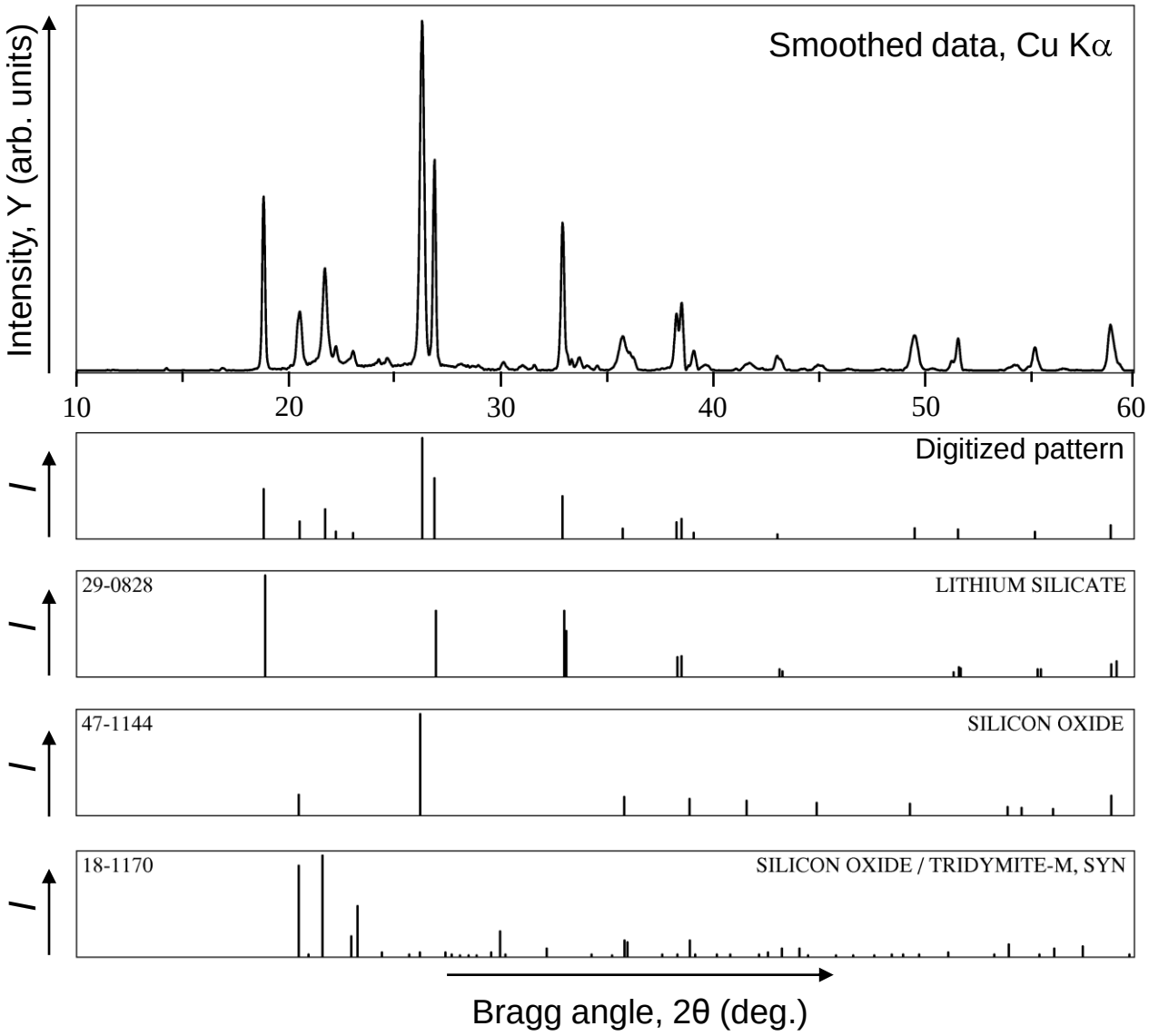
εα: ηωβ: εγ: Sign: 2V:
Ref3

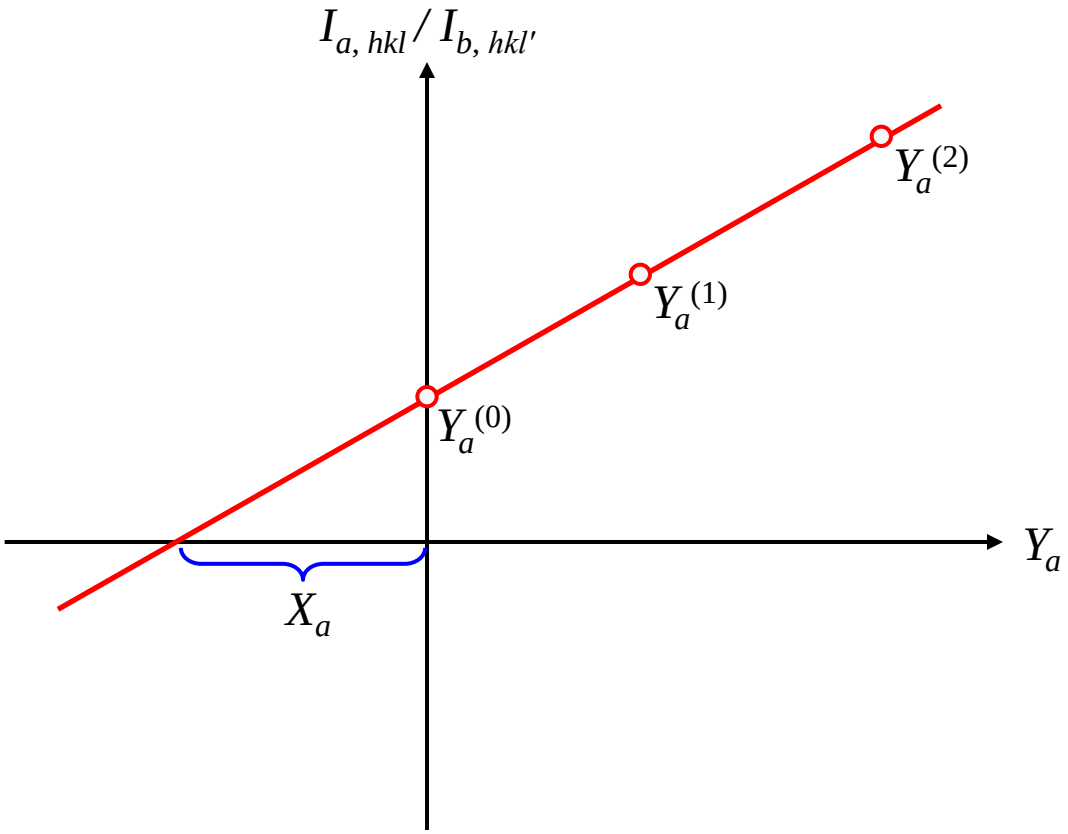
Color:

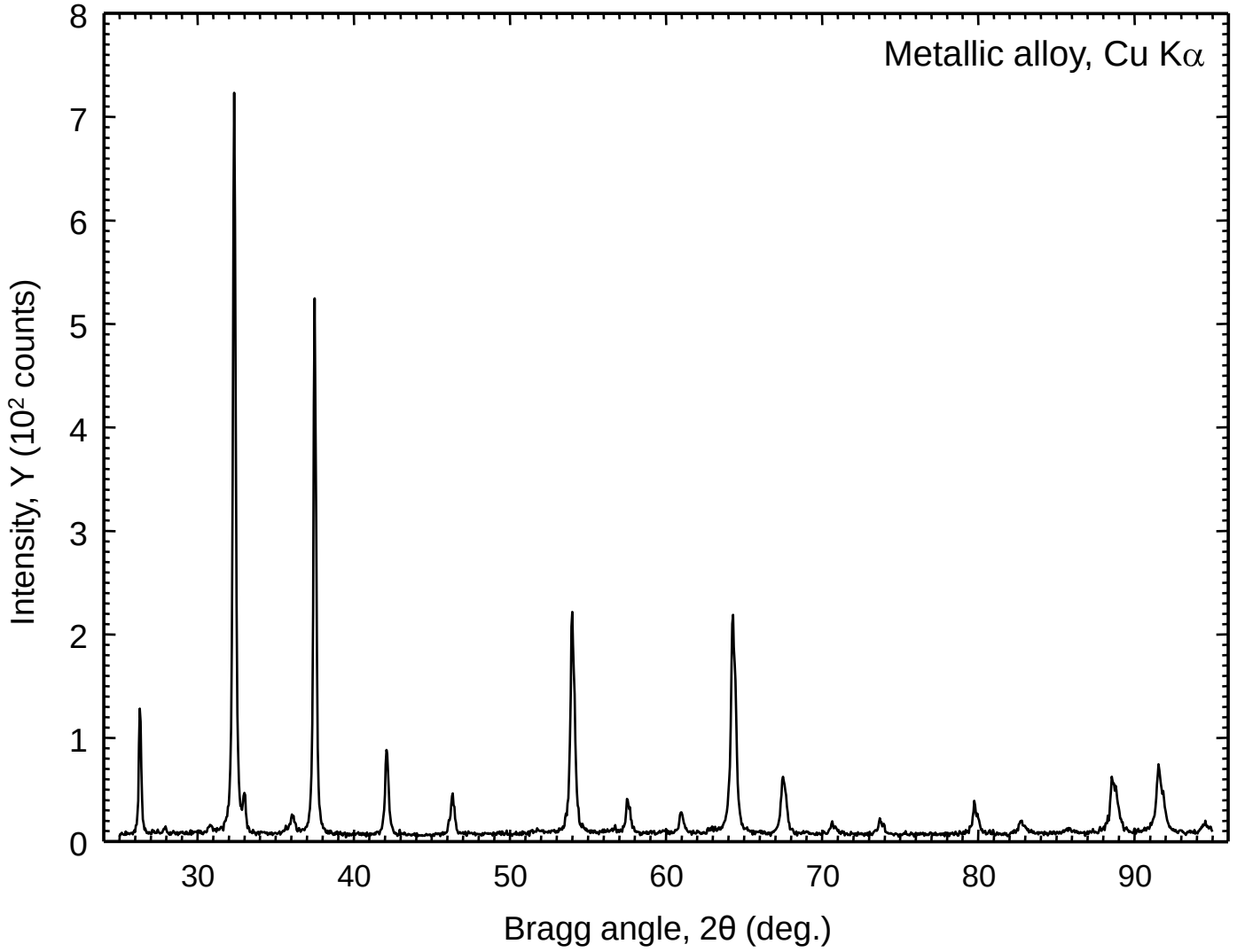
Prepared by hydrothermal treatment of tetramethylammonium permanganate, nickel acetate and lithium carbonate for 2 days at 200 C. Pattern taken at 23(1) C.

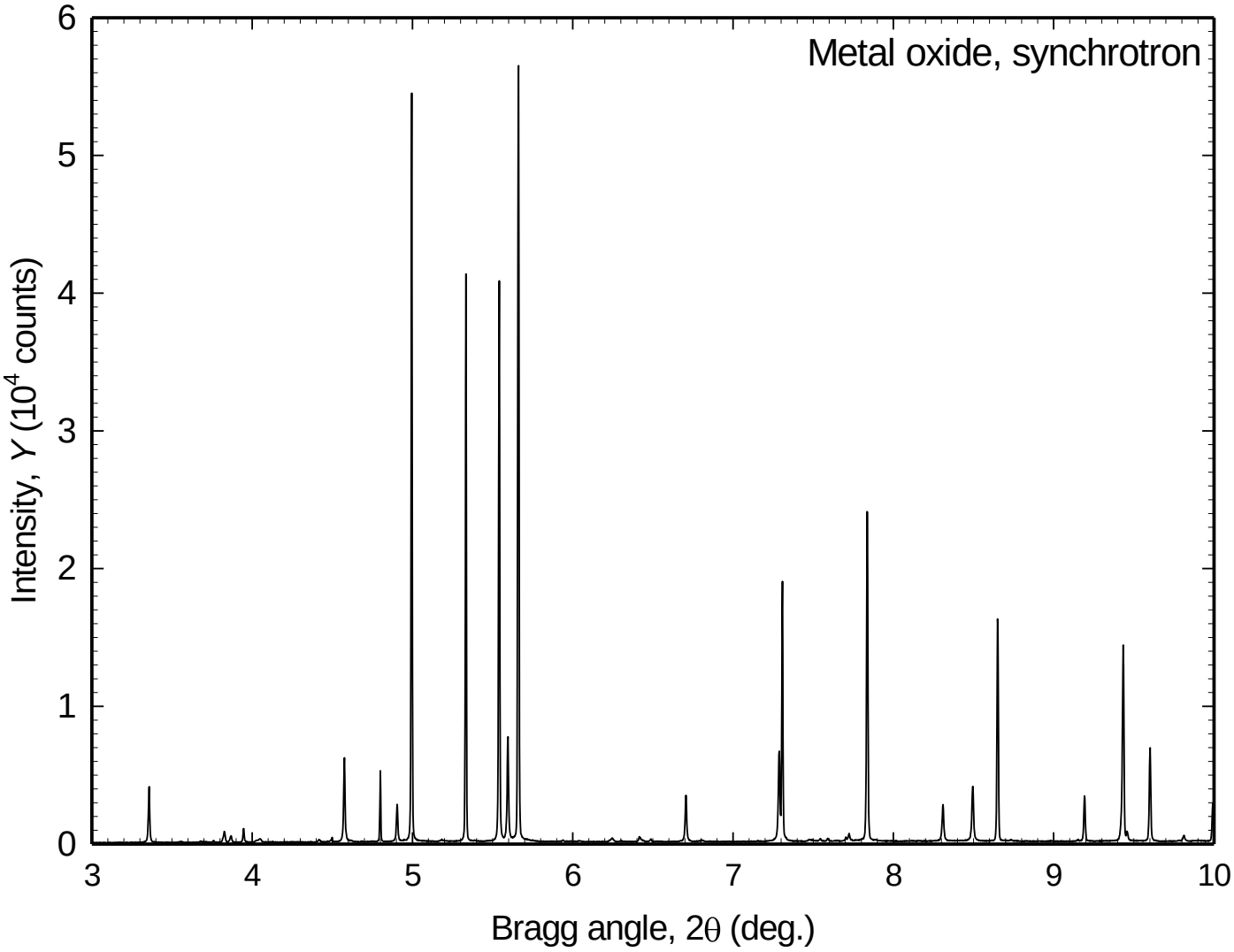
55 reflections in pattern.

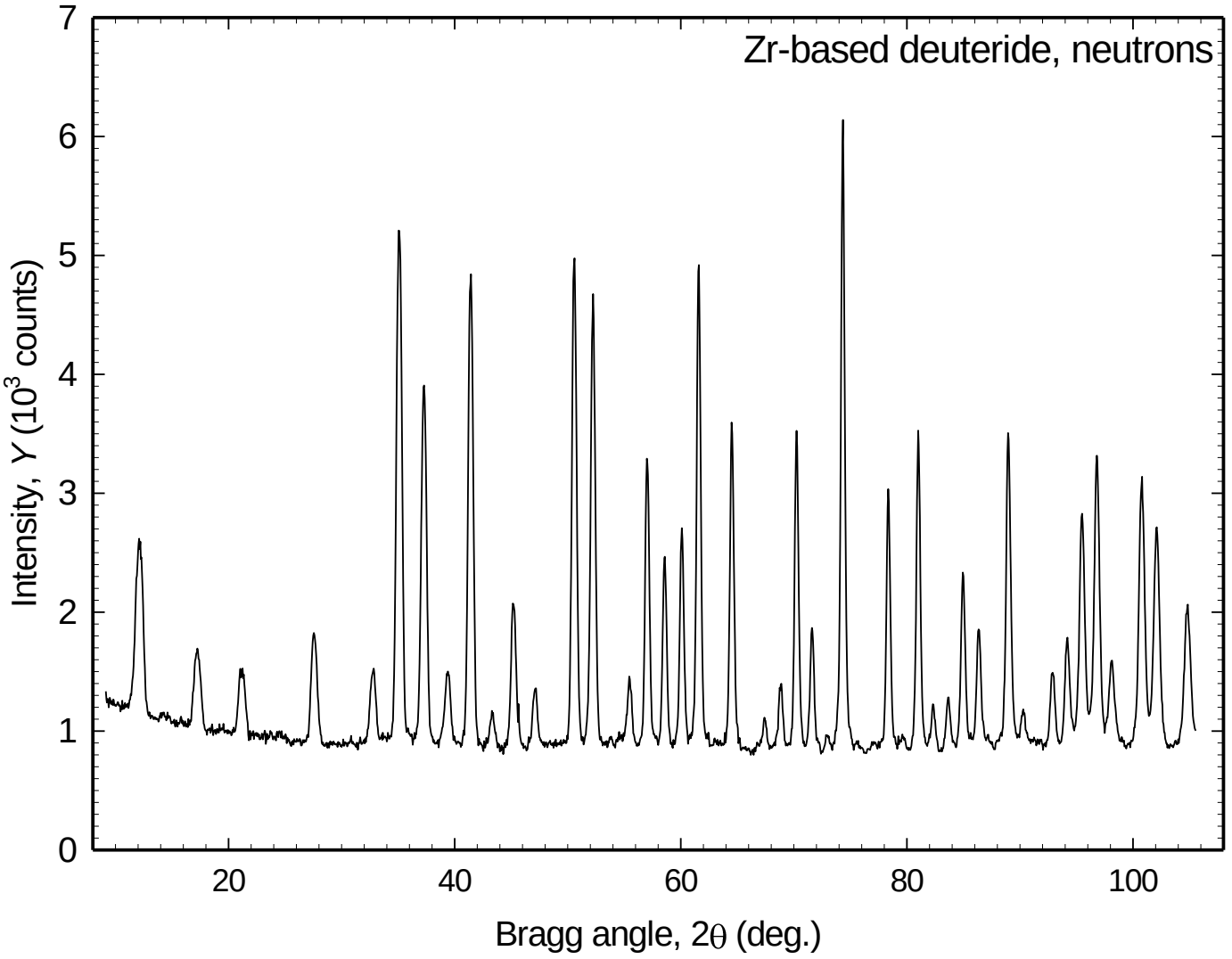
2 θ	Int.	h k l	2 θ	Int.	h k l	2 θ	Int.	h k l	2 θ	Int.	h k l
12.0679	25	0 2 0	47.6194	6	1 5 1	66.2340	1	0 10 1	80.4338	2	1 1 4
20.7417	55	0 2 1	50.6172	3	1 3 2	68.6303	14	1 9 1	80.7356	16	0 12 1
24.2722	100	0 4 0	50.9411	9	0 6 2	69.1962	2	2 2 1	80.7356	16	2 4 2
29.6756	65	0 4 1	52.8898	3	0 8 1	70.6260	5	2 4 0	81.7659	3	1 11 1
31.8434	5	1 1 0	53.6083	3	0 2 3	71.5591	12	0 0 4	83.0093	10	1 3 4
33.9955	10	0 0 2	54.2640	27	1 7 0	72.8763	3	0 2 4	83.2543	6	0 6 4
36.1914	62	0 2 2	56.9133	50	1 5 2	73.2173	4	2 4 1	88.1190	12	1 5 4
36.3735	43	1 3 0	57.2185	12	1 7 1	74.8306	3	0 8 3	88.1190	12	0 12 2
36.7901	23	0 6 0	58.1954	8	0 4 3	75.5715	1	2 0 2	88.6939	6	1 9 3
40.3213	16	1 3 1	61.6257	44	0 8 2	76.3434	3	1 9 2	89.1644	11	1 11 2
40.7014	1	0 6 1	62.2876	6	1 1 3	76.8677	11	2 2 2	93.0153	3	2 4 3
42.2153	22	0 4 2	65.1455	21	2 0 0	77.2366	3	2 6 0	95.6870	10	1 7 4
44.2102	2	1 5 0	65.3921	2	0 6 3	78.4823	4	1 7 3	96.0050	11	2 8 2
47.2644	28	1 1 2	65.6043	1	1 7 2	79.2909	4	1 11 0			

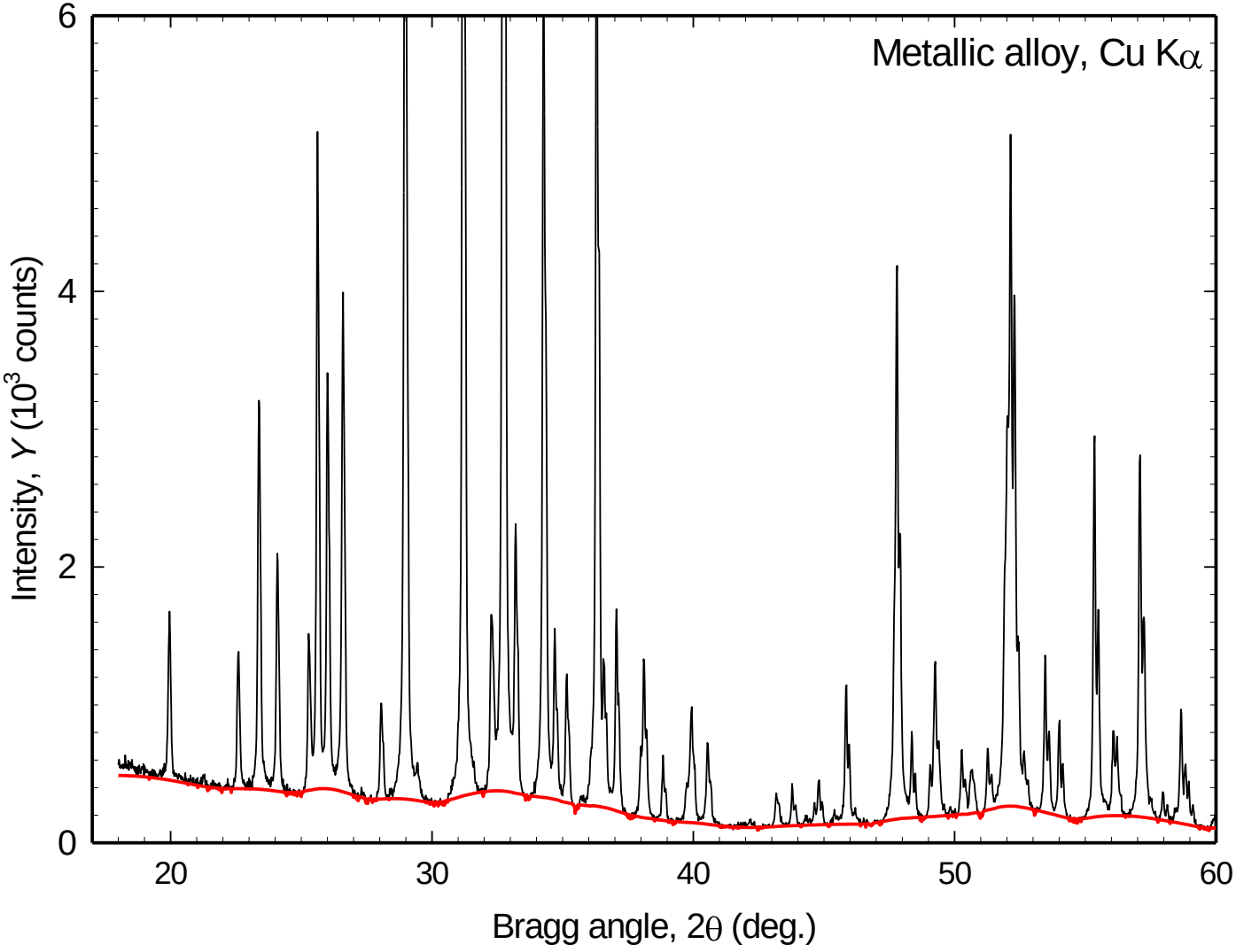

Intensity, Y (arb. units)
Smoothed data, Cu Kα
10
20
30
40
50
60
I
Digitized pattern
29-0828
LITHIUM SILICATE
47-1144
SILICON OXIDE
18-1170
SILICON OXIDE / TRIDYMITE-M, SYN
Bragg angle, 2θ (deg.)

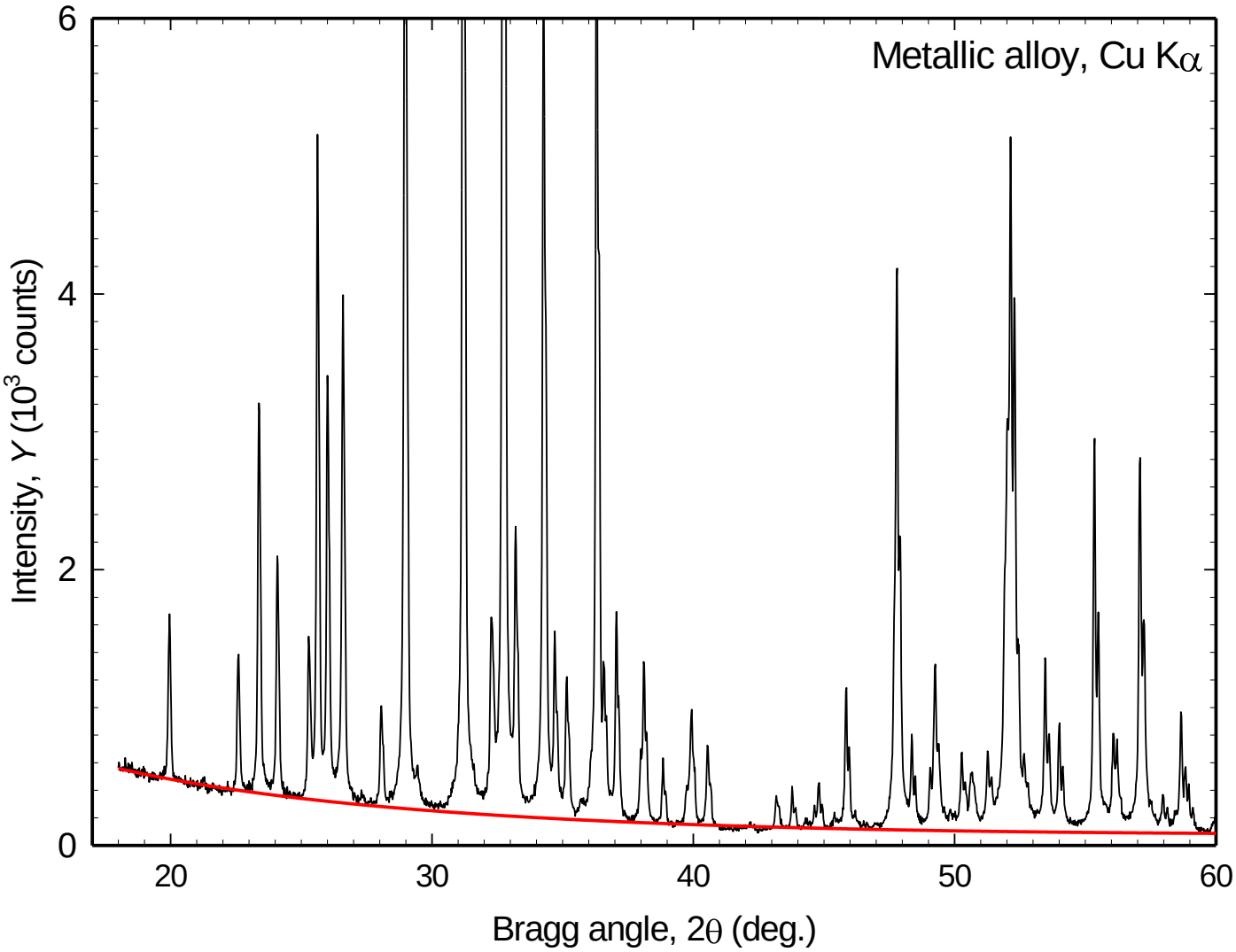

$I_{a,\,hkl} / I_{b,\,hkl'}$
$Y_a^{(2)}$
$Y_a^{(1)}$
$Y_a^{(0)}$
Y_a
X_a

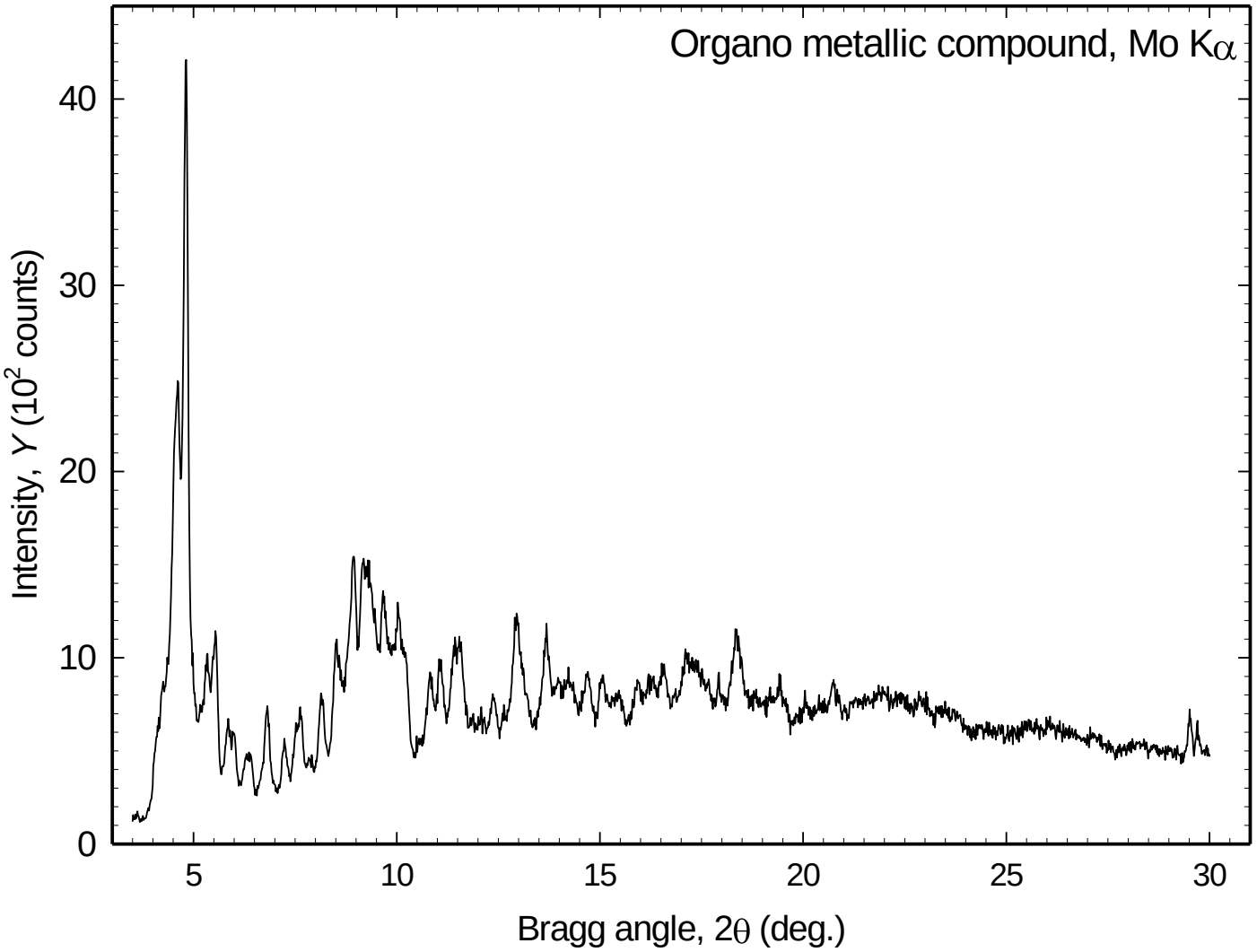

Metallic alloy, Cu Kα
Intensity, Y (10² counts)
Bragg angle, 2θ (deg.)
0
1
2
3
4
5
6
7
8
30
40
50
60
70
80
90


Metal oxide, synchrotron
Intensity, Y (10^4 counts)
Bragg angle, 2θ (deg.)
0
1
2
3
4
5
6
3
4
5
6
7
8
9
10


Zr-based deuteride, neutrons
Intensity, Y (10^3 counts)
Bragg angle, 2θ (deg.)
0
1
2
3
4
5
6
7
20
40
60
80
100


Metallic alloy, Cu Kα
Intensity, Y (10^3 counts)
Bragg angle, 2θ (deg.)
0
2
4
6
20
30
40
50
60


Metallic alloy, Cu Kα
Intensity, Y (10^3 counts)
Bragg angle, 2θ (deg.)
0
2
4
6
20
30
40
50
60


Organo metallic compound, Mo Kα
Intensity, Y (10² counts)
0
10
20
30
40
5
10
15
20
25
30
Bragg angle, 2θ (deg.)