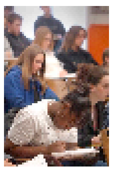
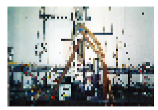
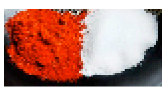
- There is no single method for designing an x-ray absorption experiment
- Think carefully about what you are trying to discover about your samples before designing your experiment.
- Aspects of your experimental design depend on the questions you are trying to answer.
- These aspects include:



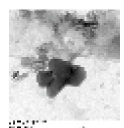
  - Expertise



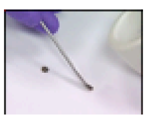
  - Choice of beamline and equipment



  - Standards



  - Complementary data



  - Samples

Choose the question you would like to answer about your sample:

- ❑ Is my sample pure X?
- ❑ Is substance X present in my sample?
- ❑ Which of several possible substances are present in my sample?
- ❑ Which of several possible substances are present in my sample, and in what proportions?
- ❑ My sample is a modified version of substance X. How is it different (bond lengths, vacancies, ordering, morphology, site occupancy, etc.)?
- ❑ My sample is highly novel or disordered. What is the immediate coordination environment of each element (ligands, coordination number, etc.)?
- ❑ What is the valence of element Y in my sample?
- ❑ My sample contains a small amount of element Y (a dopant or impurity) in an otherwise known substance X. In what environments do the atoms of Y reside?
- ❑ I am interested in how the structure of my sample (bond lengths, vacancies, ordering, morphology, site occupancy, etc.) changes with conditions (temperature, pressure, voltage, etc.). I expect only subtle changes, such as a gradual change in bond length or disorder.
- ❑ I am interested in how the structure of my sample (bond lengths, vacancies, ordering, morphology, site occupancy, phase composition, etc.) changes with conditions (temperature, pressure, voltage, etc.). I expect qualitative changes, such as the appearance of new phases.

Is my sample pure X?

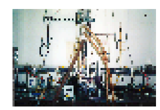
For example: “This is supposed to be a pure crystal of YBCO, but it’s suspected there may be an amorphous contaminant that does not show up in x-ray diffraction. Is it actually pure?”

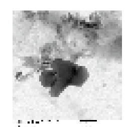
This is the simplest question to answer by XAFS, and can be done by the fingerprinting technique.


Expertise


Standards


Samples


Beamline and equipment

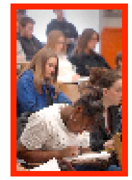

Complementary data

Return to list of questions

Is my sample pure X?

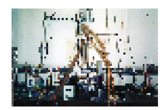
EXPERTISE

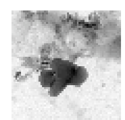
- Answering this question requires very little specialized training.


Expertise


Standards


Samples


Beamline and equipment


Complementary data

Return to list of questions

Is my sample pure X?

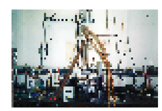
STANDARDS

- Obtain a standard of material X.
- Prepare the standard in the same way you do the sample. For instance, if your sample is powder, use a powder for a standard, not a foil.

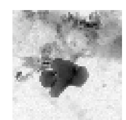
Expertise

Standards

Samples



Beamline and equipment



Complementary data

Return to list of questions

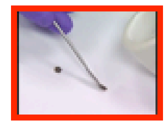
Is my sample pure X?

SAMPLES

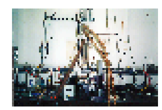
- All you need is the single sample you are interested in measuring.
- It is best if you can make it into a fine powder, but thin films or dilute mixtures (in any phase) also work well. For rough determinations a sample in almost any form will work.

Expertise

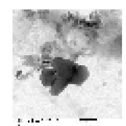
Standards



Samples



Beamline and equipment



Complementary data

Return to list of questions

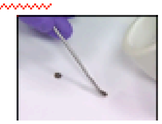
Is my sample pure X?

BEAMLINE AND EQUIPMENT

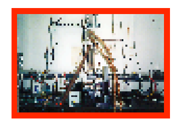
- Select a beamline with an energy range that includes as many elements in your sample as possible.
- If you can make your sample into a powder with at least 1% concentration, use transmission. If not (for example, it's a thin film or a soil sample), consider fluorescence.
- For solutions, make sure you work with beamline staff well before your experiment to discuss options.

Expertise

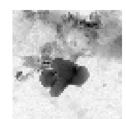
Standards



Samples



Beamline and equipment



Complementary data

Return to list of questions

Is my sample pure X?

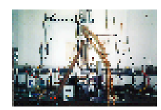
COMPLEMENTARY DATA

- For crystalline materials, x-ray diffraction will verify long-range order
- For nanoscale materials, electron microscopy is helpful

Expertise

Standards

Samples



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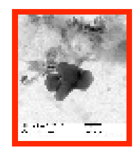


Complementary data

Return to list of questions

Is constituent X present?

For example: “Is there nickel metal in my samples?”

If there are multiple samples which vary in some way, or one sample measured under a number of conditions, this can best be answered by the PCA technique.

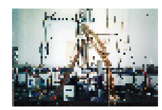
For a single sample under a single set of conditions, curve fitting to a theoretical standard, a more difficult but very powerful technique, will be necessary. If that is the case for you, switch to the “My sample is a modified version of X” information, as that corresponds more closely to your situation.

In cases where all potential constituents are known, this can be treated as a special case of “which of several phases are present?”

Expertise

Standards

Samples



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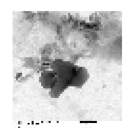


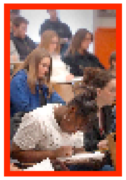
Complementary data

Return to list of questions

Is constituent X present?

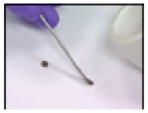
EXPERTISE

- Perhaps the best technique for answering this question is "principal component analysis" (PCA). This is a fairly simple technique to learn.
- Expect a learning curve of ten to twenty hours to become competent ith this technique.

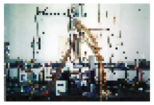


Expertise

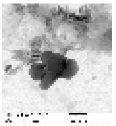
Standards



Samples



Beamline and equipment



Complementary data

Return to list of questions

Is constituent X present?

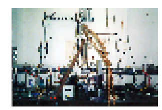
STANDARDS

- Obtain a standard of material X. It is also useful to have standards for other likely constituents in your sample, but not strictly necessary.
- If the constituents in your sample are nanoscale, but nanoscale standards are not available, bulk standards can be used.
- Prepare the standards in the same way you do the samples. For instance, if your sample is powder, use a powder for a standard, not a foil.

Expertise

Standards

Samples



Beamline and equipment

Complementary data

Return to list of questions

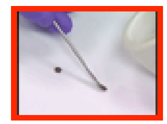
Is constituent X present?

SAMPLES

- It is highly desirable to have a series of related samples that differ in some way (this allows the use of PCA). Otherwise you have to use the more difficult analysis technique of curve fitting to a theoretical standard.
- It is best if you can make your samples into a fine powder, but thin films or dilute mixtures (in any phase) also work well.


Expertise


Standards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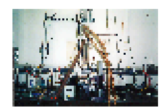
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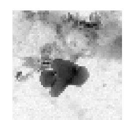

Complementary data

Return to list of questions

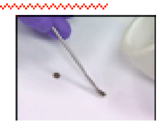
Is constituent X present?

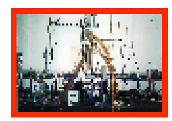
BEAMLINE AND EQUIPMENT

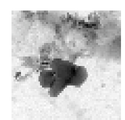
- Select a beamline with an energy range that includes as many elements in substance X as possible.
- If you can make your samples into powders with at least 1% concentration, use transmission. If not (for example, they are thin films or soil samples), consider fluorescence.
- For solutions, make sure you work with beamline staff well before your experiment to discuss options.


Expertise


Standards


Samples


Beamline and equipment


Complementary data

Return to list of questions

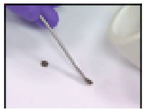
Is constituent X present?

COMPLEMENTARY DATA

- For crystalline materials, x-ray diffraction can be very helpful
- For nanoscale materials, electron microscopy is helpful
- Measurements of appropriate physical characteristics, such as density, conductivity, or magnetic properties as appropriate, as well as determination of chemical composition (“stoichiometry”), often help.

Expertise

Standards



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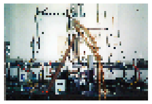
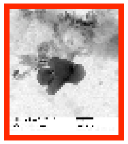


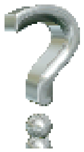
Beamline and equipment



Complementary data

◁

Return to list of questions



Which of several possible constituents are present?

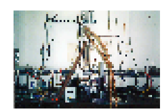
For example: “My soil samples contain a high concentration of chromium compounds, but I’m not sure which.”

This is usally best answered by a linear comination analysis, although sometimes PCA is helpful as well.

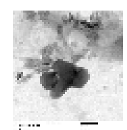
Expertise

Standards

Samples



Beamline and equipment



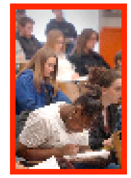
Complementary data

Return to list of questions

Which of several phases are present?

EXPERTISE

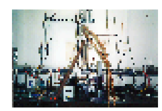
- If you have standards for all phases, this can be done by "linear combination analysis." This is a relatively straightforward technique to learn and apply.
- If you do not have standards for all phases, then use principal component analysis (PCA).
- Expect a learning curve of ten to twenty hours to become competent in either of these techniques.

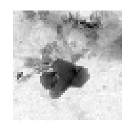


Expertise

Standards

Samples





Complementary data

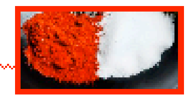
Return to list of questions

Which of several phases are present?

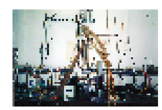
STANDARDS

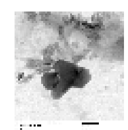
- Obtain standards for as many of the candidate constituents as you are able.
- Bulk standards are OK, even if constituents are nanoscale.
- Prepare the standards in the same way you do the samples. For instance, if your sample is powder, use a powder for a standard, not a foil.
- If an appropriate standard for one of the constituents is not available or does not exist, try to include a standard with the correct valence, and, ideally, the correct nearest-neighbor coordination. For example, $FeCl_2$ is a reasonable analog for FeI_2.


Expertise


Standards


Samples


Beamline and equipment


Complementary data

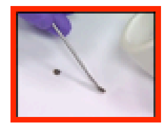
Return to list of questions

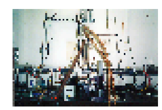
SAMPLES

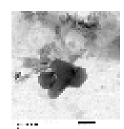
- If you do not have standards for all suspected constituents, then you need a series of related samples that differ in some way (this allows the use of PCA). If you do have standards for all suspected constituents, then you can analyze a single sample.
- It is best if you can make your samples into a fine powder, but thin films or dilute mixtures (in any phase) also work well.


Expertise


Standards


Samples


Beamline and equipment

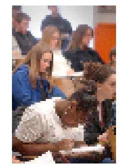

Complementary data

Return to list of questions

Which of several phases are present?

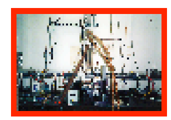
BEAMLINE AND EQUIPMENT

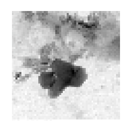
- Select a beamline with an energy range that includes as many elements in your sample as possible.
- If you can make your samples into powders with at least 1% concentration, use transmission. If not (for example, they are thin films or soil samples), consider fluorescence.
- For solutions, make sure you work with beamline staff well before your experiment to discuss options.


Expertise


Standards


Samples


Beamline and equipment


Complementary data

Return to list of questions

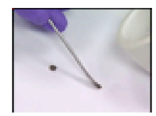
Which of several phases are present?

COMPLEMENTARY DATA

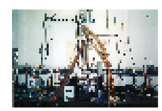
- For crystalline materials, x-ray diffraction can be very helpful
- For nanoscale materials, electron microscopy is helpful
- Measurements of appropriate physical characteristics, such as density, conductivity, or magnetic properties as appropriate, as well as determination of chemical composition (“stoichiometry”), often help.

Expertise

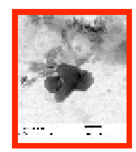
Standards



Samples



Beamline and equipment



Complementary data

Return to list of questions

Which of several possible constituents are present, and in what proportions?

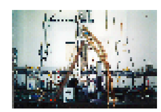
For example: “My soil samples contain a high concentration of chromium compounds, but I’m not sure which. I’d also like to know how much of each is present.”

This is usually best answered by a linear comination analysis.

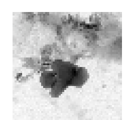
Expertise

Standards

Samples



Beamline and equipment



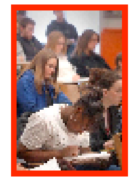
Complementary data

Return to list of questions

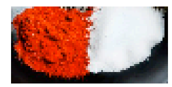
Which of several phases are present, and in what proportions?

EXPERTISE

- If you have standards for all phases, and if the phases are not substantially different from the standards (for example, both are highly crystalline), this can be done by "linear combination analysis." This is a relatively straightforward technique to learn and apply.
- For this kind of question, you must learn to carefully normalize your data, and to subtract a background from it in a consistent way.
- Expect a learning curve of ten to twenty hours to become competent in these techniques.
- If some phases do not have good standards (for example, they are nanocrystalline but the standards are bulk), then considerably more training is needed to analyze the data. See "My sample is a modified version of substance X" for more advice.

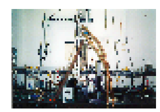


Expertise

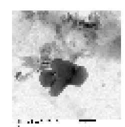


Standards

Samples



Beamline and equipment



Complementary data

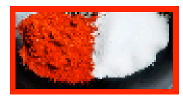
Return to list of questions

Which of several phases are present, and in what proportions?

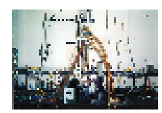
STANDARDS

- Obtain standards for as many of the candidate constituents as you are able.
- Bulk standards are OK, even if constituents are nanoscale.
- Prepare the standards in the same way you do the samples. For instance, if your sample is powder, use a powder for a standard, not a foil. Pay special attention to good sample preparation techniques.
- If an appropriate standard for one of the constituents is not available or does not exist, make sure to include a standard with the correct valence, and, ideally, the correct nearest-neighbor coordination. For example, $FeCl_2$ is a reasonable analog for FeI_2.


Expertise


Standards


Samples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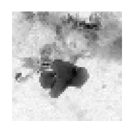

Complementary data

Return to list of questions

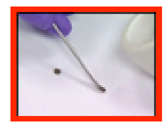
Which of several phases are present, and in what proportions?

SAMPLES

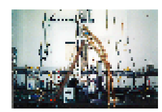
- It is best if you can make your samples into a fine powder, but thin films or dilute mixtures (in any phase) also work well.
- Pay special attention to sample preparation for this kind of analysis.

Expertise

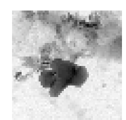
Standards



Samples



Beamline and equipment



Complementary data

Return to list of questions

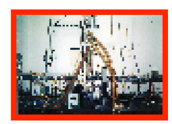
BEAMLINE AND EQUIPMENT

- Select a beamline with an energy range that includes as many elements in your sample as possible.
- If you can make your samples into powders with at least 1% concentration, use transmission. If not (for example, they are thin films or soil samples), consider fluorescence.
- For solutions, make sure you work with beamline staff well before your experiment to discuss options.

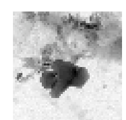
Expertise

Standards

Samples



Beamline and equipment



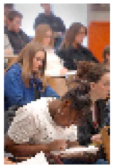
Complementary data

Return to list of questions

Which of several phases are present, and in what proportions?

COMPLEMENTARY DATA

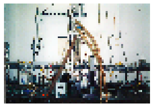
- For crystalline materials, x-ray diffraction can be helpful
- For nanoscale materials, electron microscopy may be helpful
- Measurements of appropriate physical characteristics, such as density, conductivity, or magnetic properties as appropriate, as well as determination of chemical composition (“stoichiometry”), are recommended.



Expertise

Standards

Samples



Beamline and equipment

Complementary data

Return to list of questions

My sample is a modified version of substance X. How is it different from X?

It may be different from X in many ways: bond lengths, vacancies, ordering, morphology, site occupancies, etc.

Examples:

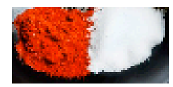
•“I have nanoscale platinum. How is it different from the bulk?”

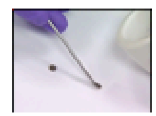
•“I have an amorphous iron oxide. I’d like to know its local structure.”

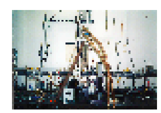
•“I have a solar cell based on copper indium selenide. But it’s the ‘defects’ that make it work. What are those defects?

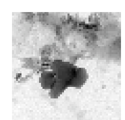
These questions require curve fitting to a theoretical standard.


Expertise


Standards


Samples


Beamline and equipment

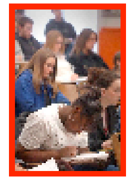

Complementary data

Return to list of questions

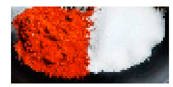
My sample is a modified version of X. How is it different?

EXPERTISE

- Answering questions of this type requires knowledge of curve fitting to a theoretical standard, a moderately advanced technique.
- It is necessary to take one or more short courses or work with someone knowledgeable in the technique in order to achieve competency.
- Expect a learning curve of forty to eighty hours to achieve basic competency in this technique.

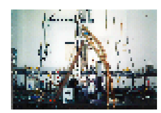


Expertise

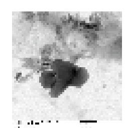


Standards

Samples



Beamline and equipment



Complementary data

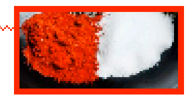
Return to list of questions

My sample is a modified version of X. How is it different?

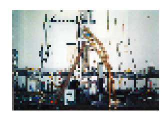
STANDARDS

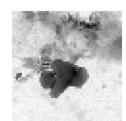
- Obtain a standard of the related material X.
- Prepare the standard in the same way you do the sample. For instance, if your sample is powder, use a powder for a standard, not a foil.


Expertise


Standards


Samples


Beamline and equipment

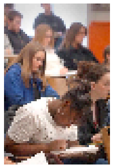

Complementary data

Return to list of questions

My sample is a modified version of X. How is it different?

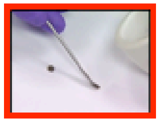
SAMPLES

- It is best if you can make your sample into a fine powder, but thin films or dilute mixtures (in any phase) also work well.

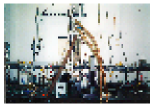


Expertise

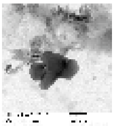
Standards



Samples



Beamline and equipment



Complementary data

Return to list of questions

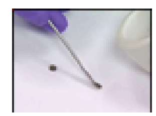
My sample is a modified version of X. How is it different?

BEAMLINE AND EQUIPMENT

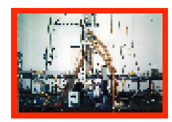
- Select a beamline with an energy range that includes as many elements in your sample as possible.
- If you can make your samples into powders with at least 1% concentration, use transmission. If not (for example, they are thin films or soil samples), consider fluorescence.
- For dilute samples, consider a beamline with relatively high flux.
- For solutions, make sure you work with beamline staff well before your experiment to discuss options.

Expertise

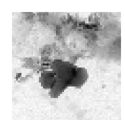
Standards



Samples



Beamline and equipment



Complementary data

Return to list of questions

My sample is a modified version of X. How is it different?

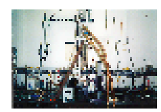
COMPLEMENTARY DATA

- Studies of this type usually involve sophisticated questions. The more you can add to the "story" by using other characterization techniques, the more convincing the analysis becomes.
- For nanoscale materials, electron microscopy may be particularly helpful.


Expertise


Standards


Samples


Beamline and equipment


Complementary data

Return to list of questions

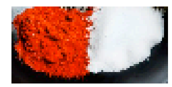
My sample is highly novel or disordered. What is the immediate coordination environment of each element?

"Immediate coordination environment" includes question like what ligands are present, their coordination number, etc..

For example: "I know uranium is present in my soil samples. But I am not sure what it is coordinated to."

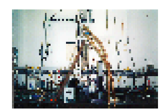
This is usually best answered by curve fitting to a theoretical standard.

Expertise

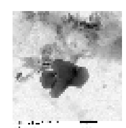


Standards

Samples



Beamline and equipment



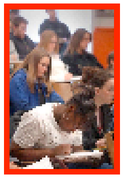
Complementary data

Return to list of questions

My sample is highly novel or disordered.
What is the immediate coordination environment of each element?

EXPERTISE

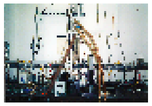
- Answering questions of this type requires knowledge of curve fitting to a theoretical standard, a moderately advanced technique.
- It is necessary to take one or more short courses or work with someone knowledgeable in the technique in order to achieve competency.
- Expect a learning curve of thirty-five to seventy hours to achieve basic competency in this technique.



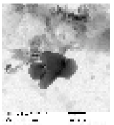
Expertise

Standards

Samples



Beamline and equipment



Complementary data

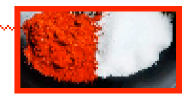
Return to list of questions

My sample is highly novel or disordered.
What is the immediate coordination environment of each element?

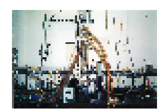
STANDARDS

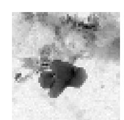
- Obtain standards for materials with coordination environments similar to those suspected to be present. For example, if a sample is expected to have a mixture of iron-sulfur and iron-oxygen bonds, it's OK to use an iron oxide and an iron sulfide as standards.
- Bulk standards are OK, even if constituents are nanoscale. Crystalline standards are OK, even if the constituents are amorphous.
- Prepare the standards in the same way you do the samples. For instance, if your sample is powder, use a powder for a standard, not a foil.


Expertise


Standards


Samples


Beamline and equipment


Complementary data

Return to list of questions

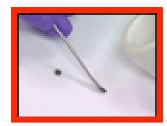
My sample is highly novel or disordered.
What is the immediate coordination environment of each element?

SAMPLES

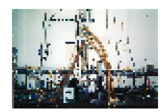
- It is best if you can make your samples into a fine powder, but thin films or dilute mixtures (in any phase) also work well.
- Pay careful attention to sample preparation for this kind of analysis.

Expertise

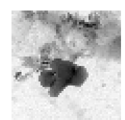
Standards



Samples



Beamline and equipment



Complementary data

Return to list of questions

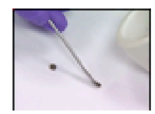
My sample is highly novel or disordered.
What is the immediate coordination environment of each element?

BEAMLINE AND EQUIPMENT

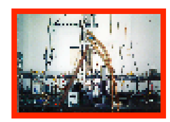
- Select a beamline with an energy range that includes as many elements in your sample as possible.
- If you can make your samples into powders with at least 1% concentration, use transmission. If not (for example, they are thin films or soil samples), consider fluorescence.
- For solutions, make sure you work with beamline staff well before your experiment to discuss options.

Expertise

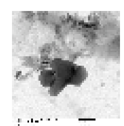
Standards



Samples



Beamline and equipment



Complementary data

Return to list of questions

My sample is highly novel or disordered.
What is the immediate coordination environment of each element?

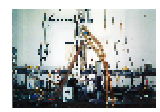
COMPLEMENTARY DATA

- Analyses of this type require careful justification. The more you can add to the "story" by using other characterization techniques, the more convincing the analysis becomes.


Expertise


Standards


Samples


Beamline and equipment


Complementary data

Return to list of questions

What is the valence of element Y?

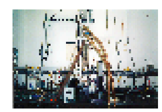
For example: “What is the valence of chromium in this sample?”

In some cases, a form of fingerprinting using XANES may be suitable. For more quantitative information when multiple valences are suspected, linear combination analysis may be appropriate.

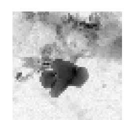
Expertise

Standards

Samples



Beamline and equipment



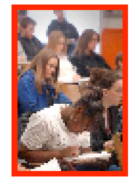
Complementary data

Return to list of questions

What is the valence of element Y?

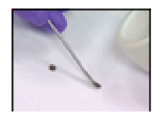
EXPERTISE

- While advanced techniques are not generally necessary to identify the valence of an element in a sample, it is very helpful to first practice on similar, known materials.
- Expect to practice five to fifteen hours to achieve competency.

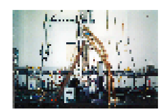


Expertise

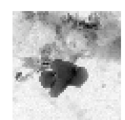
Standards



Samples



Beamline and equipment



Complementary data

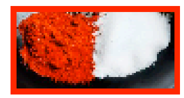
Return to list of questions

What is the valence of element X?

STANDARDS

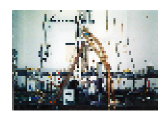
- Obtain standards for materials with absorbing element, valence, and local symmetry (e.g. coordination number) similar to those that might be present.
- Even if you are only interested in valence, try to match coordination number as well. (If coordination number is not known, include standards with different coordination numbers.) Some elements also have “high spin” and “low spin” states; in those cases, try to gather standards of both varieties.
- Bulk standards are OK, even if constituents are nanoscale. Crystalline standards are OK, even if the constituents are amorphous.
- Prepare the standards in the same way you do the samples. For instance, if your sample is powder, use a powder for a standard, not a foil.

Expertise



Standards

Samples



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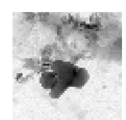


Complementary data

Return to list of questions

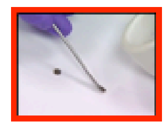
What is the valence of element Y?

SAMPLES

- All you need is the single sample you are interested in measuring.
- It is best if you can make it into a fine powder, but thin films or dilute mixtures (in any phase) also work well.

Expertise

Standards



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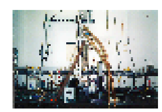
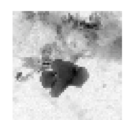


Beamline and equipment



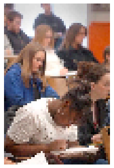
Complementary data

Return to list of questions

What is the valence of element Y?

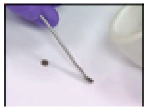
BEAMLINE AND EQUIPMENT

- Select a beamline with an energy range that includes the element that you are interested in.
- If you can make your samples into powders with at least 1% concentration, use transmission. If not (for example, they are thin films or soil samples), consider fluorescence.
- For solutions, make sure you work with beamline staff well before your experiment to discuss options.

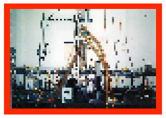


Expertise

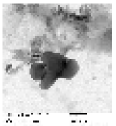
Standards



Samples



Beamline and equipment



Complementary data

Return to list of questions

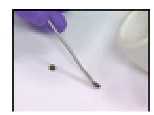
What is the valence of element Y?

COMPLEMENTARY DATA

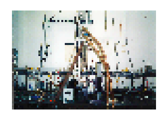
- Complementary data from other techniques, including x-ray diffraction and microscopy, can help to paint a complete picture of your sample.

Expertise

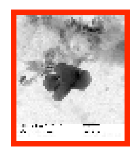
Standards



Samples



Beamline and equipment



Complementary data

◁

Return to list of questions

My sample contains a small amount of element Y in an otherwise known substance X. In what environments do the atoms of Y reside?

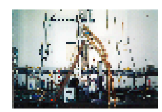
For example, a zinc oxide thin film is doped with a small amount of manganese. Does the manganese form metal clusters, substitute for zinc in the oxide, form MnO, or reside interstitially in the zinc oxide?

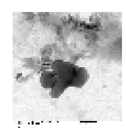
In favorable cases, fingerprinting may work, but often this calls for curve fitting to a theoretical standard.


Expertise


Standards


Samples


Beamline and equipment

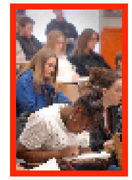

Complementary data

Return to list of questions

My sample contains a small amount of Y in an otherwise known substance X.
In what environments do atoms of Y reside?

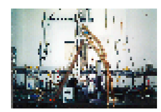
EXPERTISE

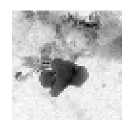
- Answering questions of this type frequently requires knowledge of curve fitting to a theoretical standard, a moderately advanced technique.
- It is necessary to take one or more short courses or work with someone knowledgeable in the technique in order to achieve competency.
- Expect a learning curve of forty to eighty hours to achieve basic competency in this technique.


Expertise


Standards


Samples


Beamline and equipment


Complementary data

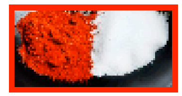
Return to list of questions

My sample contains a small amount of Y in an otherwise known substance X. In what environments do atoms of Y reside?

STANDARDS

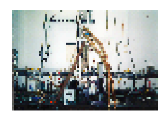
- Obtain a standard of the related material X.
- Also obtain standards for materials with a similar local environment to possibilities being considered for the environment of Y. For instance, if a small amount of Zn is doped into FeO, some possibilities are that zinc is present in metallic clusters, substituted for iron in the FeO structure, or present in ZnO clusters. Thus Zn, FeO, and ZnO standards should be measured.
- Sometimes, the related material X does not contain any elements with edges that can be measured on the beamline being used. In that case, it is not necessary to have a standard for X. (For example, many hard x-ray beamlines cannot measure the silicon edge. If you are using a beamline of that type and your sample is SiO_2 doped with chromium, it is not necessary to bring a pure SiO_2 standard.)
- Bulk standards are OK, even if constituents are nanoscale.
- Prepare the standards in the same way you do the sample. For instance, if your sample is powder, use a powder for a standard, not a foil.

Expertise

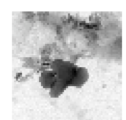


Standards

Samples



Beamline and equipment



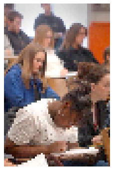
Complementary data

Return to list of questions

My sample contains a small amount of Y in an otherwise known substance X. In what environments do atoms of Y reside?

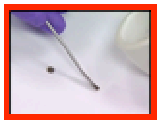
SAMPLES

- It is best if you can make your sample into a fine powder, but thin films or dilute mixtures (in any phase) also work well.

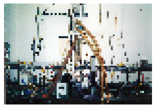


Expertise

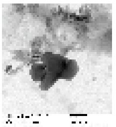
Standards



Samples



Beamline and equipment



Complementary data

Return to list of questions

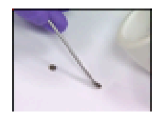
My sample contains a small amount of Y in an otherwise known substance X. In what environments do atoms of Y reside?

BEAMLINE AND EQUIPMENT

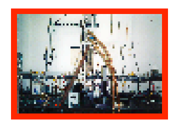
- Select a beamline with an energy range that includes element Y. It is also helpful to be able to measure edges in substance X.
- If you can make your samples into powders with at least 1% concentration, use transmission. If not (for example, they are thin films or soil samples), consider fluorescence.
- For dilute samples, consider a beamline with relatively high flux.
- For solutions, make sure you work with beamline staff well before your experiment to discuss options.

Expertise

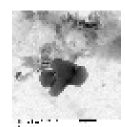
Standards



Samples



Beamline and equipment



Complementary data

Return to list of questions

My sample contains a small amount of Y in an otherwise known substance X. In what environments do atoms of Y reside?

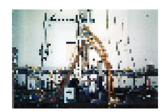
COMPLEMENTARY DATA

- Studies of this type usually involve sophisticated questions. The more you can add to the “story” by using other characterization techniques, the more convincing the analysis becomes.
- X-ray diffraction and chemical composition (“stoichiometry) are often particularly helpful.

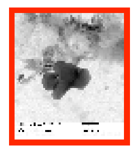
Expertise

Standards

Samples



Beamline and equipment



Complementary data

Return to list of questions

I want to know how the structure of my sample changes with conditions. I expect qualitative changes.

By “qualitative changes,” we mean changes such as the appearance of new constituents or changes in local symmetry.

For example, a copper sulfide electrode may be exposed to repeated cycling of the applied voltage. At different points in these cycles, copper metal, Cu_2S, and CuS may be present in differeing proportions.

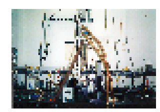
Other examples of changes in conditions are changes in temperature or pressure.

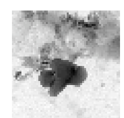
In favorable cases, linear combination analysis might work. Often, however, the individual constituents are significantly different from available standards, in which case curve fitting to a theoretical standard is called for.


Expertise


Standards


Samples


Beamline and equipment

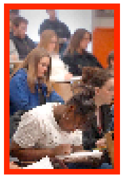

Complementary data

Return to list of questions

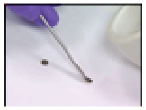
How does the structure of my sample change with conditions? (Qualitative changes expected)

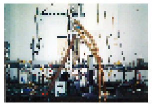
EXPERTISE

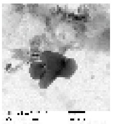
- Answering questions of this type frequently requires knowledge of curve fitting to a theoretical standard, a moderately advanced technique.
- It is necessary to take one or more short courses or work with someone knowledgeable in the technique in order to achieve competency.
- Expect a learning curve of fifty to eighty hours to achieve basic competency in this technique.


Expertise


Standards


Samples


Beamline and equipment


Complementary data

Return to list of questions

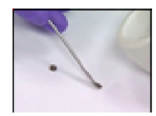
How does the structure of my sample change with conditions? (Qualitative changes expected)

STANDARDS

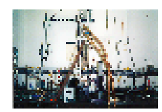
- Obtain standards for relevant pure phases. For instance, an electrode might be expected to cycle from a mixture of Cu and Cu_2S to a mixture of Cu_2S and CuS as a function of voltage and number of cycles. Cu, Cu_2S, and CuS should be used as standards.
- Prepare the standards in the same way you do the samples. For instance, if your sample is powder, use a powder for a standard, not a foil.
- If an appropriate standard for one of the constituents is not available or does not exist, make sure to include a standard with the correct valence, and, ideally, the correct nearest-neighbor coordination. For example, $FeCl_2$ is a reasonable analog for FeI_2.

Expertise

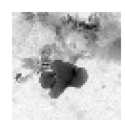
Standards



Samples



Beamline and equipment



Complementary data

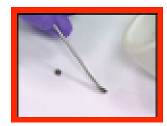
How does the structure of my sample change with conditions? (Qualitative changes expected)

SAMPLES

- For an *ex situ* measurement, identical samples are exposed to different conditions, and then prepared for measurements. The effect of annealing, for instance, will most likely be done in this way. As a rule of thumb, it is generally beneficial to prepare five to ten *ex situ* samples from a given material (e.g. cathodes removed at five to ten different points in a charging cycle).
- For *in situ* measurements, a sample may be exposed to different conditions at the beamline; for instance, temperature may be varied. Time resolved experiments are a special case of *in situ* measurements.
- In some fields, the term *operando* is used to indicate measurements on a material as it is performing its intended function; for example, a catalyst under its usual reaction conditions or a cathode in an operating battery.
- *In situ* and *operando* measurments are usually performed on only a small number of distinct materials in a given run.


Expertise


Standards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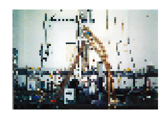
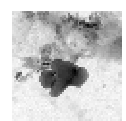

Beamline and equipment

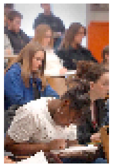

Complementary data

Return to list of questions

How does the structure of my sample change with conditions? (Qualitative changes expected)

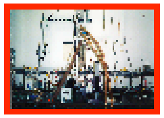
BEAMLINE AND EQUIPMENT

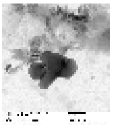
- Select a beamline with an energy range that includes as many elements in your sample as possible.
- If you can make your samples into powders with at least 1% concentration, use transmission. If not (for example, they are thin films or soil samples), consider fluorescence.
- For *in situ* or *operando* measurements, be sure to consult with beamline and safety personnel well in advance of the experiment. In some cases, they may have equipment you can use, but in all cases you will be interfacing with their equipment. As a simple example, your equipment needs to fit in their hutch!


Expertise


Standards


Samples


Beamline and equipment


Complementary data

Return to list of questions

How does the structure of my sample change with conditions?
(Qualitative changes expected)

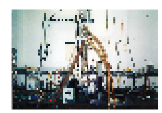
COMPLEMENTARY DATA

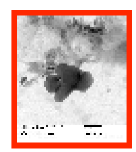
- Complementary data is usually crucial for this kind of experiment.
- Some data (e.g. conductivity) might be able to be measured at the beamline in an *in situ* experiment.
- In other cases, you will want to reproduce as closely as possible the conditions the sample is exposed to in the hutch when making measurements elsewhere, such as at the CFN or your home institution.


Expertise


Standards


Samples


Beamline and equipment


Complementary data

Return to list of questions

I want to know how the structure of my sample changes with conditions. I expect only subtle changes.

By “subtle changes,” we mean changes such as gradual changes in bond length.

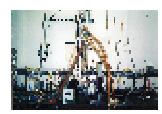
For example, a series of measurements on *tert*-butyl bromide might be made as a function of temperature to study changes in the local bromine environment that occur near the melting point.

Curve fitting to a theoretical standard will be needed for this kind of analysis.

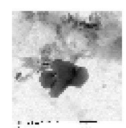
Expertise

Standards

Samples



Beamline and equipment



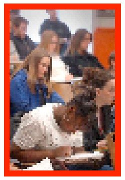
Complementary data

Return to list of questions

How does the structure of my sample change with conditions? (Only subtle changes expected)

EXPERTISE

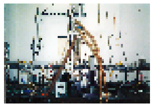
- Answering questions of this type requires curve fitting to a theoretical standard, a moderately advanced technique.
- It is necessary to take one or more short courses or work with someone knowledgeable in the technique in order to achieve competency.
- Expect a learning curve of forty to eighty hours to achieve basic competency in this technique.



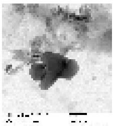
Expertise

Standards

Samples



Beamline and equipment



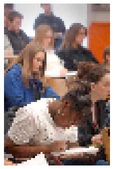
Complementary data

Return to list of questions

How does the structure of my sample change with conditions?
(Only subtle changes expected)

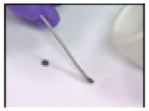
STANDARDS

- The baseline spectrum of the sample itself serves as the standard; no other standard is needed.

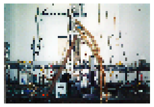


Expertise

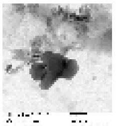
Standards



Samples



Beamline and equipment



Complementary data

Return to list of questions

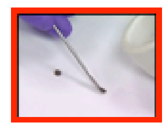
How does the structure of my sample change with conditions? (Only subtle changes expected)

SAMPLES

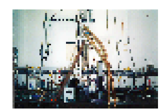
- For an *ex situ* measurement, identical samples are exposed to different conditions, and then prepared for measurements. The effect of annealing, for instance, will most likely be done in this way. As a rule of thumb, it is generally beneficial to prepare five to ten *ex situ* samples from a given material (e.g. cathodes removed at five to ten different points in a charging cycle).
- For *in situ* measurements, a sample may be exposed to different conditions at the beamline; for instance, temperature may be varied. Time resolved experiments are a special case of *in situ* measurements.
- In some fields, the term *operando* is used to indicate measurements on a material as it is performing its intended function; for example, a catalyst under its usual reaction conditions or a cathode in an operating battery.
- *In situ* and *operando* measurments are usually performed on only a small number of distinct materials in a given run.

Expertise

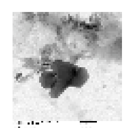
Standards



Samples



Beamline and equipment



Complementary data

Return to list of questions

How does the structure of my sample change with conditions? (Only subtle changes expected)

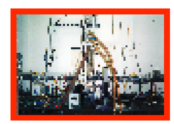
BEAMLINE AND EQUIPMENT

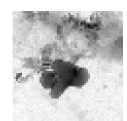
- Select a beamline with an energy range that includes as many elements in your sample as possible.
- If you can make your samples into powders with at least 1% concentration, use transmission. If not (for example, they are thin films or soil samples), consider fluorescence.
- For *in situ* or *operando* measurements, be sure to consult with beamline and safety personnel well in advance of the experiment. In some cases, they may have equipment you can use, but in all cases you will be interfacing with their equipment. As a simple example, your equipment needs to fit in their hutch!


Expertise


Standards


Samples


Beamline and equipment


Complementary data

Return to list of questions

How does the structure of my sample change with conditions?
(Only subtle changes expected)

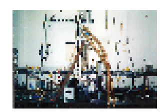
COMPLEMENTARY DATA

- Complementary data is often very important for this kind of experiment.
- Some data (e.g. conductivity) might be able to be measured at the beamline in an *in situ* experiment.
- In other cases, you will want to reproduce as closely as possible the conditions the sample is exposed to in the hutch when making measurements elsewhere, such as at the CFN or your home institution.

Expertise

Standards

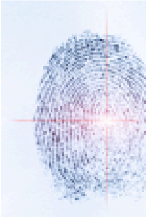
Samples



Beamline and equipment

Complementary data

Return to list of questions



Fingerprinting

In this technique, the spectrum of a sample is visually compared to the spectrum of a standard to see if they match.

This technique requires very little training, but doesn't work unless the sample is predominantly the same composition as a standard.

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Linear Combination Analysis

In this technique, a computer attempts to match the spectrum of a sample by adding together fractions of the spectra of standards.

This technique is easy to learn, but won't work unless there are standards for all constituents.

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Principal Component Analysis (PCA)

In this technique, a computer takes linear combinations of a set of spectra in such a way as to create a set of "components" that can account for the spectra, in decreasing order of importance.

This technique is best used when there are multiple related spectra, such as a series of samples or a single sample measured under a variety of conditions.

This technique can provide information about how many different constituents are present, and whether individual constituents are present.

PCA is thus useful when you have lots of data, but limited knowledge about the samples.

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Curve Fitting to a Theoretical Standard

In this technique, a computer calculates a theoretical spectrum based on a guess as to the structure of the material, and then adjusts the guessed structure so as to match the measured spectrum.

This is the only technique which can provide information about structures for which there are no good standards, and is the only way of reliably measuring quantitative information such as bond lengths.

This is also the most difficult technique to learn and apply.

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