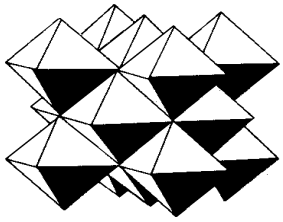
experiment 16



identification of unknown

OBJECT OF EXPERIMENT Identification of individual constituents in a mixture.

EQUIPMENT AND MATERIALS A mixture of two unknown constituents, carefully ground in an agate mortar, was passed through a 325-mesh sieve. A microscopic examination of the powder showed no shape irregularities so that shape texture was not likely to affect relative intensities. The powder, packed in a suitable holder, was examined in a diffractometer and the tracing reproduced in Fig. 16-1 was obtained. Because the size of the tracing had to be reduced from a length of about two feet in the original, the 2θ values of the first fifteen lines recorded are listed in Worksheet 16-1. Actually the 2θ values of the next 10 reflections are also included even though they are not shown in Fig. 16-1. The intensities of these reflections were weak and can be assigned approximately one-tenth the value of the first and most intense reflection.

The copper-target x-ray tube was operated at 40 kV and 20 mA during the exposure. A scintillation counter was used to record the intensities with a time constant of 4 and scale factor of 8.

The identification of the constituents requires the use of an *Index* to the *Powder data file* and a set of cards. Pertinent sections of the *Index* are reproduced in Table 16-1 while selected cards are reproduced in Fig. 16-2.

PROCEDURE It is first necessary to convert the 2θ values on Worksheet 16-1 to interplanar spacing values. Then the relative intensity of each reflection is read from the diffractometer tracing, usually by assigning a value of 100 to the most intense reflection. In the present case, the scale factor was adjusted so that the most intense reflection has a peak height just barely in excess of 100 on the chart paper so that the peak heights of the other reflections can be read off the chart

directly and entered in the third column of the worksheet. (Don't forget to subtract the background.)

It is next necessary to establish which one of the more intense reflections constitutes the second most intense line of the first constituent to be identified. Proceeding systematically, consider each of the next four possibilities as diagramed in Worksheet 16-2. Since the most intense reflection has $d = 3.23$ Å, it is only necessary to consult Hannawalt groups listing d values ranging from 3.29 to 3.25 Å and 3.24 to 3.20 Å, pertinent portions of which are reproduced in Table 16-1. Note that the third reflection in each set selected from these groups in the *Index* must have a d value actually observed, that is, listed on Worksheet 16-1. The card numbers for each possible match are also noted on Worksheet 16-2 so that the individual cards corresponding to prospective identifications can be selected easily for further comparisons. All the cards that should be consulted in the present case are reproduced in Fig. 16-2.

After the first constituent is identified, the remaining d values and their intensities, suitably renormalized, are transferred to the last two columns in Worksheet 16-1 and the above process is repeated until the second constituent is identified.

RESULTS In the present exercise, the names or chemical compositions listed on the cards correctly matching the d and I values observed represent the desired result.

ADDITIONAL ASSIGNMENTS If a *Fink Index* is available, repeat the above identification procedure using it.

DISCUSSION In the present instance, once the d values belonging to the first constituent in the mixture were eliminated, the second identification was trivially straightforward. Do you expect this to be the case usually when examining mixtures? Qualify your answer giving specific reasons.

Experience indicates that, when mixtures containing unknown numbers of constituents are examined, the identification of the first component is usually more straightforward than that of the others. Suggest possible reasons why this should be so.

REFERENCES Text chapter 19.

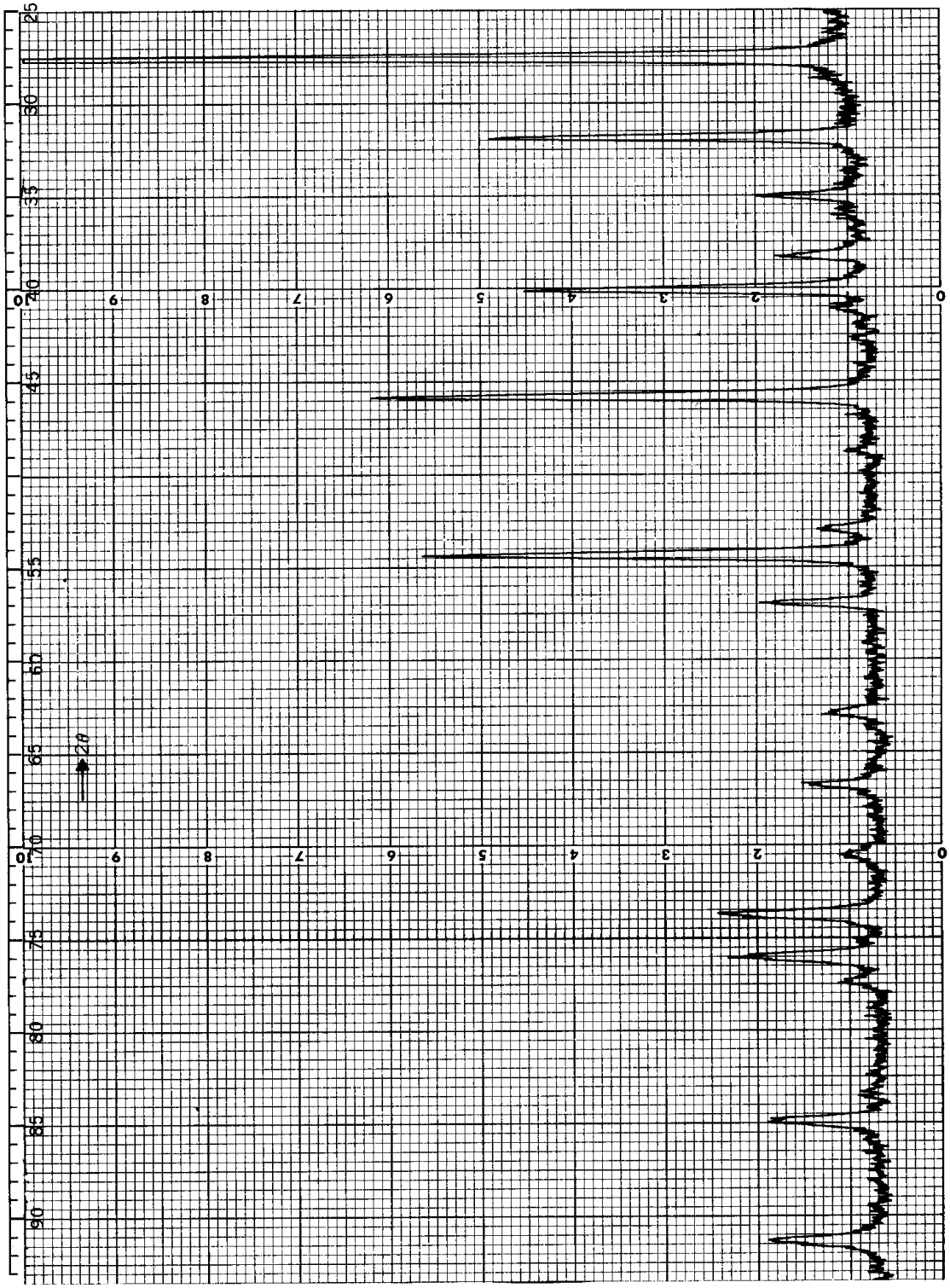
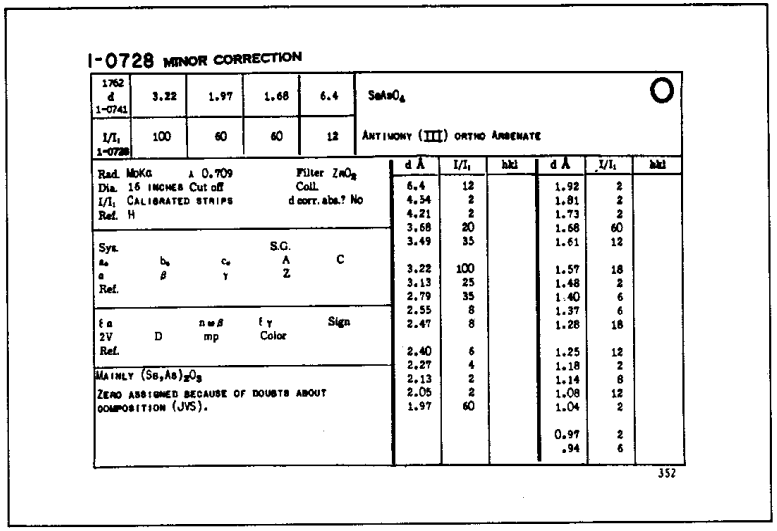


FIG. 16-1 DIFFRACTOMETER TRACING OF MIXTURE.



1-0728 MINOR CORRECTION

1762 d 1-0741	3.22	1.97	1.68	6.4	$SeAsO_4$
I/I_1 1-0728	100	60	60	12	Antimony (III) ortho Arsenate

Rad. MoKα λ 0.709 Filter ZrO_2
Dia. 16 inches Cut off Coll.
I/I_1 Calibrated strips d corr. abs.? No
Ref. H

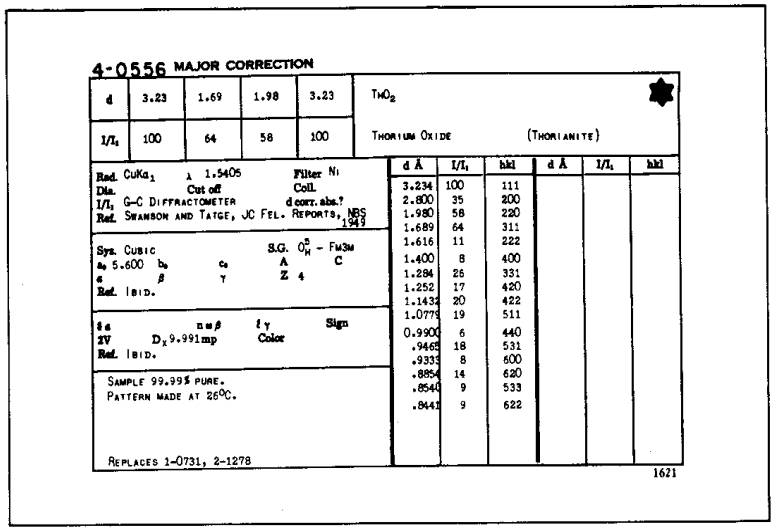
Sys. S.G.
a_o b_o c_o A C
α β γ Z
Ref.

ε α nωβ εγ Sign
2V D mp Color
Ref.

Mainly $(Se,As)_2O_3$
Zero assigned because of doubts about composition (JVS).

d Å	I/I_1	hkl	d Å	I/I_1	hkl
6.4	12		1.92	2	
4.54	2		1.81	2	
4.21	2		1.73	2	
3.68	20		1.68	60	
3.49	35		1.61	12	
3.22	100		1.57	18	
3.13	25		1.48	2	
2.79	35		1.40	6	
2.55	8		1.37	6	
2.47	8		1.28	18	
2.40	6		1.25	12	
2.27	4		1.18	2	
2.13	2		1.14	8	
2.05	2		1.08	12	
1.97	60		1.04	2	
			0.97	2	
			.94	6	

352



4-0556 MAJOR CORRECTION

d	3.23	1.69	1.98	3.23	ThO_2
I/I_1	100	64	58	100	Thorium Oxide (Thorianite)

Rad. $CuK\alpha_1$ λ 1.5405 Filter Ni
Dia. Cut off Coll.
I/I_1 G-C Diffractometer d corr. abs.?
Ref. Swanson and Tatge, JC Fel. Reports, NBS 1949

Sys. Cubic S.G. O_h^5 - Fm3m
a_o 5.600 b_o c_o A C
α β γ Z 4
Ref. Ibid.

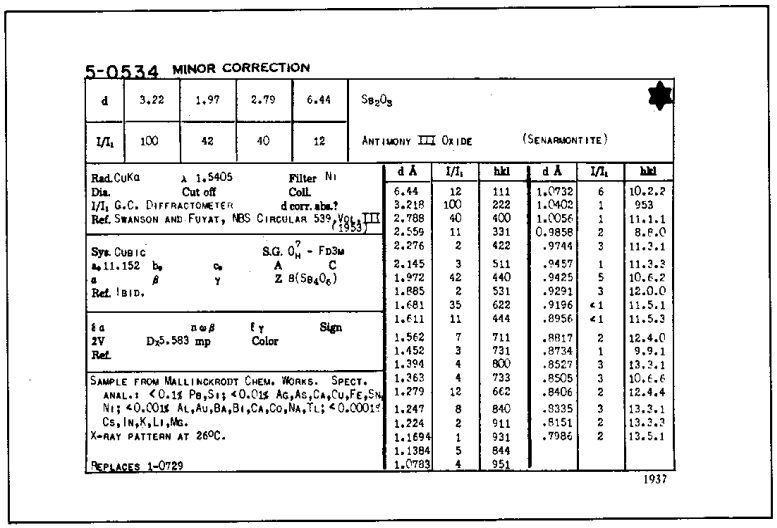
ε α nωβ εγ Sign
2V D_x 9.991 mp Color
Ref. Ibid.

Sample 99.99% pure.
Pattern made at 26°C.

Replaces 1-0731, 2-1278

d Å	I/I_1	hkl	d Å	I/I_1	hkl
3.234	100	111			
2.800	35	200			
1.980	58	220			
1.689	64	311			
1.616	11	222			
1.400	8	400			
1.284	26	331			
1.252	17	420			
1.1432	20	422			
1.0779	19	511			
0.9900	6	440			
.9465	18	531			
.9333	8	600			
.8854	14	620			
.8540	9	533			
.8441	9	622			

1621



5-0534 MINOR CORRECTION

d	3.22	1.97	2.79	6.44	Sb_2O_3
I/I_1	100	42	40	12	Antimony III Oxide (Senarmontite)

Rad. CuKα λ 1.5405 Filter Ni
Dia. Cut off Coll.
I/I_1 G.C. Diffractometer d corr. abs.?
Ref. Swanson and Fuyat, NBS Circular 539, Vol. III (1953)

Sys. Cubic S.G. O_h^7 - Fd3m
a_o 11.152 b_o c_o A C
α β γ Z 8(Sb_4O_6)
Ref. Ibid.

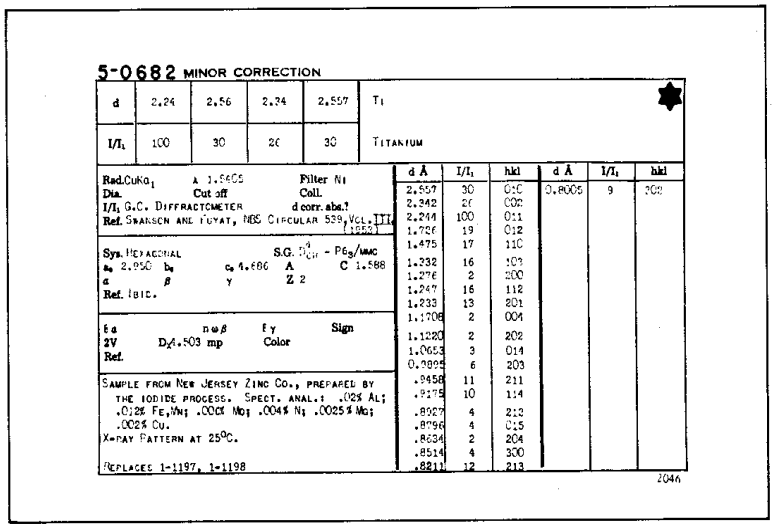
ε α nωβ εγ Sign
2V D_x 5.583 mp Color
Ref.

Sample from Mallinckrodt Chem. Works. Spect. anal.: <0.1% Pb, Si; <0.01% Ag, As, Ca, Cu, Fe, Sn, Ni; <0.001% Al, Au, Ba, Bi, Ca, Co, Na, Yl; <0.0001% Cs, In, K, Li, Mg.
X-ray pattern at 26°C.

Replaces 1-0729

d Å	I/I_1	hkl	d Å	I/I_1	hkl
6.44	12	111	1.0732	6	10.2.2
3.218	100	222	1.0402	1	953
2.788	40	400	1.0056	1	11.1.1
2.559	11	331	0.9858	2	8.8.0
2.276	2	422	.9744	3	11.3.1
2.145	3	511	.9457	1	11.3.3
1.972	42	440	.9425	5	10.6.2
1.885	2	531	.9291	3	12.0.0
1.681	35	622	.9196	<1	11.5.1
1.611	11	444	.8956	<1	11.5.3
1.562	7	711	.8817	2	12.4.0
1.452	3	731	.8734	1	9.9.1
1.394	4	800	.8527	3	13.3.1
1.363	4	733	.8505	3	10.6.6
1.279	12	662	.8406	2	12.4.4
1.247	8	840	.8335	3	13.3.1
1.224	2	911	.8151	2	13.3.3
1.1694	1	931	.7986	2	13.5.1
1.1384	5	844			
1.0783	4	951			

1937



5-0682 MINOR CORRECTION

d	2.24	2.56	2.24	2.557	Ti
I/I_1	100	30	20	30	Titanium

Rad. $CuK\alpha_1$ λ 1.5405 Filter Ni
Dia. Cut off Coll.
I/I_1 G.C. Diffractometer d corr. abs.?
Ref. Swanson and Fuyat, NBS Circular 539, Vol. III (1953)

Sys. Hexagonal S.G. D_{6h}^4 - $P6_3/mmc$
a_o 2.950 b_o c_o 4.686 A C 1.588
α β γ Z 2
Ref. Ibid.

ε α nωβ εγ Sign
2V D_x 4.503 mp Color
Ref.

Sample from New Jersey Zinc Co., prepared by the iodide process. Spect. anal.: .02% Al; .02% Fe, Mn; .002% Mo; .004% Ni; .0025% Mg; .002% Cu.
X-ray pattern at 25°C.

Replaces 1-1197, 1-1198

d Å	I/I_1	hkl	d Å	I/I_1	hkl
2.557	30	010	0.8005	9	300
2.342	20	002			
2.244	100	011			
1.726	19	012			
1.475	17	110			
1.232	16	103			
1.276	2	200			
1.247	16	112			
1.233	13	201			
1.1708	2	004			
1.1220	2	202			
1.0653	3	014			
0.9895	6	203			
.9458	11	211			
.9175	10	114			
.8927	4	213			
.8796	4	015			
.8634	2	204			
.8514	4	300			
.8211	12	213			

2046

FIG. 16-2 POWDER DATA FILE CARDS.

6-0582

d	2.27	2.58	2.24	3.93	Zr_5Si_3
I/I₁	100	80	80	30	Zirconium Silicide 5:3

Rad. CuKα λ 1.542 Filter Ni
Dia. Cut off Coll.
I/I₁ Visual d corr. abs.? ?
Ref. Schachner, Nowotny and Machenschalk, Mh. Chem. 84, 677 (1953)

Sys. Hexagonal S.G.
a_o 7.885 b_o c_o 5.558 A C 0.705
α β γ Z
Ref. Ibid.

ε α n ω β ε γ Sign
2V D mp Color
Ref.

d Å	I/I₁	hkl	d Å	I/I₁	hkl
3.93	30	110			
3.41	40	200			
3.21	50	111			
2.78	30	002			
2.59	80	210,102			
2.34	80	211			
2.27	100	300,112			
2.15	10	202			
1.97	10	220			
1.89	10	310,212			
1.85	10	221			
1.78	20	311			
1.70	30	400			
1.67	30	113			
1.61	50	222			
1.57	40	320,312			
1.50	70	321,213			
1.49	40	410			
1.455	70	402			
1.385	40	004			

6-0583

d	2.25	2.55	2.38	5.1	Ti_3Sn
I/I₁	100	80	80	20	Tin Titanium 1:3

Rad. Cu λ Filter Ni
Dia. 143.2mm Cut off Coll.
I/I₁ Visual d corr. abs.?
Ref. Pietrokowsky, J. Metals 1, 211-3 (1952)

Sys. Hexagonal S.G. D^4_{6h} - $P6_3/mmc$
a_o 5.916 b_o c_o 4.764 A C 0.805
α β γ Z 2
Ref. Ibid.

ε α n ω β ε γ Sign
2V D_x 5.194 mp Color
Ref.

Mg_3Cd type

d Å	I/I₁	hkl	d Å	I/I₁	hkl
5.1	20	100	1.014	10	214,412
3.48	50	101	0.999	50	501,403
2.95	20	110	.969	20	420
2.55	80	200	.949	80	421
2.38	80	002	.930	50b	224
2.25	100	201	.896	80b	422,205
2.16	10	102	.854	50b	215,600
1.934	10	210			
1.853	20	112			
1.792	30	211			
1.763	50	202			
1.477	50	220			
1.350	80	203			
1.289	20	400			
1.256	80	222			
1.238	50	401			
1.191	20	004			
1.129	20	402			
1.083	20	204			

9-271

d	2.24	2.53	2.35	5.06	$InTi_3$ (21 at. %In)
I/I₁	100	80	80	20	Indium Titanium

Rad. CuKα λ 1.542 Filter Ni Dia.
Cut off I/I₁ Visual
Ref. Anderko et al., Z. Metallkunde 48 57 (1957)

Sys. Hexagonal S.G.
a_o 5.84 b_o c_o 4.71 A C 0.806
α β γ Z Dx
Ref. Ibid.

Ordered structure.

d Å	I/I₁	hkl	d Å	I/I₁	hkl
5.06	20	010			
3.490	50	011			
2.928	30	110			
2.526	80	020			
2.354	80	002			
2.241	100	021			
2.160	15	012			
1.912	15	120			
1.840	20	112			
1.783	20	121			
1.726	70	022			

12-40

d	3.23	1.98	1.69	3.23	α-Na_2UF_6
I/I₁	100	80	70	100	Alpha Sodium Fluouranate (IV)

Rad. CuKα λ 1.5418 Filter Ni Dia. 9cm
Cut off 10.5A I/I₁ Visual estimate
Ref. E.V. Garner, United Kingdom Atomic Energy Authority, Capenhurst, England.

Sys Cubic S.G. Fm3m (225)
a_o 5.587 b_o c_o A C
α β γ Z Dx
Ref. Zachariasen, Acta Cryst. 2 388 (1949). (Lattice dimensions vary with composition.)

ε α n ω β ε γ Sign
2V D mp Color
Ref.

Sample prepared by fusion followed by rapid cooling of a 2:1 molar mixture of NaF and UF_4. Lattice dimensions suggest that the product contains more fluorine and less sodium than Na_2UF_6

d Å	I/I₁	hkl	d Å	I/I₁	hkl
3.23	100	111			
2.804	60	200			
1.979	80	220			
1.687	70	311			
1.615	50	222			
1.399	50	400			
1.284	60	331			
1.252	60	420			
1.142	60	422			
1.077	60	333,511			
0.989	40	440			
0.946	50	531			
0.933	40	600,442			
0.884	40	620			
0.855	20	533			
0.845	20	622			

FIG. 16-2 CONTINUED.

12-468

d	2.57	2.35	2.28	3.94	Hf_5Si_3
I/I₁	100	100	100	50	Hafnium Silicide

Rad. CuKα λ 1.542 Filter Ni Dia.
Cut off I/I₁ Visual
Ref. Nowotny et al., Monatsh. Chem. 89 701 (1958)

Sys. Hexagonal S.G. $P6_3/mcm$ (193)
a_o 7.890 b_o c_o 5.558 A C 0.705
α β γ Z 2 Dx
Ref. Ibid. (d-spacings computed from lattice parameters by Sw.)

ε α n ω β ε γ Sign
2V D mp Color
Ref.

Mn_5Si_3 type stabilized by C or B content.

d Å	I/I₁	hkl	d Å	I/I₁	hkl
3.94	50	110	1.31	30n	313,330+
3.42	60	200	1.29	30	420,204
3.22	80	111	1.28	30	331
2.78	50	002	1.23	60	510,502+
2.57	100	210,102	1.20	30	511,323
2.35	100	211	1.18	50	332,304
2.28	100	300,112			
1.97	10	220			
1.86	10	221			
1.76	20	302			
1.72	20	400			
1.68	20	113			
1.61	60	222			
1.57	40	320,312			
1.51	70	321,213			
1.49	50	410			
1.46	55	402			
1.39	30b	004			
1.37	20	500,322+			
1.35	10	223			

12-566

d	3.25	1.98	1.70	3.25	$Na_3Th_2F_{11}$
I/I₁	100	55	35	100	Sodium Fluothorate (IV)

Rad. CuKα λ 1.5418 Filter Ni Dia.
Cut off I/I₁ Counter diffractometer
Ref. Thoma et al., J. Phys. Chem. 63 1266 (1959)

Sys. S.G.
a_o b_o c_o A C
α β γ Z Dx
Ref.

ε α 1.474 n ω β ε γ Sign
2V D mp Color
Ref.

Quenched below the liquidus temperature at 40 mole % ThF_4. Decomposes on cooling into $2NaF.ThF_4$ + $NaF.ThF_4$ at 693°C.

d Å	I/I₁	hkl	d Å	I/I₁	hkl
3.25	100				
2.822	25				
1.977	55				
1.704	35				
1.413	10				

12-619

d	2.34	2.27	2.57	3.41	Ge_5Hf_5 (3 at. % C)
I/I₁	100	100	80	30	Germanium Hafnium

Rad. CrK λ 2.291 Filter Dia.
Cut off I/I₁ Visual
Ref. Parthe, Acta Cryst. 12 559 (1959)

Sys. Hexagonal S.G. $P6_3/mcm$ (193)
a_o 7.883 b_o c_o 5.537 A C 0.702
α β γ Z Dx
Ref. Ibid.

ε α n ω β ε γ Sign
2V D mp Color
Ref.

Mn_5Si_3 type

d Å	I/I₁	hkl	d Å	I/I₁	hkl
3.41	30	200	1.289	60	420
3.21	30	111	1.28	60	204,331
2.77	20	002	1.26	10B	421
2.57	80B	210,102	1.22	100B	510,502
2.34	100	211	1.219	80	214
2.27	100B	300,112	1.19	80B	511,323
1.790	10	311	1.186	80B	332
1.706	10	400	1.181	100B	304
1.67	10	113			
1.605	50	222			
1.565	10	320			
1.561	30	312			
1.50	80B	321,213			
1.489	50	410			
1.452	80	402			
1.383	50	004			
1.36	10	500,322			
1.346	1	223			
1.320	10	313			
1.31	40	330,412			

14-450

d	3.26	2.00	1.70	3.26	GaAs ★
I/I₁	100	35	35	100	Gallium Arsenide

Rad. $CuK\alpha_1$ λ 1.5405 Filter Ni Dia.
Cut off I/I₁ Diffractometer
Ref. Shirley R. Abrams, Polytechnic Inst. of Brooklyn, New York (1962)

Sys. Cubic S.G. $F\bar{4}3m$ (216)
a_o 5.6534 b_o c_o A C
α β γ Z 4 Dx
Ref. Giesecke and Pfister, Acta Cryst. 11 369 (1958)

ε α n ω β ε γ Sign
2V D mp Color
Ref.

d Å	I/I₁	hkl	d Å	I/I₁	hkl
3.26	100	111			
2.832	<1	200			
1.999	35	220			
1.704	35	311			
1.413	6	400			
1.297	8	331			
1.154	6	422			
1.088	4	333			
0.9993	2	440			
0.9556	2	531			
0.8939	4	620			
0.8622	2	533			
0.8160	4	444			
0.7916	2	711			

FIG. 16-2 CONTINUED.

15-137

d	2.24	2.55	2.37	2.55	(Ag,Ga,In)
I/I1	100	40	40	40	Gallium Indium Silver

Rad. CuKα λ 1.5418 Filter Ni Dia. 57.3mm
Cut off I/I1 visual
Ref. Campell and Reynolds, Can. J. Chem. 40 37 (1962)

Sys. S.G.
a_o b_o c_o A C
α β γ Z Dx
Ref.

ε α nωβ εγ Sign
2V D mp Color
Ref.

Cot %: 71.97 Ag, 18.03 In, 10.00 Ga
Zero assigned because unindexed. (Ed.)

d Å	I/I1	hkl	d Å	I/I1	hkl
2.55	40				
2.37	40				
2.24	100				
1.731	20				
1.469	20				
1.338	20				
1.248	20				
1.228	20				
1.186	10				
0.976	10				
.943	10				
.921	10				

15-665

d	3.24	1.99	1.71	4.58	Te_3ZrO_5 $3TeO.ZrO_2$
I/I1	100	80	80	50	Tellurium Zirconium Oxide

Rad. CuKα λ 1.5418 Filter Ni Dia. 143.2mm
Cut off I/I1 Visual
Ref. Agarwala et al., Anal. Chem. 32 729 (1960)

Sys. Tetragonal S.G. P4/mmm or P422 or P4/m
a_o 7.985 b_o c_o 5.589 A C 0.6999
α β γ Z 2 Dx 5.156
Ref. Ibid.

ε α nωβ εγ Sign
2V D 5.176 mp Color
Ref. Ibid.

d Å	I/I1	hkl	d Å	I/I1	hkl
4.575	50	101	1.512	40	511
3.241	100	201	1.439	60	521
3.000	20	211	1.395	10	004
2.817	60	201	1.374	50	530
2.645	50	102	1.355	40	114
2.516	10	221	1.336	20	413
2.209	50	320	1.317	10	204
2.056	10	321	1.296	20	214
1.994	80	400	1.279	10	611
1.934	20	410	1.266	20	620
1.866	1	003			
1.829	20	411			
1.784	10	331			
1.743	20	322			
1.707	80	421			
1.665	20	231			
1.627	20	402			
1.598	20	500,430			
1.566	10	510			
1.540	40	431,501			

16-391

d	3.23	1.98	1.69	5.73	La_6WO_{12} $3La_2O_3.WO_3$
I/I1	100	60	55	10	Lanthanum Tungstate

Rad. λ Filter Dia.
Cut off I/I1
Ref. Chang and Phillips, Inorg. Chem. 3 1792-1794 (1964)

Sys. Cubic S.G.
a_o 11.18 b_o c_o A C
α β γ Z Dx
Ref. Ibid.

ε α nωβ εγ Sign
2V D 2050° mp Color White
Ref. Ibid.

Synthetic; disordered pyrochlore structure or ordered defect fluorite structure with a_o=5.59

d Å	I/I1	hkl	d Å	I/I1	hkl
5.73	10	200			
3.23	100	222			
2.793	45	400			
2.281	4	422			
1.978	60	440			
1.685	55	622			
1.613	18	444			
1.397	10	800			
1.282	25	662			
1.249	18	840			
1.141	16	844			
1.076	14	666			
0.988	6	880			
0.945	8	10.6.2			

FIG. 16-2 CONTINUED.

TABLE 16-1 EXCERPTS FROM *INDEX* VOLUME

3.29 - 3.25

3.25	2.87	3.02	80	100	90	$(Mn,Fe,Mg,Ca)_2(PO_4)(F,OH)$	Triplite	11- 118
3.28	2.87	2.53	100	75	75	$Mn_3(PO_4)_2$	Manganese Orthophosphate	3-0465
3.28	2.86	5.08	100	55	30	$(Ba_5Pb_3)32U$	Barium Lead	14- 95
3.26	2.86	3.07	90	90	100	Th-Ca-Ce-La-U-Pb-PO-SiO	Cheralite	8- 316
3.29	2.86	2.63	100	100	100	V_3O_5	Vanadium Oxide	9- 148
*3.25	2.86	2.15	100	70	30	$4Ba0.3K_2O.3Cr_2O_3$	Barium Potassium Chromate (Pigment E)	8- 205
3.25	2.85	7.16	90	70	100	$Cu_3(SO_4)_2(OH)_2.4H_2O$	Copper Hydroxide Sulfate Hydrate	2-0107
3.26	2.85	2.65	100	80	80	$PbCuAsO_4(OH)$	Lead Copper Arsenate Hydroxide	14- 169
3.29	2.85	2.01	30	100	65	(YbAs)8F	Ytterbium Arsenide	15- 803
3.25	2.84	3.00	60	100	70	Na-Ca-Zr-Nb-Si-O-OH-F	Sodium Cal Zirconium, Niobium Silic	10- 462
3.28	2.84	2.01	25	100	65	(HoSe)8F	Holmium Selenide	15- 834
°3.28	2.84	2.01	100	60	60	AcH	Actinium Hydride	16- 470
3.28	2.84	2.01	100	100	100	Kr	Krypton	3-0466
°3.27	2.84	2.00	100	60	60	LaH	Lanthanum Hydride	16- 471
3.25	2.83	3.04	80	80	100	$CaU(PO_4)_2$	Calcium Uranium Phosphate	12- 279
3.29	2.83	2.58	100	100	100	EuI_2	Europium(II) Iodide	14- 545
3.28	2.83	1.41	100	100	100		Pharmacosiderite	13- 454
3.26	2.82	4.60	100	45	40	$ZrTe_2O_4$	Zirconium Tellurium Oxide	14- 361
*3.28	2.82	4.34	32	30	100	$RbCr(SO_4)_2.12H_2O$	Rubidium Chromium Sulfate Hydrate	7- 21
3.29	2.82	2.77	100	80	50	Sn_3O_4	Tin Oxide	16- 737
3.26	2.82	2.00	100	85	60	(PuP)8F	Plutonium Phosphide	15- 898
3.26	2.81	2.66	100	60	60	$PbSnO_3$	Lead Stannate	4-0550
3.28	2.81	2.29	70	100	80	Sr_2SiO_4	Strontium Silicate	3-0738
3.25	2.81	1.70	80	70	100	RuS_2	Ruthenium Sulfide	12- 737
3.27	2.80	3.61	80	100	90	$Al_6Ca_4O_{13}.3H_2O$	Aluminum Calcium Oxide Hydrate	16- 49
3.27	2.80	3.60	55	50	100	$Ca_4Al_6O_{13}.3H_2O$	Calcium Aluminum Oxide Hydrate	14- 464
3.25	2.80	1.99	65	100	85	$LiBiS_2$	Lithium Bismuth Sulfide	8- 408
3.27	2.79	9.87	80	80	100	$C_2Ca_2Cl_2O_4.7H_2O$	Calcium Chloride Oxalate Hydrate	14- 767
*3.26	2.78	4.61	100	90	70	$CoUO_4$	Cobalt Uranate(VI)	16- 833
3.26	2.78	1.99	100	100	80	NiI_2	Nickel Iodide	3-0473
°3.25	2.28	6.04	60	60	100	$cis\text{-}(Pt(NH_3)_2(NO_2)_2)$	cis-Dinitrodiammineplatinum(II)	16- 642
3.27	2.28	2.70	100	90	80	KNO_3 (at 115 deg C)	Potassium Nitrate (form III)	2-0991
*3.28	2.27	2.39	100	41	40	Bi	Bismuth	5-0519
3.26	2.26	1.62	55	100	70	$(Be_2Hf)3H$	Beryllium Hafnium	15- 399
3.28	2.26	1.56	100	100	100	$CaZnF_3$	Cesium Fluozincate	12- 60
*3.26	2.23	3.63	90	60	100	$RbClO_4$	Rubidium Perchlorate	15- 777
*3.27	2.23	1.71	100	96	73	MgF_2	Magnesium Fluoride	6-0290
3.29	2.19	4.62	67	67	100	$Hg(ClO_4)_2$	Mercury(II) Perchlorate	1-0314
3.29	2.19	4.60	75	75	100	$Co(NO_3)_2.6H_2O$	Cobalt(II) Nitrate Hydrate	1-0317
3.26	2.19	1.83	90	100	100	$Ca_3Al_2(Y,Ce)(SO_4)F_{13}.10H_2O$	Calcium Aluminum Fluoride Sulfate	14- 61
3.28	2.01	2.07	100	100	80	$CdGeAs_2$	Cadmium Germanium Arsenide	12- 139
*3.29	2.01	1.72	100	59	36	γ-CuBr	gamma Copper(I) Bromide	6-0292
*3.28	2.01	1.72	100	60	35	Cu_2SnSe_4	Copper(II) Tin(IV) Selenide	16- 670
3.28	2.01	1.71	100	80	80	PuOF	Plutonium Oxy Fluoride	6-0291
3.28	2.01	1.71	100	60	60	PuOF	Plutonium Fluoride Oxide	6-0293
*3.27	2.00	1.71	100	70	44	ZnSe	Zinc Selenide	5-0522
*3.27	2.00	1.71	100	57	39	Ge	Germanium	4-0545
3.26	2.00	1.70	100	100	100	$TiBr_4$	Titanium (IV) Bromide	11- 64
*3.26	2.00	1.70	100	35	35	GaAs	Gallium Arsenide	14- 450
3.27	1.99	1.70	100	80	80	Bi_4MoO_9	Bismuth Molybdenum Oxide	12- 149
3.25	1.99	1.41	40	100	100	Fe_3Si	Iron Silicide	11- 616
3.25	1.98	1.70	100	55	35	$Na_3Th_2F_{11}$	Sodium Fluothorate(IV)	12- 566
3.28	1.96	8.40	100	89	66		Chromium Borate	1-0691
3.27	1.96	2.82	70	60	100	$Ag_4PbBi_4S_9$	Silver Lead Bismuth Sulfide	11- 63
3.25	1.92	5.50	100	50	100	CuSCN	Copper(I) Thiocyanate	1-0206
3.29	1.90	2.33	100	100	60	Rb_3ZrF_7	Rubidium Fluozirconate	10- 133
*3.28	1.89	2.56	40	25	100	$ErSi_2$ (hexagonal)	Erbium Silicide	12- 37
3.27	1.89	1.74	100	80	80	$Pb_8Al_2Si_4O_{19}$	Lead Aluminum Silicate	3-0469
*3.27	1.83	2.63	95	65	100	YBO_3	Yttrium Borate	16- 277
3.27	1.81	3.16	100	100	90	$Ca_4Pb_6Si_6O_{21}Cl_2$	Calcium Lead Chloride Silicate	14- 328
3.27	1.81	1.25	80	100	100	$SbBiO_4$	Antimony Bismuth Oxide	7- 191
*3.29	1.76	2.94	65	42	100	$NaClO_3$	Sodium Chlorate	5-0610
3.27	1.71	2.00	100	70	65	δ-Bi_2O_3 (at 750 deg C)	delta Bismuth Oxide	16- 654
3.26	1.70	2.94	100	90	80	$Fe(II)(Nb,Ta)_2Ti_1O_2$	Iron Niob., Tant. Titan. Oxide	11- 397
3.28	1.70	2.51	100	100	90	$Fe_3(Nb,Ta)_2Ti_1O_2$	Niobian Rutile (Ilmenorutile)	16- 934
*3.25	1.69	2.49	100	50	41	TiO_2	Titanium(IV) Oxide	4-0551
*3.29	1.69	2.05	100	55	45	$Pb(IO_3)_2$	Lead Iodate	11- 85
3.26	1.65	2.05	80	70	100	β-$FeTe_{0.9}$	beta Iron Tellurium	7- 140
3.28	1.56	2.84	100	100	80	$K_4V_{10}O_{27}$	Potassium Vanadate	12- 677
3.26	1.30	2.04	100	100	75	$Cs(PuO_2)_2F_5.3H_2O$	Cesium Plutonyl Fluoride Hydrate	15- 55

TABLE 16-1 EXCERPTS FROM *INDEX* VOLUME (CONTINUED)

3.24 - 3.20

3.22	2.88	2.15	100	60	25	α-$Ba_2ZrAs_2O_9$	alpha Barium Zirconium(IV) Arsenate	16- 255
3.23	2.87	2.15	80	100	80	β-6PbO.$PbCl_2$	beta Lead Chloride Oxide	6-0435
3.23	2.87	1.88	40	100	80	$Ca(OCl)_2$	Calcium Hypochlorite	3-0702
3.23	2.86	2.27	100	70	50	La	Lanthanum	1-0718
3.23	2.85	3.28	90	50	100	$PbUO_4$	Lead(IV) Uranate(IV)	13- 98
3.20	2.85	2.76	100	60	60	$Ag(Bi_{0.9}Sb_{0.1})S_2$	Silver Antimony Bismuth Sulfide	11- 352
3.23	2.84	1.97	100	67	27	$3(Sr.K_2)CrO_4.2H_2O$	Strontium, Potassium Chromate Hydrate	1-0719
3.21	2.84	1.85	100	55	40	(Zn,Cu,Si)S (compd. B)	Copper Silicon Zinc Sulfide	14- 542
3.24	2.83	3.42	40	100	40	SnS	Tin (II) Sulfide	1-0984
3.24	2.82	1.98	70	100	80	α-$AgBiS_2$	alpha Silver Bismuth Sulfide	4-0699
3.22	2.82	1.86	100	83	66	Al_2S_3	Aluminum Sulfide	1-0726
3.21	2.81	3.59	95	75	100	$Ba_2MgP_4O_{13}$	Barium Magnesium Phosphate	16- 640
3.23	2.81	3.04	35	25	100	$Eu._{48}Sm._{52}PO_4$	Europium, Samarium Phosphate	12- 430
3.20	2.80	7.00	100	80	100	$Ag_4(PO_2NH)_4$	Silver Tetrametaphosphimate	14- 226
3.21	2.80	2.98	71	100	86	$Na_2S.9H_2O$	Sodium Sulfide Hydrate	3-0745
3.22	2.80	2.94	61	100	71	PbO.$PbBr_2$ (L form)	Lead Bromide Oxide	6-0451
3.23	2.80	1.98	100	50	40	$PbUO_4$	Lead(IV) Uranate(IV)	13- 99
3.21	2.80	1.97	100	30	20	Ca	Calcium	1-0735
3.20	2.80	1.49	100	100	100	Ca-Ce-Nd-La-Pr-Si-O-F	Cefluosil syn	15- 202
3.22	2.79	4.58	100	35	30	$SnTe_3O_8$	Tin(IV) Tellurium Oxide	15- 698
3.20	2.35	2.03	70	60	100	Bi_2Te_3	Bismuth Tellurium	10- 54
3.20	2.30	2.21	100	50	45	Ca_3P_2	Calcium Phosphide	16- 730
3.24	2.29	1.87	100	70	25	(LaSb)8F	Antimony Lanthanum	15- 893
3.24	2.29	1.45	100	70	30	(BiTe)8F	Bismuth Telluride	15- 820
3.23	2.28	1.95	100	80	80	(Cu,Ag)I	Copper Silver Iodide	2-0499
3.23	2.28	1.44	100	80	50	PbTe	Lead Telluride	8- 28
3.23	2.28	1.44	100	70	30	(BiPr)8F	Bismuth Praseodymium	15- 879
3.21	2.27	1.85	100	70	25	(CeSb)8F	Antimony Cerium	15- 821
*3.21	2.27	1.61	100	40	18	LaTe	Lanthanum Telluride	16- 569
3.21	2.27	1.44	100	70	30	(BiNd)8F	Bismuth Neodymium	15- 878
3.21	2.27	1.31	60	100	80	RhSc	Rhodium Scandium	11- 490
3.20	2.26	3.70	80	80	100	AsH_3	Arsenic(III) Hydride	2-0334
3.20	2.26	3.69	100	90	100	$RbNH_2$ (at 50 deg C)	Rubidium Amide	12- 748
3.24	2.26	2.83	80	80	100	$TiBr_2$	Titanium(II) Bromide	14- 642
3.21	2.26	1.74	70	100	100	$(NH_4)_3VF_6$	Ammonium Fluovanadate(III)	2-1136
3.20	2.26	1.73	70	100	100	$(NH_4)_3CrF_6$	Ammonium Fluochromate(III)	2-1137
3.24	2.24	2.90	80	80	100	VBr_2	Vanadium (II) Bromide	12- 80
3.21	2.22	2.26	70	70	100	$Zn_5(Zn_{0.4}Th_{0.6})$	Zinc Thorium compound	6-0580
3.24	2.21	2.45	80	40	100	Mg_2C_3	Magnesium Carbide	1-1138
*3.22	2.01	3.02	100	30	25	$AgIO_4$	Silver (meta) Periodate(VII)	10- 368
*3.22	2.01	1.65	100	30	27	$SrWO_4$	Strontium Tungstate	8- 490
*3.22	2.01	1.64	100	30	25	$SrMoO_4$	Strontium Molybdate	8- 482
°3.20	2.00	3.25	80	100	80		Copper Selenide	14- 479
*3.20	2.00	2.05	100	52	44	CeF_3	Cerium(III) Fluoride	8- 45
*3.21	1.99	3.30	95	70	100	α-$Ca_2P_2O_7$	alpha Calcium Pyrophosphate	9- 345
3.24	1.99	1.71	100	80	80	Te_3ZrO_5	Tellurium Zirconium Oxide	15- 665
3.24	1.98	2.83	80	60	100	CrI_3	Chromium(III) Iodide	6-0446
3.23	1.98	1.69	100	60	55	La_6WO_{12}	Lanthanum Tungstate	16- 391
3.23	1.98	1.69	100	80	70	α-Na_2UF_6	alpha Sodium Fluouranate(IV)	12- 40
*3.22	1.97	2.79	100	42	40	Sb_2O_3	Antimony(III) Oxide	5-0534
3.24	1.97	1.88	100	100	100	$K_2PuO_2F_4$	Potassium Plutonyl Fluoride	15- 60
°3.22	1.97	1.68	100	60	60	$SbAsO_4$	Antimony(III) Arsenate	1-0728
3.20	1.96	4.96	100	80	100	Na_3UF_7	Sodium Fluouranate (IV)	12- 49
*3.24	1.96	3.49	100	65	60	GdF_3	Gadolinium Fluoride	12- 788
3.24	1.96	2.99	100	100	90	EuF_3 (Ortho.)	Europium(III) Fluoride	5-0535
*3.20	1.96	2.77	100	45	45	HgF_2	Mercury(II) Fluoride	15- 754
3.21	1.96	1.67	100	100	100	$AsSb_3$	Antimony Arsenide	2-1449
3.21	1.95	3.40	80	80	100	CuCl (high temp.)	Copper(I) Chloride	9- 17
3.21	1.95	1.71	100	100	100	$HgAl_2Se_4$	Mercury Aluminum Selenide	8- 271
3.22	1.94	2.82	40	30	100	$Ag(Sb,Bi)S_2$	Silver Antimony,Bismuth Sulfide	4-0696
3.21	1.74	1.20	50	45	100	$Be_{12}GeO_{20}$	Beryllium Germanium Oxide	16- 797
3.23	1.71	2.82	60	50	100	$Ag(Bi_{0.3}Sb_{0.7})S_2$	Silver Antimony Bismuth Sulfide	12- 135
3.23	1.69	2.48	100	90	80	Fe(II)(Nb,Ta)$_2TiO_2$	Iron Niob., Tant. Titan. Oxide	11- 396
°3.24	1.69	1.98	90	90	100	β-MnS	beta Manganese Sulfide	3-1065
*3.23	1.69	1.98	100	64	58	ThO_2	Thorium Oxide	4-0556
3.22	1.68	2.68	60	100	75	$Fe_2(TiO_3)_3$	Iron Titanate	13- 326
3.21	1.66	3.31	60	40	100	$(Fe,Mn)Fe_2(PO_4)_2(OH)_2$	Iron,Manganese Hydrox Phosphate	14- 310
3.20	1.66	2.43	100	70	40	$CrVO_4$	Chromium(III) Vanadate	15- 296
3.24	1.66	2.02	100	90	80	$PbWO_4$	Lead Tungstate	8- 108
3.20	1.63	6.19	90	90	100	$H_2(UO_2)(MoO_4)_2.3H_2O$	Uranyl Hydrogen Molybdate Hyd	13- 27

TABLE 16-1 EXCERPTS FROM *INDEX* VOLUME (CONTINUED)

2.29 - 2.25

*2.28	2.67	6.18	36	27	100	WS_2	Tungsten Sulfide	8- 237
*2.27	2.66	2.74	40	100	60	Ag_2CO_3	Silver Carbonate	12- 766
2.27	2.66	2.18	100	35	30	$PrRh_2$	Praseodymium Rhodium	12- 332
2.28	2.66	2.18	100	35	35	$NdRh_2$	Neodymium Rhodium	12- 319
2.27	2.65	2.17	100	35	35	$CeRh_2$	Cerium Rhodium	12- 330
2.27	2.64	3.02	40	100	40	$DyGe_2$	Dysprosium Germanium	13- 223
2.26	2.64	2.16	100	35	30	$CeRu_2$	Cerium Ruthenium	12- 374
2.25	2.63	2.45	100	80	100	$(Al_2Zr)12H$	Aluminum Zirconium	13- 510
*2.27	2.62	1.60	25	100	30	BP	Boron Phosphide	11- 119
2.28	2.59	2.40	100	60	60	epsilon' Ag-Sb (74 pc Ag)	epsilon' Antimony Silver	2-1123
2.26	2.58	3.41	80	75	100	YbI_2	Ytterbium(II) Iodide	16- 68
2.27	2.58	2.34	100	80	80	Zr_5Si_3	Zirconium Silicide 5:3	6-0582
2.28	2.57	2.35	100	100	100	Hf_5Si_3	Hafnium Silicide	12- 468
2.27	2.57	2.34	100	80	100	Ge_3Hf_5 (3 at. pc C)	Germanium Hafnium	12- 619
2.25	2.55	2.38	100	80	80	Ti_3Sn	Tin Titanium 1:3	6-0583
2.26	2.54	3.47	50	100	65	Ti_2B_5	Titanium Boride 2:5	6-0528
°2.25	2.54	2.39	80	40	100	AgGa (79.2 wt pc Ag)	Gallium Silver	15- 161
*2.28	2.52	2.56	60	100	70	U	Uranium	11- 628
2.25	2.52	1.46	100	80	80	$(Au_{0.75}Cd_{0.035}In_{0.215})O$	Cadmium Gold Indium	16- 428
2.28	2.51	2.54	50	100	90	Cd_2Ca	Cadmium Calcium 2:1	6-0530
2.26	2.39	2.33	100	100	100	$AuHg_2$ (65.8 pc Hg)	Gold Mercury	4-0780
2.28	2.39	1.76	100	80	80	γ-Ag_3Sn	gamma Silver Tin	4-0800
2.29	2.38	2.36	80	100	80	$(Ge_3Sc_5)16H$	Germanium Scandium	15- 187
*2.28	2.37	2.60	100	30	20	Mo_2C	Molybdenum Carbide	11- 680
2.26	2.36	2.77	30	100	40	$(Al_2Y)24F$	Aluminum Yttrium	16- 84
2.28	2.35	2.69	60	100	60	$(GdSe_2)12O$	Gadolinium Selenide	14- 610
2.27	2.35	2.34	60	100	100	$(Sc_5Si_3)16H$	Scandium Silicide	15- 189
2.28	2.35	2.34	100	50	45	$(SbTi_3)32U$	Antimony Titanium	15- 303
2.29	2.34	2.24	80	100	100	(Ti,Co,Si)O	Cobalt Titanium Silicide	15- 727
2.27	2.34	2.13	75	100	100	(Re-Ta)30T	Rhenium Tantalum	15- 89
2.27	2.33	2.24	90	40	100	$(Ti_5Ga_4)18H$	Gallium Titanium	16- 139
2.26	2.33	2.00	70	100	90	Ge_2Nb	Germanium Niobium	9- 199
2.28	2.31	2.90	80	100	80	(CaGe) 8Q	Calcium Germanium	13- 132
2.25	2.31	2.87	70	100	80	AgTe (low temp.)	Silver Telluride	12- 695
2.25	2.31	2.10	75	100	100	(Re-Mo)30T	Rhenium Molybdenum	15- 116
2.25	2.30	2.50	100	90	50	$NdOs_2$	Neodymium Osmium	12- 314
2.25	2.30	2.10	75	100	100	(Re-W)30T	Rhenium Tungsten	15- 115
2.25	2.29	2.48	100	95	45	Re_2Y	Rhenium Yttrium	12- 312
2.25	2.28	2.27	60	100	100	Au_3Be	Beryllium Gold	11- 472
°2.27	2.28	2.07	80	50	100	$VO_{0.53}$	Vanadium Oxide	15- 455

2.24 - 2.20

2.20	2.66	6.20	80	75	100	$(Mg_{0.88}Mn_{0.12})BO_3H$	acid Magnesium, Manganese Borate	12- 179
*2.23	2.64	3.06	80	75	100	$Mg_2SiO_4.MgF_2$	Magnesium Fluoride Silicate	11- 686
2.23	2.63	1.43	100	80	80	Zn_2Zr	Zinc Zirconium	7- 97
2.23	2.62	1.42	100	65	35	$(GdFe_2)24F$	Gadolinium Iron	15- 424
*2.23	2.60	2.95	20	100	20	YSi_2	Yttrium Silicide (orthorhombic form)	11- 324
2.23	2.60	2.53	30	20	100	$Na_2CS_3.2H_2O$	Sodium Thiocarbonate Hydrate	14- 605
2.22	2.60	1.50	100	90	80	$(HfV_2)24F$	Hafnium Vanadium	15- 190
*2.22	2.57	1.57	100	62	58	MnO	Manganese(II) Oxide	7- 230
2.24	2.56	2.40	80	100	100	$(Zr_3Si_2)10T$	Zirconium Silicide	14- 368
*2.24	2.56	2.34	100	30	26	Ti	Titanium	5-0682
°2.24	2.55	2.37	100	40	40	(Ag,Ga,In)	Gallium Indium Silver	15- 137
2.21	2.55	2.28	100	80	100	Pd_4S	Palladium Sulfide	10- 335
2.23	2.54	2.30	100	70	70	Pd_2Te	Palladium Telluride	11- 450
2.20	2.54	2.29	100	80	100	$Ge_3Nb_5(Ge_{0.67}Nb)$	Germanium Niobium	8- 199
*2.22	2.53	2.72	70	57	100	$MgTiO_3$	Magnesium Titanate	6-0494
2.21	2.53	2.38	80	100	100	$(Hf_3Si_2)10T$	Hafnium Silicide	14- 427
2.24	2.53	2.35	100	80	80	$InTi_3$ (21 at. pc In)	Indium Titanium	9- 271
2.23	2.52	1.84	50	100	80	(ZrP)8H	Zirconium Phosphide	16- 34
2.21	2.51	2.85	70	100	70	Nb_5Si_3	Niobium Silicon	9- 222
2.20	2.50	1.33	100	50	35	TiH_2	Titanium Hydride	9- 371
*2.23	2.39	2.11	34	32	100	Re	Rhenium	5-0702
2.21	2.39	1.34	100	80	70	Au_2Mn	Gold Manganese	11- 572
°2.20	2.37	2.33	100	80	80	Pd_5As_2	Palladium Arsenide	16- 58
2.20	2.37	1.33	100	90	90	γ-Mo_2B	gamma Molybdenum Boride	6-0593
2.24	2.36	2.34	60	100	100	β Au-Cd(50.1 at. pc Cd)	β-Cadmium Gold	5-0675
2.24	2.36	2.34	100	80	100	(Nb,Co,Si)O	Cobalt Niobium Silicide	15- 719
2.21	2.36	2.25	80	100	100	(Nb,Ni,Si)O	Nickel Niobium Silicide	15- 722
2.20	2.36	2.24	100	100	100	(Ta,Fe,Si)O	Iron Tantalum Silicide	15- 715
2.24	2.34	2.74	30	100	70	$GdMn_2$	Gadolinium Manganese	10- 240
2.21	2.32	1.70	100	70	60	$AlTi_2$ (25 pc Al)	Aluminum Titanium	9- 98

worksheet 16–1

NAME: _____

2θ, deg	d	I	d	I
27.6	3.23 Å	100		
31.8				
35.0				
38.3				
40.1				
45.8				
53.0				
54.4				
57.0				
62.9				
66.8				
70.5				
73.7				
76.0				
77.4				
84.9		10		
91.3		10		
102.4		10		
109.2		10		
111.4		10		
121.0		10		
129.0		10		
131.8		10		
145.5		10		
158.8		10		

worksheet 16–2

NAME: _____

d_1	d_2	d_3	I_1	I_2	I_3	*Card No.*
3.23	2.81	—	100	38	—	unknown
3.28	2.83	1.41	100	100	100	13–454
Others even less likely						
3.23	2.25	—	100	35	—	
3.23	1.98	—	100	52	—	
3.23	1.69	—	100	48	—	