

Supporting Information

Designing and Teaching a Course about Characterization Techniques for Solid State Materials in an Undergraduate Institution

Alexandre H. Pinto*

Ithaca College, Department of Chemistry, Center for Natural Sciences,

953 Danby Road, Ithaca, NY 14850, USA.

apinto2@ithaca.edu

TABLE OF CONTENTS:

Course Syllabus for CHEM 38106 01 – Selected Topics in Chemistry: Modern Techniques for Solid State Materials Characterization	2
List of Youtube Videos Used during the Course	5
List of Books Used for Class Preparation	6
Sample Homework Assignment Homework Week 02 for CHEM 38106 01 – Fall 2017	9
Details about the survey of materials chemistry related terms (Figure 1)	12

Course Syllabus for CHEM 38106 01 – Selected Topics in Chemistry: Modern Techniques for Solid State Materials Characterization

Credit Number: 1.5

Instructor: Alex Pinto, PhD

Email: apinto2@ithaca.edu

(Some sections of the syllabus were edited for publication purposes)

Course Description:

The goal of this course is to introduce students to techniques frequently used in solid state materials characterization but generally not taught in a conventional Instrumental Analysis course. The course will primarily focus on spectroscopy and electron microscopy techniques. Some techniques that will be studied during the course are: Powder X-Ray Diffraction, Infrared Spectroscopy, Raman Spectroscopy, Scanning Electron Microscopy, Transmission Electron Microscopy, and Techniques for Elemental Analysis. For each technique, the students will be introduced to the physical principles underlying the technique, study real examples present in the current scientific literature, and discuss how the technique could be applied to other experimental situations involving metallic, inorganic, and polymeric materials. By the end of the course, students are expected to have acquired a deep understanding of the physical principles, capabilities, and limitations of each technique.

Student Learning Outcomes:

During this course students are expected to:

- Properly describe the physical phenomenon underlying each Characterization Technique.
- Describe the potential and limitation associated with each Characterization Technique.
- Read, analyze and issue some opinion about how these Characterization Techniques have been applied in the current scientific literature.

- Apply the knowledge acquired during the study of the techniques to simple situations involving characterization of materials.

Required Course Materials:

It is not simple to find a single textbook covering all the techniques we will be studying during the course. To prepare the classes, Instructor will be consulting multiple references, so you are not required to buy any textbook. Most of times the literature will be provided by the Instructor and distributed to the students. The means used to distribute that to the students will be explained during the first day of class.

Course Assessment:

Homework Assignment (HW): Every week there will be a homework assignment. This homework assignment can be either an application of a certain technique, analysis of a literature paper, or a combination of both components. In general, students will have one-week to complete them. Although, it can vary depending on the situation. (60% of the Final Grade)

Final Paper (FP): The Final Paper will be based on data present in scientific literature. The Instructor will provide a data set about a certain material alongside some background information regarding the experimental procedure used to prepare this material. Then, students will write a scientific paper based on the data set and pieces of information provided by the Instructor. The scientific paper must contain the sections Abstract, Introduction, Experimental Procedure (most likely provided by the Instructor), Results & Discussions, Conclusions and References, and it should be typed into a ACS Chemistry of Materials journal template. The final paper needs to be initially submitted on the week from December 11th – 15th (the right due date will be informed later in the course). Then, Instructor will read like he was an actual journal referee and return to the students with one of the outcomes: Accepted as is, Major Revision, or Minor Revision.

If the outcome is Accepted as is, student does not need to change anything, and it will be granted the highest possible grade for their work. If the outcome is either Major or Minor Revision, students have two options: Accept the partial grade they received based on the initial quality of the paper, or do all the modification suggested by the referee, and re-submit the paper on the week of December 18th – 22th, and if all the revisions were properly addressed, it is likely the student will get the highest grade possible for their work. (40% of the Final Grade)

Tentative Course Schedule:

	Tuesday	Thursday	
Week 1	24-Oct	26-Oct	Introduction to Solid State Chemistry
Week 2	31-Oct	2-Nov	Powder X-Ray Diffraction
Week 3	7-Nov	9-Nov	Infrared and Raman Spectroscopy
Week 4	14-Nov	16-Nov	Scanning Electron Microscopy
Week 5	28-Nov	30-Nov	Transmission Electron Microscopy
Week 6	5-Dec	7-Dec	Elemental Analysis Techniques ICP-OES, ICP-MS, X-ray Fluorescence, EDS, EELS
Week 7	12-Dec	14-Dec	Final Papers Initial Submission
Week 8	Final Papers Due		

List of Youtube Videos Used during the Course:

UNIT ABOUT INFRARED AND RAMAN SPECTROSCOPIES:

Video Title: Confocal Raman Microscopy by MVA Scientific Consultants

Channel: mvascientific

Posted in: Oct 17th, 2010

Video URL: <https://www.youtube.com/watch?v=v4lHUPwkEs0>

UNIT ABOUT SCANNING ELECTRON MICROSCOPY:

Video Title: Salt and Sugar under Scanning Electron Microscope

Channel: Captain Corrosion

Posted in: Feb 28th, 2017

Video URL: https://www.youtube.com/watch?v=YamiDCNX_Z4

UNIT ABOUT TRANSMISSION ELECTRON MICROSCOPY:

Video Title: Au nanoparticle sintering (video 2)

Channel: Gatan, Inc

Posted in: May 23rd, 2015

Video URL: <https://www.youtube.com/watch?v=H5ohOFtJ3Yg>

Video Title: Preparing for TEM

Channel: Gonzaga Biology

Posted in: July 16th, 2012

Video URL: <https://www.youtube.com/watch?v=F0ZNUykXovk>

List of Books Used for Class Preparation:

UNIT ABOUT INTRODUCTION TO SOLID STATE CHEMISTRY:

CALLISTER, W. D. - Materials Science and Engineering: an Introduction. 7th Edition. John Wiley & Sons, Inc., New York, USA, (2007).

Chapters:

2 - Atomic Structure and Interatomic Bonding

3 - The Structure of Crystalline Solids

UNIT ABOUT POWDER X-RAY DIFFRACTION:

BRANDON, D. and KAPLAN, W. D. – Microstructural Characterization of Materials. 1st Edition. John Wiley & Sons, Inc., Chichester, England, (1999).

Chapters:

1 – The Concept of Microstructure

2 – Diffraction Analysis of Crystal Structure

SURYANARAYANA, C. and GRANT NORTON, M. – X-Ray Diffraction A Practical Approach. 1st Edition. Plenum Press, New York, USA, (1998).

Chapters:

1 – X-Rays and Diffraction

2 – Lattices and Crystal Structures

Module 1 – Crystal Structure Determination I: Cubic Structures

DA RÓZ, A. L.; FERREIRA, M.; LEITE, F. L.; and OLIVEIRA Jr, O. N. (Editors) – Nanocharacterization Techniques. 1st Edition. Elsevier-William Andrew Applied Science Publishers, Cambridge, USA, (2017)

Chapter:

5 - X-Ray Diffraction and Scattering by Nanomaterials

UNIT ABOUT INFRARED AND RAMAN SPECTROSCOPIES:

SARDELA, M. (Editor) – Practical Materials Characterization. 1st Edition. Springer, New York, USA, (2014).

Chapter:

2.4 - Fourier Transform Infra-Red Spectroscopy (FTIR)

NAKAMOTO, K. – Infrared and Raman Spectra of Inorganic and Coordination Compounds. 4th Edition. John Wiley & Sons, New York, USA, (1986).

Chapter:

I-1 – Origin of Molecular Spectra

I-2 – Vibration of Diatomic Molecule

UNITS ABOUT SCANNING ELECTRON MICROSCOPY AND TRANSMISSION ELECTRON MICROSCOPY:

WILLIAMS, D. B. and BARRY CARTER, C. – Transmission Electron Microscopy. 2nd Edition. Springer, New York, USA, (2009).

Chapters:

1 – The Transmission Electron Microscope

2 – Scattering and Diffraction

5 – Electron Sources

6 – Lenses, Apertures, and Resolution

10 – Specimen Preparation

11 – Diffraction in TEM

23 – Phase Contrast Images

UNITS ABOUT SCANNING ELECTRON MICROSCOPY AND TRANSMISSION ELECTRON MICROSCOPY:

WILLIAMS, D. B. and BARRY CARTER, C. – Transmission Electron Microscopy. 2nd Edition. Springer, New York, USA, (2009).

Chapters:

32 – X-ray Spectrometry

37 – Electron Energy-Loss Spectrometers and Filters

UNITS ABOUT ELEMENTAL ANALYSIS TECHNIQUES:

BRUNDLE, C. R.; EVANS JR, C. A.; and WILSON, S. – Encyclopedia of Materials Characterization – Surfaces, Interfaces, Thin Films. 1st Edition. Butterworth-Heinemann, Stoneham, USA, (1992).

Chapters:

6 – X-ray Emission Techniques

Sample Homework Assignment

Homework Week 02 for CHEM 38106 01 – Fall 2017

1) Derive the rule of systematic absences for a face centered cubic (fcc) lattice. (10 points)

2) Suppose that you work for a chemical manufacturing company and your supervisor just received a XRD result from a raw material provider. This XRD result is a XRD pattern from a mixture of two different metal oxides minerals. The provider guaranteed to your supervisor the two oxides can be any two of those: MnO_2 , CeO_2 , TiO_2 , V_2O_5 , or Al_2O_3 . Your job is to identify which ones are the two oxides present in this XRD pattern. (30 points total, 15 for each material correctly identified)

To do that follow the instructions:

- You should use a software called Jade 9, located in CNS 385 (XRD room) it is installed in the computer on the left side of the powder XRD equipment (the white, smaller diffractometer in the room).
- There is an instruction file posted alongside this homework sheet, follow the instructions present there about how to use the software.
- In case you never used the software before, and do not feel confident using by yourself just following the instructions, schedule a time with Alex to go with you.
- Each student will analyze a different pattern, the order of the patterns was assigned according to the surname order in the attendance sheet for the class. Then, to clarify the patterns are:

Student	Pattern
Student 1	Material 1
Student 2	Material 2
Student 3	Material 3
Student 4	Material 4
Student 5	Material 5
Student 6	Material 6

3) After having completed the task above, your supervisor assigned you a new one. He also received some XRD results about an Iron (Fe) sample, and he asks you to check if the lattice parameter of this iron sample matches what is expected by theory. So, first you have to:

a) Calculate the theoretical lattice parameter for Fe, knowing that Fe has a BCC, and an atomic radius equal to 0.126 nm. (Here you should calculate in the way we learned in the first week, using the hard sphere models, like you did for some problems in the first homework) (5 points)

And second:

b) Calculate the Iron lattice parameters using the spreadsheet method we learned in the last class. The results handed to you by your supervisor show the following list of 2 theta peaks for Fe. (20 points)

Peak	2 θ
1	44.73
2	65.07
3	82.40
4	99.00
5	116.45
6	137.24

The data was collected in diffractometer using CuK α radiation source, so the wavelength of the incident X-rays is 0.1540 nm. (On this point, you should submit your Excel spreadsheet by email to Alex, in the same day you submit the hard copy of your homework). As a guide, the example we solved on class is posted on Sakai, completely solved.

c) Compare the results obtained in part a) and b), in such a way you can convince your supervisor that you are right. (5 points)

4) One of your coworkers is in trouble, because he received a XRD pattern from a provider, but the data sent by the provider was obtained on a powder X-ray diffractometer using CoK α radiation source ($\lambda = 0.1789$ nm), and the database you have available in your job only contain data for patterns collected using CuK α radiation source ($\lambda = 0.1540$ nm). So, the peaks always will be shifted in relation your database.

In order to keep your coworker's job, explain and derive to him some equation where he can relate the 2 θ (or θ) angle for CoK α radiation source with the 2 θ (or θ) angle for CuK α radiation source. (15 points)

(Hint: To solve this problem you only need Bragg's Law, and the fact that d-spacing does not change, regardless the radiation source used).

5) Find some paper where powder XRD was used, and answer the following questions: (15 points)

- a) What are the materials analyzed in this paper?
- b) Which information would authors like to obtain from the powder XRD?
- c) Did the authors successfully reach their goal?
- d) What do you like most and least about this paper you chose and read?

Details about the survey of materials chemistry related terms (Figure 1)

To obtain the results shown in Figure 1 a), the survey was carried out by looking for the terms: X-ray diffraction, Raman spectroscopy, electron microscopy, nanoscience or nanotechnology, and solid state chemistry. This search was performed in the detailed course descriptions present either in the graduate program website or in the graduate academic catalog.

Correlate terms, for instance, nanochemistry, nanomaterials, Raman spectrometry were also computed in the survey, under its pertaining category.

A total of 20 Graduate Programs in Chemistry offering the concentration in Materials Chemistry in the USA were consulted in this survey. The list of the Programs consulted is presented in the following table:

Counting	Institution	State
1	California Institute of Technology	CA
2	Carnegie Mellon University	PA
3	Colorado School of Mines	CO
4	Colorado State University	CO
5	Northwestern University	IL
6	Old Dominion University	VA
7	Oregon State University	OR
8	Princeton University	NJ
9	Purdue University	IN
10	University of California Los Angeles	CA
11	University of California Santa Barbara	CA
12	University of Chicago	IL
13	University of Illinois at Urbana-Champaign	IL
14	University of Maine	ME
15	University of Michigan	MI
16	University of North Carolina at Chapel Hill	NC
17	University of Rochester	NY
18	University of South Dakota	SD
19	University of Tulsa	OK
20	University of Wisconsin - Madison	WI

To obtain the results shown in Figure 1 b), the survey was carried out by looking for the terms: X-ray diffraction, Raman spectroscopy, electron microscopy, nanoscience or nanotechnology, and solid state chemistry. This search was performed in the detailed course descriptions present either in the undergraduate program website or in the graduate academic catalog.

Correlate terms, for instance, nanochemistry, nanomaterials, Raman spectrometry were also computed in the survey, under its pertaining category.

A total of 173 Bachelor's in Science (BS) in Chemistry from primarily undergraduate institution in the USA were consulted. From these 173 undergraduate programs, 86 were offered by public institutions and 87 were offered by private institutions. Institutions of 45 different USA states were consulted, in order to provide a nationally representative dataset.

As primarily undergraduate institutions were considered those institutions that do not offer a PhD in Chemistry as terminal degree. Most institutions surveyed offered a BS in Chemistry as terminal degree and a minority of them also offer a Master in Chemistry as terminal degree. The institutions were chosen according their position in the state ranking present in the website www.bestcolleges.com. In some occasions, it was necessary to consult the Wikipedia entry "List of Colleges and Universities in State Name" to find additional institutions.

It was initially aimed to choose four institutions for each contiguous USA state, being two public and two private institutions. For the states it was not possible, the reason of this impossibility is explained after presentation of the following table.

Counting	Institution	Location	State	Public or Private
1	Judson College	Marion	AL	Private
2	Oakwood University	Huntsville	AL	Private
3	University of North Alabama	Florence	AL	Public
4	Jacksonville State University	Jacksonville	AL	Public
5	Harding University	Searcy	AR	Private
6	Hendrix College	Conway	AR	Private
7	Arkansas State University	Jonesboro	AR	Public
8	Henderson State University	Arkadelphia	AR	Public
9	Harvey Mudd College	Claremont	CA	Private
10	Azusa Pacific University	Azusa	CA	Private
11	California State University San Marcos	San Marcos	CA	Public

12	California State University Long Beach	Long Beach	CA	Public
13	Colorado College	Colorado Springs	CO	Private
14	Regis College	Denver Springs	CO	Private
15	Adam State University	Alamosa	CO	Public
16	Metropolitan State University of Denver	Denver	CO	Public
17	Connecticut College	New London	CT	Private
18	University of New Haven	New Haven	CT	Private
19	Southern Connecticut State University	New Haven	CT	Public
20	Western Connecticut State University	Danbury	CT	Public
21	Delaware State University	Dover	DE	Public
22	Rollins College	Winter Park	FL	Private
23	Nova Southeastern University	Fort Lauderdale	FL	Private
24	University of North Florida	Jacksonville	FL	Public
25	University of West Florida	Pensacola	FL	Public
26	Oglethorpe University	Atlanta	GA	Private
27	Mercer University	Macon	GA	Private
28	Georgia Southern University	Statesboro	GA	Public
29	Kennesaw State University	Kennesaw	GA	Public
30	Brigham Young University	Rexburg	ID	Private
31	College of Idaho	Caldwell	ID	Private
32	Lewis-Clark State College	Lewiston	ID	Public
33	Principia College	Elsah	IL	Private
34	Illinois Wesleyan University	Bloomington	IL	Private
35	Governor's State University	University Park	IL	Public
36	Illinois State University	Normal	IL	Public
37	Goshen College	Goshen	IN	Private
38	Rose-Hulman Institute of Technology	Terre-Haute	IN	Private
39	Ball State University	Munice	IN	Public
40	Purdue University Northwest	Hammond	IN	Public
41	Grinnell College	Grinnell	IA	Private
42	Drake University	Des Moines	IA	Private
43	University of Northern Iowa	Cedar Falls	IA	Public
44	MidAmerica Nazarene University	Olathe	KS	Private
45	Baker University	Badwin City	KS	Private
46	Pittsburg State University	Pittsburg	KS	Public
47	Emporia State University	Emporia	KS	Public
48	Berea College	Berea	KY	Private
49	Asbury University	Wilmore	KY	Private

50	Murray State University	Murray	KY	Public
51	Morehead State University	Morehead	KY	Public
52	Centenary College of Louisiana	Shreveport	LA	Private
53	Louisiana College	Pineville	LA	Private
54	Louisiana State University Shreveport	Shreveport	LA	Public
55	Southeastern Louisiana University	Hammond	LA	Public
56	Bowdoin College	Brunswick	ME	Private
57	Colby College	Waterville	ME	Private
58	Bates College	Lewiston	ME	Private
59	University of South Maine	Portland	ME	Public
60	Washington College	Chestertown	MD	Private
61	Salisbury University	Salisbury	MD	Private
62	Towson University	Towson	MD	Public
63	Frostburg State University	Frostburg	MD	Public
64	Williams College	Williamstown	MA	Private
65	Wellesley College	Wellesley	MA	Private
66	Bridgewater State University	Bridgewater	MA	Public
67	Fitchburg State University	Fitchburg	MA	Public
68	Madonna University	Livonia	MI	Private
69	Kalamazoo College	Kalamazoo	MI	Private
70	Grand Valley State Michigan University	Allendale	MI	Public
71	Lake Superior University	Sault Ste. Marie	MI	Public
72	Carleton College	Northfield	MN	Private
73	University of Saint Thomas	Saint Paul	MN	Private
74	Winona State University	Winona	MN	Public
75	Bemidji State University	Bemidji	MN	Public
76	Mississippi College	Clinton	MS	Private
77	Millsaps College	Jackson	MS	Private
78	Delta State University	Cleveland	MS	Public
79	Alcorn State University	Lorman	MS	Public
80	College of the Ozarks	Point Lookout	MO	Private
81	Webster University	Saint Louis	MO	Private
82	Truman State University	Kirksville	MO	Public
83	Missouri State University	Springfield	MO	Public
84	Carroll College	Helena	MT	Private
85	Rocky Mountain College	Billings	MT	Private
86	Montana Tech	Butte	MT	Public
87	Montana State University Billings	Billings	MT	Public
88	Creighton University	Omaha	NE	Private
89	College of Saint Mary	Omaha	NE	Private

90	University of Nebraska Kearney	Kearney	NE	Public
91	Wayne State College	Wayne	NE	Public
92	Saint Anselm College	Manchester	NH	Private
93	Keene State College	Keene	NH	Public
94	Plymouth State University	Plymouth	NH	Public
95	Drew University	Madison	NJ	Private
96	Seton Hall University	South Orange	NJ	Private
97	Ramapo College of New Jersey	Mahwah	NJ	Public
98	Montclair State University	Montclair	NJ	Public
99	New Mexico Highlands University	Las Vegas	NM	Public
100	Western New Mexico University	Silver City	NM	Public
101	Eastern New Mexico University	Portales	NM	Public
102	Vassar College	Poughkeepsie	NY	Private
103	Hamilton College	Clinton	NY	Private
104	State University of New York at Geneseo	Geneseo	NY	Public
105	State University of New York at Brockport	Brockport	NY	Public
106	Davidson College	Davidson	NC	Private
107	Salem College	Salem	NC	Private
108	Appalachian State University	Boone	NC	Public
109	Western Carolina University	Cullowhee	NC	Public
110	University of Mary	Bismarck	ND	Private
111	University of Jamestown	Jamestown	ND	Private
112	Dickinson State University	Dickinson	ND	Public
113	Mayville State University	Mayville	ND	Public
114	Kenyon College	Gambier	OH	Private
115	Oberlin College	Oberlin	OH	Private
116	Central State University	Wilberforce	OH	Public
117	Shawnee State University	Portsmouth	OH	Public
118	Oklahoma Baptist University	Shawnee	OK	Private
119	Oklahoma Christian University	Edmond	OK	Private
120	East Central University	Ada	OK	Public
121	Northwestern Oklahoma State University	Alva	OK	Public
122	Reed College	Portland	OR	Private
123	Lewis & Clark College	Portland	OR	Private
124	Western Oregon University	Monmouth	OR	Public
125	Eastern Oregon University	La Grande	OR	Public
126	Haverford College	Haverford	PA	Private
127	Swarthmore College	Swarthmore	PA	Private
128	Clarion University of Pennsylvania	Clarion	PA	Public

129	East Stroudsburg University of Pennsylvania	East Stroudsburg	PA	Public
130	Providence College	Providence	RI	Private
131	Salve Regina University	Newport	RI	Private
132	Roger Williams University	Bristol	RI	Private
133	Rhode Island College	Providence	RI	Public
134	Furman University	Greenville	SC	Private
135	South Wesleyan University	Central	SC	Private
136	University of South Carolina Aiken	Aiken	SC	Public
137	University of South Carolina Upstate	Spartanburg	SC	Public
138	Augustana University	Sioux Falls	SD	Private
139	University of Sioux Falls	Sioux Falls	SD	Private
140	Northern State University	Aberdeen	SD	Public
141	Black Hills State University	Spearfish	SD	Public
142	Rhodes College	Memphis	TN	Private
143	Sewanee – The University of the South	Sewanee	TN	Private
144	University of Tennessee at Chattanooga	Chattanooga	TN	Public
145	University of Tennessee at Martin	Martin	TN	Public
146	Trinity University	San Antonio	TX	Private
147	LeTourneau University	Longview	TX	Private
148	Texas State University	San Marcos	TX	Public
149	Tarleton State University	Stephenville	TX	Public
150	Snow College	Ephraim	UT	Private
151	Utah Valley University	Orem	UT	Public
152	Southern Utah University	Cedar City	UT	Public
153	Weber State University	Ogden	UT	Public
154	Middlebury College	Middlebury	VT	Private
155	Saint Michael's College	Colchester	VT	Private
156	Marlboro College	Marlboro	VT	Private
157	Castleton University	Castleton,	VT	Public
158	Washington and Lee University	Lexington	VA	Private
159	Liberty University	Lynchburg	VA	Private
160	College of William & Mary	Williamsburg	VA	Public
161	Christopher Newport University	Newport News	VA	Public
162	Whitman College	Walla Walla	WA	Private
163	Gonzaga University	Spokane	WA	Private
164	Western Washington University	Bellingham	WA	Public
165	Central Washington University	Ellensburg	WA	Public
166	Bethany College	Bethany	WV	Private
167	University of Charleston	Charleston	WV	Private

168	Shepherd University	Shepherdstown	WV	Public
169	Marshall University	Huntington	WV	Public
170	St Norbert College	De Pere	WI	Private
171	Carroll University	Waukesha	WI	Private
172	University of Wisconsin La Crosse	La Crosse	WI	Public
173	University of Wisconsin Whitewater	Whitewater	WI	Public

Observations:

The states of Arizona, Nevada, and Wyoming were not present in this survey because it was not found a four-year non-PhD granting institution offering a Chemistry major in these three states.

For Rhode Island, a second public institution offering a Chemistry major was not found, a third private institution completed the group of four institutions per state.

For New Mexico, only 3 institutions (all public) were analyzed because no other public or private non-PhD granting institution was found offering a Major in Chemistry. The situation was the same for New Hampshire, but two institutions were public and one was private.

For Iowa, a second public institution offering a Chemistry major was not found, but due to time limitation a third private institution was not chosen, closing only 3 institutions for this state in the survey.

