Mystery Molecules: Identifying Materials with Nanoscale Characterization Tools

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Introduction: Novel materials are currently being developed for numerous applications including in medicine, textiles, and computers. To test and characterize these materials, scientists use a number of different analytical tools. Many of these explore what is going on at the atomic level, which is on the nanoscale. Here they can see how different atoms are behaving and even look at what is going on in dynamic or changing environments. These same tools can also help to identify materials. In this lesson plan, students will be given several similar looking materials and be asked to identify them by observing them at the macro and micro-scale. They will then be exposed to different analytical tools and describe how they can be used to explore materials at the nanoscale. A unique component of the lesson plan is to access RTNN facilities at Duke, NC State, or UNC (remotely or by field trip) to collect/analyze data with tools described in the lesson.

Standards:

North Carolina Essential Science Standards:

Chm.1.1 Analyze the structure of atoms and ions

Chm.1.2 Understand the bonding that occurs in simple compounds in terms of bond type, strength, and properties

Chm.1.3 Understand the physical and chemical properties of atoms based on their position in the Periodic Table

Phy.2.2. Analyze the behavior of waves

PSc.2.1. Understand types, properties, and structure of matter

PSc.2.2 Understand chemical bonding and chemical interactions

PSc.3.2 Understand the nature of waves

Next Generation Science Standards:

HS-PS1-1 Use the periodic table as a model to predict the relative properties of elements based on the patterns of electrons in the outermost energy level of atoms.

HS-PS3-1 Create a computational model to calculate the change in the energy of one component in a system when the change in energy of the other component(s) and energy flows in and out of the system are known.

HS-PS3-2 Develop and use models to illustrate that energy at the macroscopic scale can be accounted for as a combination of energy associated with the motions of particles (objects) and energy associated with the relative positions of particles (objects).

English Common Core Standards:

Reading: Integration of Knowledge and Ideas

7. Integrate and evaluate content presented in diverse formats and media including visually and quantitatively, as well as in words.

Writing: Text Types and purposes

1. Write arguments to support claims in an analysis of substantive topics or texts, using valid reasoning and relevant and sufficient evidence

2. Write informative/explanatory texts to examine and convey complex ideas and information clearly and accurately through the effective selection, organization, and analysis of content.

9. Draw evidence from literary or informational texts to support analysis, reflection, and research.

Speaking and listening standards

1. Prepare for and participate effectively in a range of conversations and collaborations with diverse partners, building on others' ideas and expressing their own clearly and persuasively

4. Present information, findings, and supporting evidence such that listeners can follow the line of reasoning and the organization, development, and style are appropriate to task, purpose, and audience.

Learning Objectives:

- Students will be able to discuss limitations of macroscale observations in regards to identifying unknown compounds
- Students will be able to describe and compare different nanoscale characterization techniques
- Students will be able to explain what the characterization techniques can tell us about a molecule/material's structure
- Students will be able to determine the type of data they will obtain with specific characterization techniques and relate this to a material's molecular structure

Appropriate Grade Level: 9-12

Group Size: Groups of 3-4 students

Setting: Classroom

Approximate time of lesson: 2 hours for in class lesson, 1.5 – 2 hours for remote access session or classroom visit (plan for 2-3 hours at a facility if scheduling a field trip)

Resources Needed for Student:

- Magnifying glass and/or basic light microscope
- Salt
- Sugar (Sucrose)
- Epsom Salt (magnesium sulfate)
- MSG (monosodium glutamate)
- (Other white powders that looks similar: for example, baking soda, baking powder, Splenda, etc.)
- Printouts of molecular structures of various powders
- Handouts on different characterization techniques

Resources needed for educators:

- Method to display YouTube videos

Lesson Activity:

Students are divided into groups of 3-4 students. They are given different unknown white powders and chemical structures of potential molecules. Ask students what differences they see in the molecular structures (Bonding, elemental composition, bonds between elements, crystal structure). Students then examine the powders at the macro level and answer questions like: What differences can you see between the powders? Can you make any connections between what you observe and the molecular structure? Make a guess to what each powder is. Explain why you think so. Students share responses with class.

Groups look at powders under the microscope or magnifying glass and answer the same questions as above. Share responses with class.

Each group will then be given a different analytical technique description and/or a link to a YouTube video with a short description. (Optional: students are assigned a technique and research it as a homework assignment.) Each group will describe how they could use that technique to determine what the unknown powders are. Answer questions like: What kinds of information would this technique give you? How does this relate to the molecules' structures? Would the information obtained be sufficient to determine the molecule or would you need to use other techniques? How would the data be used to differentiate between the different powders?

Schedule a remote session or field trip to an RTNN facility to see instruments in action and collect and analyze data. *Or* send samples to RTNN to get analyzed! Experts will run samples, provide data sets, and help analyze the data.

After these activities, ask students some follow-up questions:

- What would be some advantages and disadvantages of these analytical techniques?
- How could these techniques be applied in real-world scenarios? What would they be used for?
- Can you think of other ways that you might be able to distinguish between the mystery powders?

Background reading for teachers:

Links for additional information:

www.rtnn.org

http://www.periodictable.com/index.html

It may be helpful to give students a little bit of information about crystal structure and how molecules pack together to form crystals.

- https://en.wikipedia.org/wiki/Crystal_structure
- https://saylordotorg.github.io/text_general-chemistry-principles-patterns-and-applications-v1.0/s16-03-structures-of-simple-binary-co.html
- Crystal Structure Review: https://www.youtube.com/watch?v=iPb8vRtroLU

MIT Class: Introduction to Solid State Chemistry (http://ocw.mit.edu/courses/materials-science-and-engineering/3-091sc-introduction-to-solid-state-chemistry-fall-2010/)

- http://ocw.mit.edu/courses/materials-science-and-engineering/3-091sc-introduction-to-solid-state-chemistry-fall-2010/crystalline-materials/15-introduction-to-crystallography/
- http://ocw.mit.edu/courses/materials-science-and-engineering/3-091sc-introduction-to-solid-state-chemistry-fall-2010/crystalline-materials/16-crystallographic-notation-x-rays/
- http://ocw.mit.edu/courses/materials-science-and-engineering/3-091sc-introduction-to-solid-state-chemistry-fall-2010/crystalline-materials/17-x-ray-emission-absorption/
- http://ocw.mit.edu/courses/materials-science-and-engineering/3-091sc-introduction-to-solid-state-chemistry-fall-2010/crystalline-materials/18-x-ray-diffraction-techniques/

Information about the electromagnetic spectrum

- https://en.wikipedia.org/wiki/Electromagnetic_spectrum
- https://www.khanacademy.org/science/chemistry/electronic-structure-of-atoms/bohr-model-hydrogen/a/light-and-the-electromagnetic-spectrum

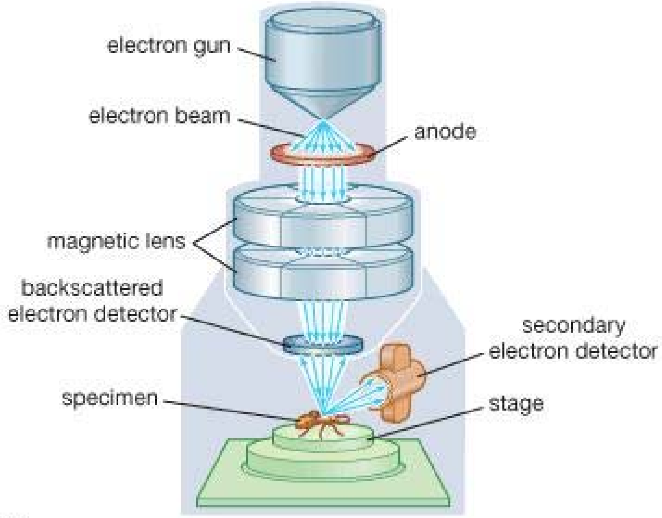
Links to other lesson plans:

There are several lesson plans that this is modified from. Some of these could be paired with this lesson.

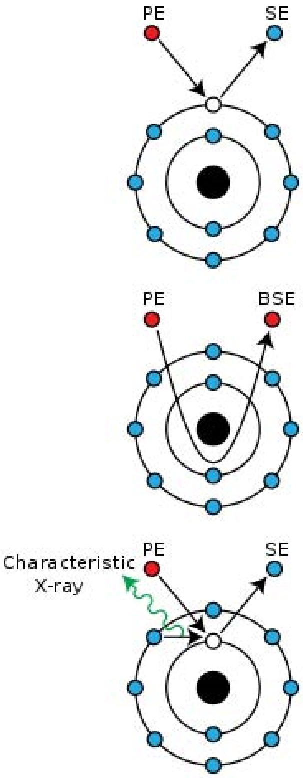
- http://www.middleschoolchemistry.com/lessonplans/chapter6/lesson6
- http://www.nsta.org/publications/news/story.aspx?id=48629
- https://www.uu.edu/books/greenchemistrylabs/pdf/lab11-st.pdf
- http://microscopy4kids.org/Making_and_Observing_Crystals

Scanning Electron Microscopy (SEM)

A **scanning electron microscope** (**SEM**) is a microscope that produces images of a sample by scanning it with a focused beam of electrons produced by an electron gun. The electrons interact with atoms in the sample, producing various signals (scattered electrons, X-rays, etc.) that contain information about the sample's surface topography and composition. The electron beam is generally scanned across the surface, and the beam's position is combined with the detected signal to produce an image. SEM can achieve resolution better than 1 nanometer (nm). Specimens can be observed in high vacuum, in low vacuum, in wet conditions (in environmental SEM), and at a wide range of cryogenic or elevated temperatures.



Scanning Electron Microscope Diagram
(https://www.britannica.com/technology/scanning-electron-microscope)

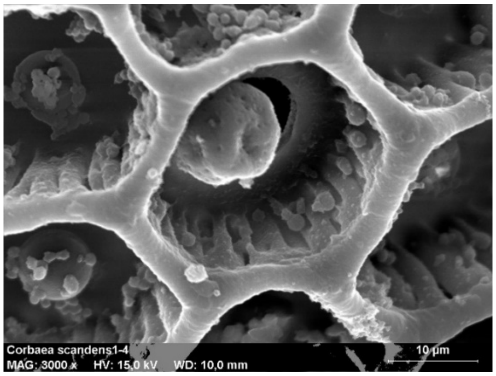


Mechanisms of emission of secondary electrons, backscattered electrons, and characteristic X-rays from atoms of the sample By Rob Hurt - Own work, CC BY-SA 4.0, https://commons.wikimedia.org/w/index.php?curid=50931451

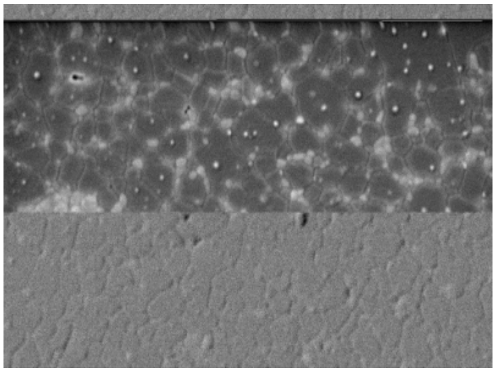
The signals result from interactions of the electron beam with atoms at various depths within the sample. In the most common or standard detection mode, secondary electron imaging or SEI, the secondary electrons are emitted from very close to the specimen surface. Consequently, SEM can produce very high-resolution images of a sample surface, revealing details less than 1 nm in size. Back-scattered electrons (BSE) are beam electrons that are reflected from the sample by elastic scattering. They emerge from deeper locations within the specimen and consequently the resolution of BSE images is generally poorer than SE images. However, BSE are often used in analytical SEM along with the spectra made from the characteristic X-rays, because the intensity of the BSE signal is strongly related to the atomic number of the specimen. BSE images can provide information about the distribution of different elements in the sample. For the same reason, BSE imaging can image colloidal gold immuno-labels of 5 or 10 nm diameter, which would otherwise be difficult or impossible to detect in secondary electron images in biological specimens. Characteristic X-rays are emitted when the electron beam removes an inner shell electron from the sample, causing a higher-energy electron to fill the shell and release energy. These characteristic X-rays are used to identify the composition and measure the abundance of elements in the sample.

Due to the very narrow electron beam, SEM micrographs have a large depth of field yielding a characteristic three-dimensional appearance useful for understanding the surface structure of a sample. A wide range of magnifications is possible, from about 10 times (about equivalent to that of a powerful hand-lens) to more than 500,000 times, about 250 times the magnification limit of the best light microscopes.

Example Data:



SEM image of Cobaea scandens pollen. Note the scale bar. By Marie Majaura - Own work, CC BY-SA 3.0, https://commons.wikimedia.org/w/index.php?curid=3105565



Comparison of SEM techniques: Top: backscattered electron analysis – composition Bottom: secondary electron analysis – topography Public Domain, https://en.wikipedia.org/w/index.php?curid=14672854

References:

Much of the above text was modified from https://en.wikipedia.org/wiki/Scanning_electron_microscope

- http://serc.carleton.edu/research_education/geochemsheets/techniques/SEM.html
- https://cmrf.research.uiowa.edu/scanning-electron-microscopy
- http://science.howstuffworks.com/scanning-electron-microscope.htm

Video Links:

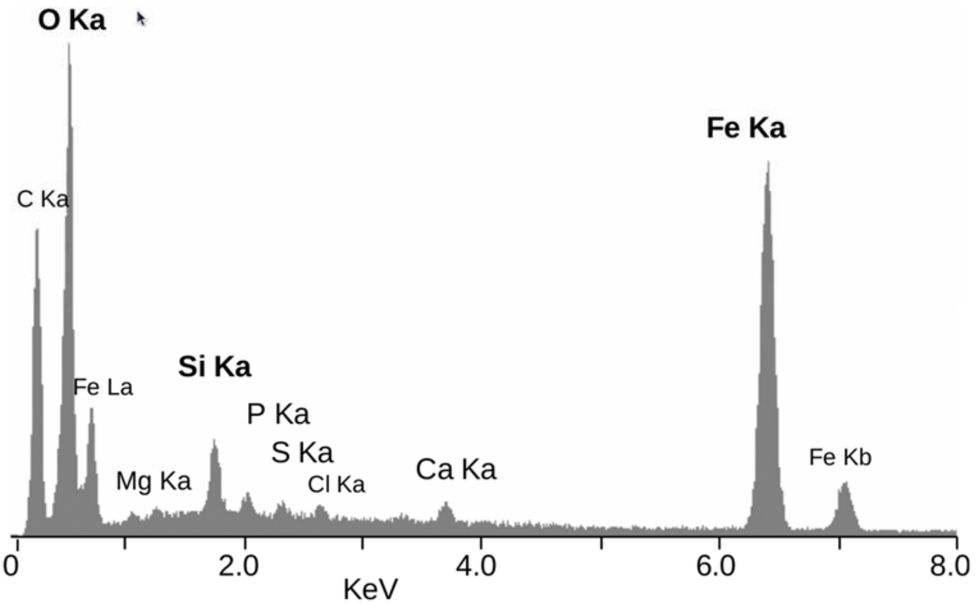
- https://www.**youtube**.com/watch?v=sfogToduqGQ
- ttps://www.**youtube**.com/watch?v=bfSp8r-YRw0
- https://www.**youtube**.com/watch?v=GY9lfO-tVfE

Energy-dispersive X-ray spectroscopy

Energy-dispersive X-ray spectroscopy (**EDS**, **EDX**, or **XEDS**), sometimes called **energy dispersive X-ray analysis** (**EDXA**) or **energy dispersive X-ray microanalysis** (**EDXMA**), is an analytical technique used for the elemental analysis or chemical characterization of a sample. It relies on an interaction of some source of X-ray excitation and a sample. Its characterization capabilities are due in large part to the fundamental principle that each element has a unique atomic structure allowing a unique set of peaks on its electromagnetic emission spectrum (which is the main principle of spectroscopy).

To stimulate the emission of characteristic X-rays from a specimen, a high-energy beam of charged particles such as electrons (like the beam used in scanning electron microscopy) or protons, or a beam of X-rays, is focused into the sample being studied. At rest, an atom within the sample contains ground state (or unexcited) electrons in discrete energy levels or electron shells bound to the nucleus. The incident beam may excite an electron in an inner shell, ejecting it from the shell while creating an electron hole where the electron was. An electron from an outer, higher-energy shell then fills the hole, and the difference in energy between the higher-energy shell and the lower energy shell may be released in the form of an X-ray. The number and energy of the X-rays emitted from a specimen can be measured by an energy-dispersive spectrometer. As the energies of the X-rays are characteristic of the difference in energy between the two shells and of the atomic structure of the emitting element, EDS allows the elemental composition of the specimen to be measured.

Example Data:



EDS spectrum of the mineral crust of the vent shrimp Rimicaris exoculata *Most of these peaks are X-rays given off as electrons return to the K electron shell.(K-alpha and K-beta lines) One peak is from the L shell of iron.CC BY 3.0, https://commons.wikimedia.org/w/index.php?curid=7676417*

Video Links:

https://www.**youtube**.com/watch?v=TLeKyoGHiMA

https://www.**youtube**.com/watch?v=Zmvm-eX4H18

ttps://www.**youtube**.com/watch?v=Kl_K4N3EHbA

References:

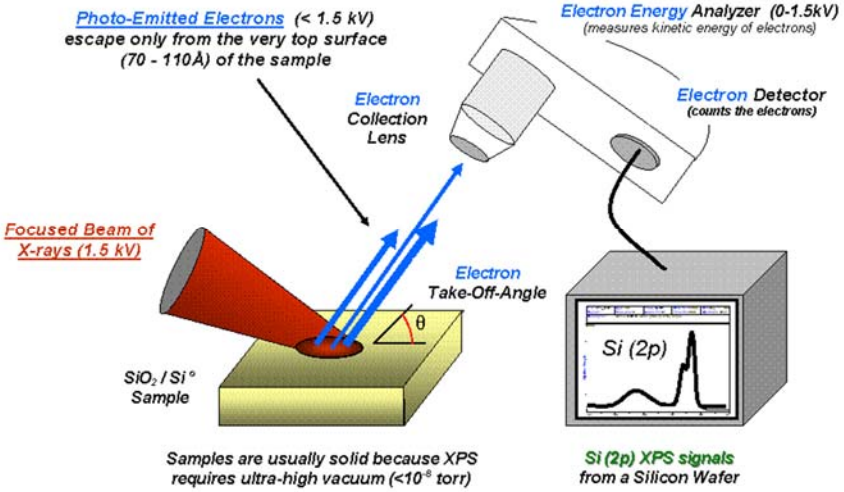
Much of the above text was modified from https://en.wikipedia.org/wiki/Energy-dispersive_X-ray_spectroscopy

http://www.charfac.umn.edu/instruments/eds_on_sem_primer.pdf

X-ray Photoelectron Spectroscopy (XPS)

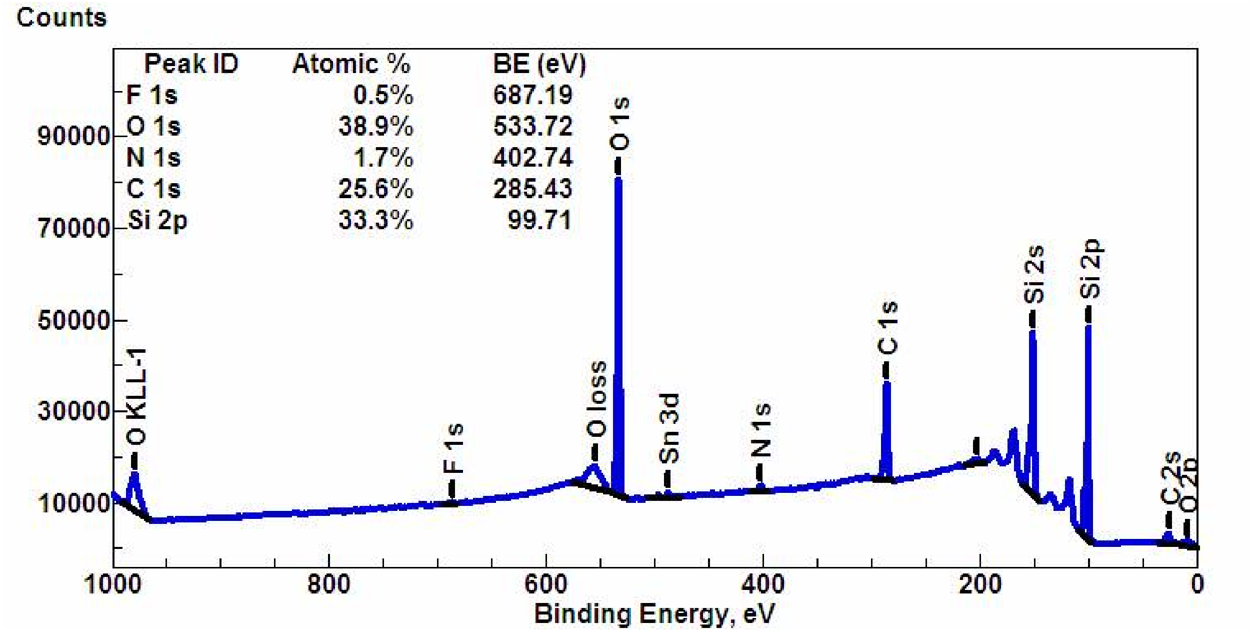
(Other name: Electron Spectroscopy for Chemical Analysis, ESCA)

X-ray photoelectron spectroscopy (XPS) is a surface-sensitive quantitative spectroscopic technique that measures the elemental composition at the parts per thousand range, empirical formula, chemical state and electronic state of the elements that exist within a material. XPS spectra are obtained by irradiating a material with a beam of X-rays while simultaneously measuring the kinetic energy and number of electrons that escape from the top 0 to 10 nm of the material being analyzed. XPS requires high vacuum ($P \sim 10^{-8}$ millibar) or ultra-high vacuum (UHV; $P < 10^{-9}$ millibar) conditions. A typical XPS spectrum is a plot of the number of electrons detected (sometimes per unit time) (y-axis) versus the binding energy of the electrons detected (x-axis). Each element produces a characteristic set of XPS peaks at characteristic binding energy values that directly identify each element that exists in or on the surface of the material being analyzed. (Electron binding energy is a measure of the energy required to free electrons from their atomic orbits. For each electron in a molecule there will be a specific binding energy required to remove it.) These characteristic spectral peaks correspond to the electron configuration of the electrons within the atoms, e.g., 1*s*, 2*s*, 2*p*, 3*s*, etc. The number of detected electrons in each of the characteristic peaks is directly related to the amount of element within the XPS sampling volume.



To count the number of electrons during the acquisition of a spectrum with a minimum of error, XPS detectors must be operated under ultra-high vacuum (UHV) conditions because electron counting detectors in XPS instruments are typically one meter away from the material irradiated with X-rays. This long path length for detection requires such low pressures.

Example Data:



Wide-scan or survey spectrum of a silicon wafer, showing all elements present. A survey spectrum is usually the starting point of most XPS analyses because it shows all elements present on the sample surface and allows one to set up subsequent high-resolution XPS spectra acquisition. The inset shows a quantification table indicating all elements observed, their binding energies, and their atomic percentages. By Bvcrist - Own work (enwiki), CC BY-SA 3.0, https://commons.wikimedia.org/w/index.php?curid=17267639

References:

Much of the above text was modified from https://en.wikipedia.org/wiki/X-ray_photoelectron_spectroscopy

https://en.wikipedia.org/wiki/Ionization_energy

http://xpssimplified.com/whatisxps.php

http://xpssimplified.com/knowledgebase.php

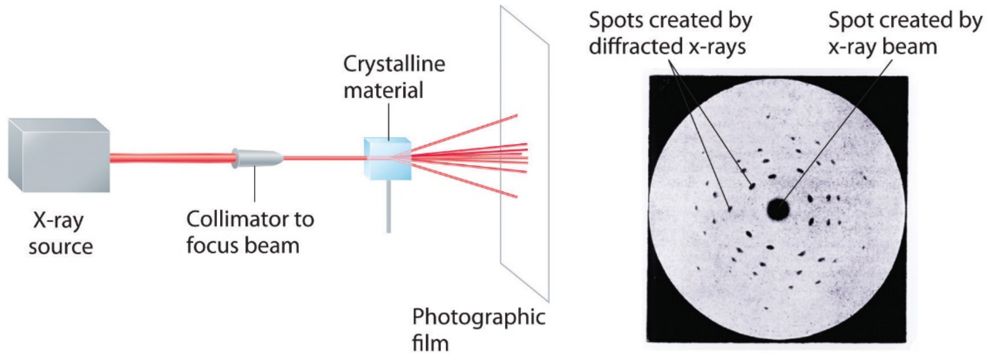
Video Links:

https://www.**youtube**.com/watch?v=8njmZdnvjZs

https://www.**youtube**.com/watch?v=rR7GwTqxFOE

X-ray Diffraction

X-ray diffraction (XRD) is a tool used for identifying the atomic and molecular structure of a crystal, in which the crystalline atoms cause a beam of incident X-rays to diffract into many specific directions. Many materials can form crystals—such as salts, metals, minerals, semiconductors, as well as various inorganic, organic and biological molecules. In a single-crystal X-ray diffraction measurement, a crystal is mounted on a goniometer. The goniometer is used to position the crystal at selected orientations. The crystal is illuminated with a finely focused monochromatic beam of X-rays, producing a diffraction pattern of regularly spaced spots known as *reflections*.



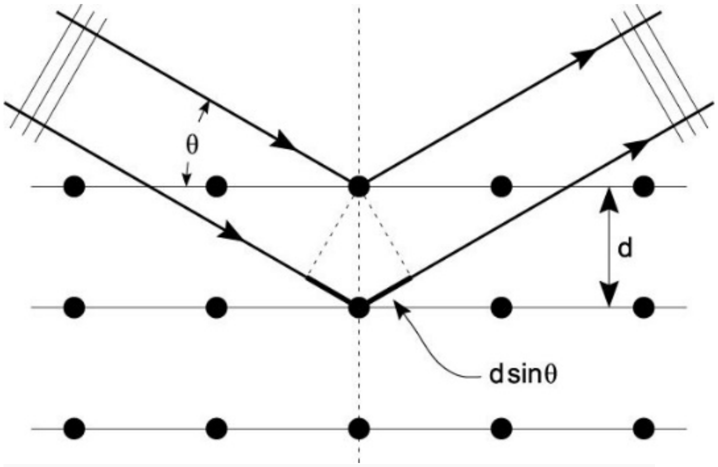
(a) X-ray diffraction

(b) X-ray diffraction pattern captured on photographic film

These illustrations show (a) a schematic drawing of x-ray diffraction and (b) the x-ray diffraction pattern of a zinc blende crystalline solid captured on photographic film.

https://saylordotorg.github.io/text_general-chemistry-principles-patterns-and-applications-v1.0/s16-03-structures-of-simple-binary-co.html

Crystals are regular arrays of atoms, and X-rays can be considered waves of electromagnetic radiation. Atoms scatter X-ray waves, primarily through the atoms' electrons. Just as an ocean wave striking a lighthouse produces secondary circular waves emanating from the lighthouse, so an X-ray striking an electron produces secondary spherical waves emanating from the electron. This phenomenon is known as elastic scattering, and the electron (or lighthouse) is known as the *scatterer*. A regular array of scatterers produces a regular array of spherical waves. Although these waves cancel one another out in most directions through destructive interference, they add constructively in a few specific directions: when conditions satisfy Bragg's Law: $2d \sin\theta = n\,\lambda$

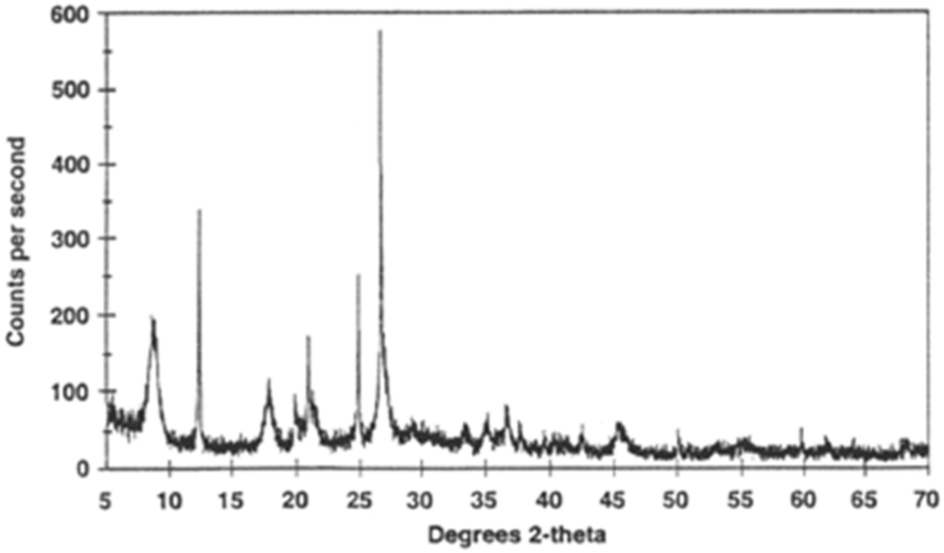


The incoming X-ray (coming from upper left) causes each atom (scatterer) to re-radiate a small portion of its intensity as a spherical wave. If atoms are arranged symmetrically with a separation d, *these spherical waves will be in sync (add constructively) only in directions where their path-length difference* 2d *sin θ equals an integer multiple of the wavelength λ. In that case, part of the incoming beam is deflected by an angle 2θ, producing a* reflection *spot in the diffraction pattern. By Hydrargyrum - Own work, CC BY-SA 3.0,*

https://commons.wikimedia.org/w/index.php?curid=17543875

Here d is the spacing between diffracting planes, θ is the incident angle, n is any integer, and λ is the wavelength of the beam. This law relates the wavelength of electromagnetic radiation to the diffraction angle and the lattice spacing in a crystalline sample. These diffracted X-rays are then detected, processed and counted. By scanning the sample through a range of 2θangles, all possible diffraction directions of the lattice should be attained due to the random orientation of the material. Conversion of the diffraction peaks to d-spacings allows identification of the material because each material has a set of unique d-spacings. Typically, this is achieved by comparison of d-spacings with standard reference patterns.

Example Data:



X-ray powder diffractogram. Peaks occur where the X-ray beam has been diffracted by the crystal lattice. The unique set of d-spacings derived from this pattern can be used to 'fingerprint' the material. http://serc.carleton.edu/details/images/8418.html

Video Links:

https://www.youtube.com/watch?v=UfDW0-kghmI

https://www.youtube.com/watch?v=A1mbgDXag7c

References:

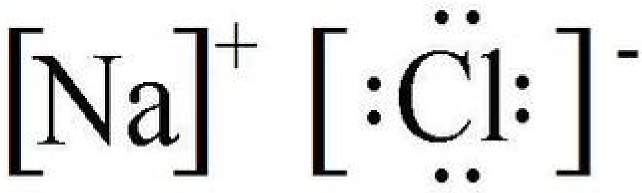
https://en.wikipedia.org/wiki/X-ray_crystallography

http://serc.carleton.edu/research_education/geochemsheets/techniques/XRD.html

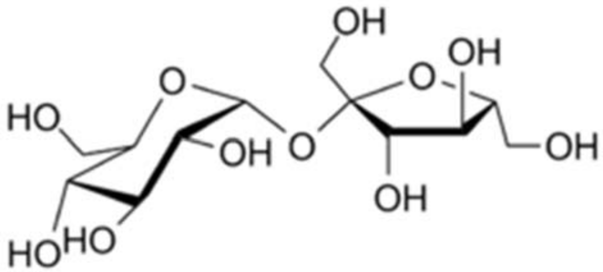
http://ocw.mit.edu/courses/materials-science-and-engineering/3-091sc-introduction-to-solid-state-chemistry-fall-2010/crystalline-materials/18-x-ray-diffraction-techniques/

https://saylordotorg.github.io/text_general-chemistry-principles-patterns-and-applications-v1.0/s16-03-structures-of-simple-binary-co.html

Molecular Structures of Mystery Materials



https://www.khanacademy.org/science/chemistry/atomic-structure-and-properties/introduction-to-compounds/a/paul-article-2



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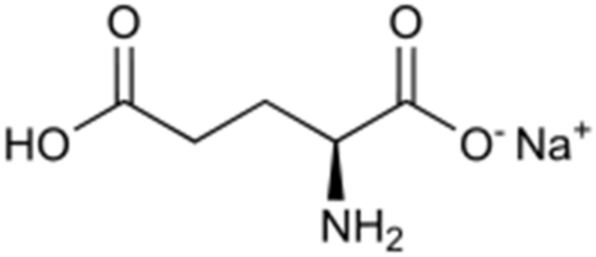
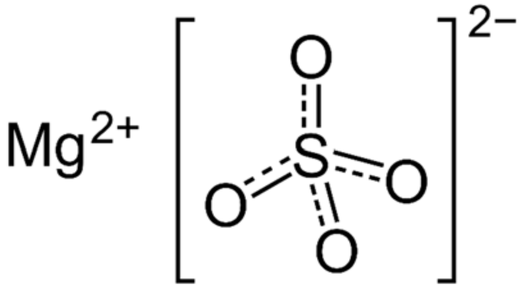


https://en.wikipedia.org/wiki/File:Monosodiumglutamate.svg



https://commons.wikimedia.org/wiki/File:Magnesium_sulfate.svg