

GENESIS SAMPLE ANALYSIS: The bottom line



Science Education Module Grades 9–12



Featuring:

Teacher Guide

★ Mass Spectrometry

Student Texts

- ★ Secondary Ion Mass Spectrometry—SIMS
- ★ Mass Spectrometry—A Historic Technique of Great Importance to Genesis
- ★ Magic Bullets for Elemental Analysis

Student Activity

★ SIMS Simulation

Mission Partners:

Jet Propulsion Laboratory California Institute of Technology Johnson Space Center Los Alamos National Laboratory Lockheed Martin Astronautics McREL

http://genesismission.jpl.nasa.gov

Education

Genesis Sample Analysis: The Bottom Line

GENESIS

Science Mini-Module Overview

Target Grade Level: High School, 9-12

Target Content Areas: Science, History and Nature of Science, Technology

Estimated Time: Entire Mini-Module—1 week Interactive Activity—1 day

Objectives: This mini-module focuses on the analysis of the Genesis solar wind samples, with the goal of helping students to understand: a) the minute size of the entire collected sample and its extremely low concentration in the collectors; b) how the highly dispersed solar wind particles will be removed from the collectors for analysis; c) how mission scientists have



addressed the enormous challenge of sorting, identifying, and quantifying the elements and their isotopes in the collected solar wind sample after the particles are removed from the collectors; and d) how a special technique involving neutron bullets will be utilized for additional analyses.

Learning Activities: By reading texts, students will learn about the two fundamental analytical procedures to be employed and about the special technique of secondary ion mass spectrometry (SIMS) that will be used to remove and analyze samples from the Genesis collectors. Then the mini-module features a hands-on, interactive, computer simulation activity that focuses on the SIMS technique. In this activity, the students will learn: a) how a SIMS experiment is carried out, and b) how to interpret a mass spectrum to identify elements.

Special Note: The SIMS activity requires Macromedia Shockwave, which can be downloaded at no cost. When you access the activity, you will be given the opportunity to download this software. If you download the activity through a phone line, be patient. Depending on the speed of your modem, it may take several minutes for the download to be complete.

Background Information: How was the Solar System formed? Why does life as we know it exist on Earth but not on other planets in the Solar System? Why are some planets solid and rocky, but others are gaseous? These are three of the many questions that scientists will try to answer with help from the Genesis mission, launched by the National Aeronautics and Space Administration (NASA) on August 8, 2001.

The Sun contains nearly all of the matter that makes up our Solar System. Scientists believe that the components of the Sun that are spewed into outer space in the form of the **solar wind** have the same elemental composition as the solar nebula—the cloud of interstellar gas and dust from which the various bodies in our Solar System formed some 4.6 billion years ago. It is not possible for humans (or spacecraft for that matter) to travel directly to the Sun to analyze its elemental composition without being destroyed by the intense heat of that nuclear furnace, nor is it possible for scientists to travel back in time to see how the Solar System was formed. Thus, scientists are forced to take an indirect route to gaining an understanding of the evolution of our Solar System by collecting particles of the Sun from a safedistance away, and this is what the Genesis mission set out to do—capture particles of the solar wind and bring them back to Earth for study.

After more than two years of collecting elements of the solar wind while circling between the Earth and the Sun at what is called the L1 point, the Genesis payload was returned to eagerly awaiting scientists as of September 8, 2004. Now, and for the next few years, it will be the time for scientists to pursue the final goal of the mission—conducting a complete analysis of the elemental and isotopic abundances as found in the solar wind and developing and testing models of Solar System evolution based on the data provided by the Genesis mission.

Genesis Sample Analysis: The Bottom Line provides a simulation of one of the fundamental scientific approaches to be used in the analysis of solar wind samples. While the intimate details of experiments on solar wind samples that will be carried out in scientific labs likely exceed the scientific expertise of most high school students, the simulation in this mini-module accurately provides a realistic depiction of one of the *fundamental* ideas to be used in these labs. The accompanying standards-aligned texts provide students with a solid background from which they may expand their knowledge should they be so inclined. The simulation also contains links to additional exercises dealing with analysis of mass spectra, providing some flexibility for the teacher to expand on the science of the simulation. Also included is a link to a table of isotopes that contains a wealth of information. Inquisitive students will find the isotopic distribution data interesting, and they may be stimulated to pursue the concept of isotopes in more detail after perusing these data. For teachers who want to further extend the mission-related learning, this minimodule provides links to suggested activities presented in other Genesis modules. For more information about Genesis modules visit: http://www.genesismission.org/educate/scimodule/moduleoverview.html.

Genesis Sample Analysis: The Bottom Line

Description	Teacher	Student	Standards
of Activities	Materials	Materials	Addressed
As an introduction to mass spectrometry, students read two texts and engage in a class discussion. The first text, "Secondary Ion Mass Spectrometry—SIMS," focuses on the analytical tools used in the Genesis mission. The second text, "Mass Spectrometry—A Historic Technique of Great Importance to Genesis," shows that mass spectrometry is rooted in history, dating back to 1919.	Mass Spectrometry • <u>Teacher Guide</u> (pages 4 - 9)	Secondary Ion Mass Spectrometry—[] SIMS • <u>Student Text</u> (pages 10 - 12) Mass Spectrometry—A Historic Technique of Great Importance to Genesis • <u>Student Text</u> (pages 13 - 15)	 Science and Technology Understanding about science and technology Physical Science Structure and changes in properties of matter Structure of atoms Motions and forces History and Nature of Science Science as a human endeavor Nature of science and scientific knowledge
Students experiment with a computer interactive, which simulates the Secondary Ion Mass Spectrometry (SIMS) technology used to analyze solar wind particles collected during the Genesis mission. Not only will students develop a better understanding of this specialized instrumentation, they will also learn how to interpret data and identify elements.	Mass Spectrometry • <u>Teacher Guide</u> (pages 4 - 9)	 SIMS Simulation Student Activity (pages 16 - 20) Online Interactive 	 Science as Inquiry Abilities necessary to do scientific inquiry Understanding about scientific inquiry Earth and Space Science The origin and evolution of the Earth system The origin and evolution of the universe Science and Technology Understanding about science and technology Structure and changes in properties of matter Structure of atoms Motions and forces
The final student text describes additional analysis techniques that involve neutron bullets.	Mass Spectrometry • Teacher Guide (pages 4 - 9)	Magic Bullets for Elemental Analysis • <u>Student Text</u> (pages 21 - 23)	 Science and Technology Understanding about science and technology Physical Science Structure and changes in properties of matter Structure of atoms Motions and forces History and Nature of Science Science as a human endeavor Nature of science and scientific knowledge

This education mini-module, *Genesis Sample Analysis: The Bottom Line*, was developed by educators at <u>Mid-continent Research for Education and Learning</u> in collaboration with the Jet Propulsion Laboratory.

Education

Genesis Sample Analysis: The Bottom Line

GENESIS

Mass Spectrometry

TEACHER GUIDE

BACKGROUND INFORMATION

The primary objective of NASA's Genesis mission is to obtain precise_measures of solar elemental and isotopic abundances by sending a spacecraft to collect samples of the solar wind and return these samples to Earth for detailed <u>analysis</u>. On the basis of this information, models of Solar System formation, evolution, and early nebular composition will be tested and revised as suggested by the newly acquired data.

The *total* amount of collected material is expected to be very small (on the order of 0.4 milligrams) or a single grain of salt. Nevertheless, this ambitious project hopes to analyze completely this small sample of material for its constituent chemical elements and their isotopic distributions. To do so will require exquisitely sensitive instrumentation.

SIMS: Secondary Ion Mass Spectrometry



Various laboratories are developing new analytical methods and enhanced instrumental capabilities to meet the specific and exacting needs of the Genesis program.

Instrumentation

If one visits the Genesis technical Web site where scientific details are presented, one finds a listing of the instruments considered for use in the analyses. With one exception, the acronyms for these instruments all end with "MS." Further reading reveals that "MS" stands for Mass Spectrometry. Thus, the principal type of instrumentation to be used in the solar wind analysis phase of the Genesis project is generally based on this old technique that was developed in 1919 and was very, very important in the historical advance of modern physics. Today the technique continues to find wide application in various scientific disciplines. Workers in the field usually shorten "mass spectrometry" to "mass spec." A student text on the fundamentals of mass spectrometry entitled "Mass Spectrometry—A Historic Technique of Great Importance to Genesis" is included as part of this module.

Although mass spectrometry in various forms is a very old, well understood, and highly developed modern technique, analysis of the solar wind samples requires much more precision than is provided by off-the-shelf, commercially available instruments. Hardware and software enhancements of basic mass spectrometric techniques that address the needs of the Genesis analytical program are under development. In most cases, these extensions are very complex. However, the principle behind one of the techniques lends itself to a student activity and is especially relevant to the activities in the <u>Dynamic</u> <u>Design: A Collection Process</u> module that deals with the solar wind collectors. This technique is Secondary Ion Mass Spectrometry, or SIMS for short.

In a SIMS experiment, an accelerated beam of primary ions, such as Cs⁺, is directed toward a surface containing a material of interest. Energy is transferred to atoms in the upper layers of the target material, causing them to leave the surface and enter the gas phase as ions, atoms, or other more complex species. In effect, the primary ions "vaporize" the target material to provide, among other things, "secondary ions." These secondary ions are a mix of elements from both the original collector and the

implanted solar wind. The collection of ions so generated can then be directed into a mass spectrometer where they can be sorted and identified on the basis of their individual masses. This technique is particularly appropriate for near surface analyses like those in the Genesis project where solar wind particles are trapped in the upper layers of a collector matrix.

SIMS Simulation

In this interactive Web-based activity, students are presented with a simulation of a SIMS experiment in which solar wind particles trapped at different depths in the matrix of a collector disk are removed from successive layers of the matrix by increasing the pulse time of the primary beam. The collector matrix material is assumed to be ultra pure, ultra clean silicon in this simulation, but it might be diamond or another pure material. For complete



"maps" of wafer materials used for collection, see the activity <u>"Mapping it Out</u>" from the <u>Dynamic Design:</u> <u>The Cleanroom</u> module. With each increase in pulse time, the secondary ions vaporized out of the matrix are directed into a mass spectrometer. The results of the mass spectrometric analysis are presented in strip-chart form such that the mass of the ions can be determined. Accompanying the activity is a student text describing the analytical issues facing the Genesis scientists and on SIMS. This activity strongly addresses a number of Grade 9-12 National Science Educational Standards in Physical Science and Science and Technology. Physical Science concepts include the structure of the atom and elemental isotopes that students analyze in the simulation. For Science and Technology, the simulation and texts provide a vivid example of how a relatively old technology (i.e., mass spectrometry) continues to contribute to many significant advances in science in general and chemistry in particular.

An Additional Analytical Technique

The only technique expected to be used in the analysis of Genesis samples that does not include some form of mass spectrometry is radiochemical neutron activation analysis, or RNAA for short. Included in this module is a student text that outlines the principles of this technique. You should ask the students to read this student text so that they will have a complete introduction to the methodology to be used by Genesis scientists as they begin to address the final and concluding portion of the Genesis project. It also brings up the point that there may be other techniques useful for analyzing the Genesis samples as researchers implement innovative new ideas for the future.

MATERIALS

- Student Text, "Secondary Ion Mass Spectrometry—SIMS"
- Student Text, "Mass Spectrometry—A Historic Technique of Great Importance to Genesis"
- For each student, team or for the class, a computer that will permit them to access the Web site below. Depending on your particular circumstances you might wish to pursue this activity as a teacher demonstration instead of a hands-on activity. A printer should be made available that permits students to print the results of their simulations.
- For each student, team or for the class, the online interactive simulation activity "<u>Secondary Ion Mass</u> <u>Spectrometry</u>" available at: <u>http://genesismission.jpl.nasa.gov/multimedia/index.html</u>
- A table of the known isotopes of the elements that shows their masses. Alternatively you might direct the student's attention to an authoritative Web site that provides detailed information about isotopic masses and abundances. Available at: <u>http://www.physics.curtin.edu.au/iupac/docs/Final97.pdf</u>
- Student Activity, "SIMS Simulation"
- Student Text, "Magic Bullets for Elemental Analysis"

PROCEDURE

- 1. Before class make copies of the following:
 - Student Text "Secondary Ion Mass Spectrometry—SIMS"
 - Student Text "Mass Spectrometry—A Historic Technique of Great Importance to Genesis"
- Distribute copies of the student texts. Students should read the texts before engaging in the activity. Depending on your students, it may be best to guide the reading of these texts. If you choose to assign independent reading, it would be helpful to use a reading strategy such as SQ3R (Survey, Question, Read, Recite, Review), which is explained in the box to the right.
- 3. After the students have read the texts, gather them together for a class discussion. As an opening to the discussion, remind the class of the primary mission of the Genesis project and set the timeline—i.e. samples have been collected and now have been returned to Earth for analysis. Facilitate a class discussion by challenging students to provide answers to the following discussion prompts:
 - Ask the students if they could see with their naked eyes a sample of matter having a mass of 0.4 mg and seek a class consensus as an answer to the question.
 - b) Once the students have reached the conclusion that 0.4 mg of elemental solar matter is just a tiny speck of material, ask them to imagine spreading and shallowly burying that tiny speck of matter over a surface of 17 ping-pong tables, which approximates the collection surface of the Genesis spacecraft. Then engage them in a discussion of how difficult it is to find particles that are so thinly dispersed and also how seemingly impossible it would be to identify the particles once they have been found. It is like finding a needle in a haystack—only much worse.
 - c) The students should now have an idea of the immense difficulty faced by scientists who have been assigned the responsibility of analyzing the Genesis samples. If the students have carried out quantitative analytical experiments in

SQ3R

An effective strategy for engaging students in the reading process through a series of steps:

- 1. **Survey** before reading. Preview the title, subheadings, and pictures.
- 2. **Question.** Turn title and headings into questions.
- 3. **Read** actively. The search for answers helps to guide the reading.
- 4. **Recite** answers to guiding questions aloud or in writing.
- 5. **Review.** Summarize the information learned through writing, creating a graphic organizer, or participating in a group discussion.

Billmeyer, R. & Barton, M. (1998). *Teaching reading in the Content Areas: If not me, then who?* Alexandria, VA: ASCD.

Teacher Tip

An effective strategy for helping students understand a new concept is to have them develop an analogy to something familiar. Select one or more passages that are especially conducive to analogies. For example:



Marzano, R., Pickering, D., & Pollock, J. (2001). *Classroom instruction that works: Research-based strategies for increasing student achievement.* Alexandria, VA: Association for Supervision and Curriculum Development.

carried out quantitative analytical experiments in a chemistry lab, ask them to discuss whether or not any of the techniques they utilized would suffice for the analysis of Genesis samples.

- d) The solar wind sample will be analyzed in an ultimate "clean room" environment. Why are these conditions necessary? For more information, refer to the text "<u>How Clean is Clean?</u>" from the module <u>Dynamic Design: The Cleanroom</u>.
- 4. If necessary, review with the students the concept of isotopes and remind them of the standard way of designating isotopes, e.g. ¹²C for the mass of the isotope of carbon having a mass of 12 atomic

mass units. It also will be appropriate to review with the students the concept of atomic mass units and the relation of isotopic mass to mass number, the latter being the sum of the number of protons and neutrons in an atom. Establish the fact that for individual isotopes the mass of the isotope and its mass number are essentially the same numerically. If you wish to do so, it might be appropriate to discuss why isotopic mass and mass number are not precisely the same (because of the mass-defect effect), but this is not necessary in order to complete this module. Emphasize again that the Genesis collectors will collect particles of the solar wind, which are atomic, not molecular, in nature. Inform the students that they can assume that the ions entering the mass spectrometer in the simulation have a single negative charge. For more information, see the teacher guide for "<u>Mass, Mass</u>—<u>Who Has the</u> <u>Mass?</u>" from the module <u>Cosmic Chemistry; The Sun and Solar Wind</u>.

- 5. After the students have considered the questions above, give them instructions for loading the online interactive simulation that can be opened at the Web site: <u>http://genesismission.jpl.nasa.gov/multimedia/index.html</u>. Emphasize to students that in the simulation, the mass spectra of negative ions will be studied, although historically mass spectrometry was used largely to identify positive ions. Distribute a copy of the student activity "<u>SIMS Simulation</u>" to each student. For the remainder of this activity, it will be assumed that each student will pursue the online activity independently.
- 6. Ask the students to complete the activity and answer the questions posed on the activity sheet. They should then turn their printed spectra and answers in to you. Post the student results around the room.
- 7. After the students have completed the activity and their results have been posted, return to a classroom discussion that focuses on the following aspects of the Genesis analysis program:
 - a) Determine whether or not there is agreement as to the identity of the solar wind particles found in the simulation. If there is not, have the class discuss why different individuals obtained different results and continue the discussion until consensus is reached.
 - b) Based on the simulation results, it is possible to consider eliminating several isotopes as possible components of the solar wind. Ask the students to ponder the question: What isotopes could possibly be eliminated from further consideration in this <u>hypothetical</u> situation? (Answers should include the isotopes of lithium, boron, nitrogen, oxygen, and neon-20.) You might wish to go ahead and pursue the question of why it would not be safe to conclude absolutely that these isotopes are not components of the solar wind based solely on this simulation. (Answers might include the following: relative abundances are too low to register on the intensity scale shown in the simulation; ions formed by these isotopes during the bombardment by Cs ions are positively instead of negatively charged; some solar wind elements such as oxygen might react with the silicon matrix atoms during the high energy bombardment and not form oxygen ions; and the solar wind particles are buried too deeply to be removed.)
 - c) Might there be other ways to remove the embedded solar wind particles from the collector surface? If so, what suggestions do they have? (Answers might include heating the samples (thermal desorption) and the application of lasers to vaporize a small area.)
 - d) The contaminant particles shown on the surface of the collector were not observed in the mass spectrum, although they could have entered the mass spectrometer. What would this tell us about the nature of these contaminant particles? (Answers might include the following: they have a mass that is too high to be registered on the scale shown in the mass spectra; they have a negative instead of a positive charge under the bombardment conditions used.)
 - e) Small molecules such as HCN have been observed in outer space. If this were one of the "contaminants," where would it show up on a mass spectrum (i.e. at what mass value)?

- f) In an *actual* SIMS experiment on solar wind particles, would one expect to see peaks in the mass spectrum in the mass range 1 to 4? Why?
- g) Tell the students that the relative terrestrial abundances of ²⁴Mg, ²⁵Mg, and ²⁶Mg are 79%, 10%, and 11%, respectively. Ask them to discuss what the mass spectrum in a SIMS experiment might look like in the mass range 23 to 27 based on this information. You might also follow up with a discussion of what the mass spectrum in a SIMS experiment might look like in the mass range 1 to 30 based on this information.
- h) How would the appearance of the mass spectra be changed if the ions carried a charge twice as big as the ones in the simulation? Hint: refer to student text on mass spectrometry.
- One of the collector materials used in the Genesis mission is silicon. During the violent bombardment of the surface of the silicon by the primary ions, some unusual species such as Si² may be formed. Would it be possible to observe these kinds of ions under the proper conditions? How would the experiment have to be changed from that seen in the simulation?
- j) Ask the students to discuss some of the factors that might determine how deeply a given solar wind particle gets buried in the matrix. Particle mass, velocity, and charge should enter into the discussion, as well as collector composition and density. For more information, see the teacher guide for "<u>Modeling Solar Wind Collection</u>" from the module <u>Dynamic Design: A Collection</u> <u>Process</u>.
- k) What will the analysis of the collected solar wind particles tell us about the composition of the Sun and the origin of the Solar System? In order to answer this question, it will be necessary for students to be familiar with earlier modules in the Genesis series. For instance, refer to the teacher guide for "<u>Are We Related?</u>" from the module <u>Cosmic Chemistry: Planetary Diversity</u>.
- 8. **OPTIONAL:** The student text, "<u>Magic Bullets for Elemental Analysis</u>" describes additional techniques for analyzing samples. Distribute copies of the text. Read the text together as a class or individually while employing a reading strategy such as SQ3R, which is described on page 6.

TEACHER RESOURCES

ADVANCED WORK: For advanced students who wish to pursue details of the depth to which ions are buried in the matrix, refer them to the following Web site: <u>http://www.srim.org/SRIM/SRIM2003.htm</u>. This is a site that contains a wealth of information presented at the professional level.

A table of the known isotopes of the elements that shows their masses. Alternatively you might direct the student's attention to an authoritative Web site that provides detailed information about isotopic masses and abundances. Available at: <u>http://www.physics.curtin.edu.au/iupac/docs/Final97.pdf</u>.

NATIONAL SCIENCE STANDARDS ADDRESSED

(Source - National Science Education Standards)

Grades 9-12

Science as Inquiry

- Abilities necessary to do scientific inquiry
- Understanding about scientific inquiry
- Science and Technology

• Understanding about science and technology Earth and Space Science

- The origin and evolution of the Earth system
- The origin and evolution of the universe

Physical Science

- Structure and changes in properties of matter
- Structure of atoms
- Motions and forces

History and Nature of Science

- Science as a human endeavor
- Nature of science and scientific knowledge

(View a full text of the National Science Education Standards.)



Genesis Sample Analysis: The Bottom Line

Mass Spectrometry—SIMS

Education

STUDENT TEXT

Introduction

The Genesis spacecraft has collected pieces of the Sun for more than two years by exposing panels and wafers of various collector materials to bombardment by the solar wind ionic particles while circling a point in space called L1. [See <u>Dynamic Design: A Collection Process</u> and <u>Dynamic Design: The Cleanroom</u> for activities that model how solar wind was collected and the type of collectors and instruments used in Genesis.]

The solar wind particles have struck the collectors and shallowly buried themselves much like a bullet would be buried in a tree trunk after it had been fired at the tree from a gun. On September 8, 2004, the collectors were returned to Earth. What is next? The answer is that laboratory science and technology will take over and focus on the task of identifying and measuring the concentration of the captured solar wind particles. These are expected to consist of the various elements found in the periodic table of the elements [see <u>Cosmic Chemistry: An Elemental Question</u>.]

This task will not be easy. In the first place the amount of collected material is really small because, after all, outer space is an extremely empty place. For comparison purposes we note that near the Earth's surface one finds about 10¹⁸ particles per cubic centimeter (about ¼ teaspoon), whereas in outer space there is only 1 particle per cubic centimeter on the average. To put this another way, it can be said that one "would have to sweep up a volume of space as large as the Earth to collect enough matter to fill a small flower pot" [D. H. Menzel, *The Atlantic Monthly*, November, 1958]. Thus, although the Genesis spacecraft has been in place for many months, it is expected that the <u>total</u> mass of all collected particles will be only around 0.4 milligrams. Or putting it another way, there will be approximately 10²⁰ particles in the entire collection. Working with this miniscule sample, scientists will attempt to obtain *precise measures of solar elemental and isotopic abundances.*

Analyses' Procedures

The analyses obviously require exacting methods. Nevertheless, with one exception (see text "<u>Magic</u> <u>Bullet For Elemental Analysis</u>"), there is a theme that runs through all of the techniques to be used. Turning to the <u>Genesis technical Web site</u> where scientific details are presented, one finds a list of acronyms for the analytical instrumentation that may be applied to the problem. These acronyms are SIMS, GSMS, TIMS, and RIMS. Clearly, the theme focuses on "MS", which stands for Mass Spectrometry (or Mass Spectroscopy). It is the case that MS, in one form or another, is the workhorse analytical tool that will be applied in the analysis of the Genesis samples. The accompanying student text titled "Mass <u>Spectrometry—A Historic Technique of Great Importance to Genesis</u>" provides a basic introduction to the science behind this technique.

Since the solar wind particles will be buried near the surface of the collector materials, it is necessary to somehow remove them from the collectors before they can be analyzed by mass spectrometry. Note that for the most part, the collectors consist of single crystals having an ordered atomic structure often referred to as a "matrix."

One way to accomplish the removal and analysis of the buried particles is through a rather straightforward approach called Secondary Ion Mass Spectrometry [SIMS]. This is regarded one of the most sensitive of all commonly employed surface analytical techniques. The remainder of this text will be devoted to a description of the SIMS experiment.

In a SIMS analysis, the surface of a sample is bombarded with high-energy ions (called "primary ions") that are generated and accelerated to a selected velocity (and kinetic energy) by an "ion gun." This is a violent event comparable to shooting a bowling ball into a brick wall. As the bowling ball (the "primary ion") comes to a halt in the wall (the "matrix"), it creates a hole and delivers its kinetic energy to the bricks in the wall, causing individual bricks and blocks of bricks to be dislodged and ejected from the wall matrix. In the SIMS experiment, bombardment with primary ions analogously leads to the ejection (or *sputtering*) of surface particles, but only from the top few nanometers of the matrix surface. Typically, a series of bursts or pulses of primary ions are swept across the surface under study. The longer pulses sputter particles from lower and lower levels below the surface, leaving deeper and deeper holes (which can be seen under a microscope). The species ejected with each pulse may include ions, atoms, clusters of atoms, and molecular fragments of the solar wind particles as well as of the matrix.



The ejected ions, which are called "secondary ions," are then introduced into a mass spectrometer, where their masses are determined. This leads to their identification. In traditional SIMS, it is only the positive ions that are mass analyzed, but modern instruments permit negatively charged ions to be mass analyzed just as well. The sensitivity of modern mass spectrometers is very high; therefore, only relatively few ions must be sputtered from the sample in order to complete an analysis. The ability to characterize only a few atoms is one of the reasons why mass spectrometry is so important to the Genesis mission. Although the collected atoms have little total mass, there are many of them-plenty in fact for SIMS analysis.

The primary ions employed vary with the goals of

the analysis and the nature of the sample. They have included such species as I⁺, Cs⁺, and O⁻. Shown on the next page are the results of two SIMS investigations of the surface of Teflon® (also known as PTFE), which is a plastic containing carbon and fluorine. Note the differences between the two spectra. One of them presents the masses of the positive ions sputtered from the surface and the other shows the masses of the sputtered negative ions. Also note that in both cases single ions such as F⁻ and C⁺ were identified as materials blasted off the surface along with a collection of positively and negatively charged polyatomic fragments such as CF⁺. The height of the line (peak intensity) corresponding to the mass of each species provides an indication of the stability and abundance of that species. In these spectra several unidentified minor constituents present in low abundance also can be seen, as indicated by their low peak intensities. Often the origin of these minor components of the spectrum is not clear, as they might arise from impurities in the PTFE, from surface contamination, or from instrumental "noise."



The SIMS analysis of Genesis samples will involve a variety of primary ions, and both positive and negative ion spectra will be recorded.



Education

Genesis Sample Analysis: The Bottom Line

Mass Spectrometry– A Historic Technique of Great Importance to Genesis

STUDENT TEXT

History of Mass Spectrometry

Mass spectrometric techniques have played an important role in science (particularly in chemistry), and it is very satisfying to recognize that this old and historically important technology is likely to play a major role in the final, or analysis, phase of NASA's Genesis project.

Francis Aston in Cambridge, England developed an instrument called a mass spectrograph in 1919. The importance of this new technology was immediately recognized, and Aston was awarded the Nobel Prize for his work in 1922. So, what does a mass spectrometer (in modern terminology) do, and how does it work? In the following few paragraphs, the *classical technology* will be described, and then a few words will be devoted to modern instrumentation of the type that may be used for analysis of Genesis samples after they are returned to laboratories here on Earth.

How Modern Mass Spectrometers Work

Mass spectrometers permit the experimental determination of atomic and molecular masses with great accuracy. Aston's mass spectrometer had a precision of one part in 10,000, which was sufficient for him to discover the isotopes of many elements. Modern instruments are even more precise.

Mass spectrometers operate under conditions of high vacuum, typically 10⁻⁸ Torr. (In comparison, the pressure in outer space may be on the order of 10⁻¹² Torr.) Such high vacuum conditions are necessary in order to limit the effects of air molecules, which would severely interfere with the analysis if they were present at elevated levels. Under the high vacuum conditions, small gaseous samples to be analyzed are fed into the spectrometer's ionization chamber where they are exposed to a beam of rapidly moving, energetic electrons generated by an electron gun. The samples can be in the form of a gaseous element such as neon, the vapor of a solid or liquid element such as mercury, or even the vapor of a molecule such as water or methane. In fact, with modern technology it is possible to transform a wide variety of materials, including mixtures, into a gaseous form that can be directed into a mass spectrometer.

When an atom or molecule introduced into the ionization chamber collides with an accelerated electron from the electron gun, an electron is knocked out of the sample, leaving it with a positive charge. In other words, electrically charged gaseous cations are formed. (*Note: In modern mass spectrometry, it is not uncommon to create negative ions for study instead of positive ions. The analysis principles are the same for both types of ions.*)

The ions so formed are then pushed out of the ionization chamber by an electric field applied between two metal grids. This is a simple application of Coulomb's law in that the positive grid repels the positive ions, while the negative grid attracts them, providing a net acceleration toward the negatively charged grid. The negative grid, which is full of holes, allows the accelerated ions to pass through it and exit the ionization chamber. The kinetic energy of the exiting ions can be pre-determined by the applied electric field of the grid. Now, recall that the kinetic energy of a moving particle is directly proportional to its mass

and its velocity squared. So in the final analysis, the velocity reached by an ion as it is accelerated by the electric field to a fixed kinetic energy is determined by its mass, with the lighter ions reaching higher speeds than the heavier ones. It also is the case that the ions accelerated from the sample generate a magnetic field of their own (in common with all moving charged particles).



In the mass spectrometer, the accelerated ions are next passed through an externally applied magnetic field. The magnetic field of each ion interacts with the external field, the net result being that the trajectory of the charged particle is circularly bent to an extent that depends on its speed (and therefore its mass). If the beam of a mixture of ions having different masses then is allowed to impinge on a photographic plate, it will be seen that ions of different masses converge at different points, corresponding to the different radii of the semicircular paths taken. The mathematical equation that describes this phenomenon is:

$$m/e = H^2 r^2 / 2V$$

where m is the mass of the ion, e is the charge of the ion, H is the magnetic field strength, r is the radius of the semicircular path, and V is the accelerating potential.

The radius of the semicircular path clearly is proportional to the mass of the particle. A photographically detected mass spectrum of natural germanium obtained under conditions of constant magnetic field and accelerating potential would look like the figure shown here:



The relative abundances of the ions can be determined from the densities of the photographic images they produce.

Since H, V, and r can be varied or determined experimentally, the ratio m/e can be determined in one of several fashions. In a typical modern mass spectrometric experiment, the strength of the external magnet is slowly varied, causing the paths of the various ions to sweep past an exit point where the mass spectrometer's ion detector is located. In other words a signal is produced at the detector when the magnetic field is just strong enough to bend the pathway of ions of a given mass so that they arrive at the detector. The mass of an individual ion may be calculated from the accelerating voltage and the strength of the magnetic field required to produce the signal. The mass spectrum is a graph of detector signal versus the sweep of magnetic field strength. The positions of the peaks on the graph are used to

calculate the masses of the ions, and the relative heights of the peaks indicate the relative proportions of the ions of various types that were in the sample. If the ion has a charge of one then the mass is determined uniquely.

Note that a dipositive ion of mass 64 gives rise to the same value of m/e as a monopositive ion of mass 32. Under the conditions usually selected for operating a mass spectrometer, the ions produced are singly charged. Figure 1: Below is a diagram of one of the very advanced, highly specialized, and complex SIMS instruments that will be utilized in the analysis phase of the Genesis mission.



Alternatively it is possible (and actually more convenient) to vary the accelerating voltage while keeping the magnetic field constant, leading once again to a spectrum that can be analyzed to provide ion masses and relative abundances. Modern mass spectrometers are linked to computers that do the analysis and produce a graph (spectrum) showing mass in atomic mass units (amu) plotted on the x-axis and relative intensity on the y-axis. The relative intensity is a measure of how many ions of a given mass are detected. Note also that units of daltons are sometimes used in place of amu: 1 amu = 1 dalton. With modern low-resolution instruments, one can expect to obtain accuracy of plus or minus 1 amu up to 1000 mass units. High resolution instruments can provide 2 to 3 parts per million (ppm) accuracy up to masses of 3000 amu.

There are many variations on the theme of mass spectrometry, but all of them are fundamentally based on the principles outlined above—ions are generated and are then sorted and detected according to their masses. Of particular note is a time-of-flight instrument in which the ions are produced in spurts and allowed to diffuse toward the detector in a straight line. No magnetic field is used. The heavy ions move more slowly than the light ones, and the spectrum is a plot of intensity versus time of flight of the particle. Some Genesis sample analyses will employ this or another technique instead of the traditional method described above.

Mass spectrometers will play a major role in the final phase of Genesis when the samples are returned to Earth for analysis. This is because the samples will be very small, in some cases amounting only to perhaps a few atoms. Exquisitely sensitive instruments such as new generation mass spectrometers will be required in order to maximize the information obtained from these precious solar wind samples.



STUDENT ACTIVITY

PART 1: Data Collection

SIMS: Secondary Ion Mass Spectrometry



Simulation Features

In Part One of this activity, you will carry out a simulated SIMS experiment. Make sure you have read the two student texts handed out by your teacher before pursuing this activity. After you receive instructions from your teacher about accessing the Web site where the activity is located, open the site: http://genesismission.jpl.nasa.gov/multimedia/index.html. There you will find a graphic simulation of a SIMS experiment that includes the following features:

- a) On the top left—an ion gun that generates primary ions and accelerates them toward the surface of the sample to be studied.
- b) On the left center—buttons that permit you to select a *simulation* for one of three primary beam pulse times. Note carefully that in this simulation the time that ions impinge on the sample does not actually correspond to the time selected by the buttons because this is only a representation of the actual experiment.

- c) On the lower left inside of a box—a legend that identifies the various components of the wafer used for collection, including captured solar wind particles.
- d) On the far right—a representation of a collector wafer from one of the Genesis collector arrays. For purposes of this simulation, assume that the collection wafer itself is composed of extremely pure silicon that contained no impurities before it was exposed to the solar wind.
- e) In the middle of the display—a representation of a very small surface segment of the collection wafer containing the solar wind particles defined in the legend. This is the surface that will be struck by primary ions during the analysis.
- f) To the right, immediately above the surface—a representation of a mass spectrometer into which particles are directed as they are blasted out of the collector matrix by the primary ions.
- g) Immediately above the mass spectrometer—the mass spectrum, which is the graphical output of the mass spectrometer. In other words, this is a graphical representation of the mass of the observed ion versus the intensity (number of counts) that each ion registers on the detector of the mass spectrometer. For purposes of this activity, the intensity can be taken as a direct measure of the relative abundance of a particular ion. Note that the mass spectrum can be saved and printed in an expanded form so that you can use it for more detailed study.

Procedure

- 1. Once you have a clear understanding of the layout of the simulation, you can begin the simulation by pushing the "Clean Surface" button. This sweeps the surface clean of contaminants and readies it for analysis. Now, begin the actual simulation of the analysis for solar wind particles by "pushing" the button for the shortest primary beam pulse time. Observe what happens to the surface of the sample as it is bombarded by the primary ions. Note that the mass spectrometer analyzes the sample and provides a mass spectrum. Print out a copy of this spectrum and label it carefully so that you will know that it was provided at the selected pulse time. On the printout, write down the number of particles of a given color that enter the detector of the mass spectrometer.
- 2. After you have printed the mass spectrum, repeat the experiment by "pushing" the button for the next pulse time. Once again observe what happens to the particles. As you did before, print out and label a copy of the mass spectrum. Also write down the number of particles of a given color that enter the detector of the mass spectrometer.
- 3. Finally repeat the experiment once again by using the longest pulse time. Print out and label the mass spectrum obtained. Record the number of particles of a given color.
- 4. At this point, you are through collecting data from the simulated SIMS experiment. Return to your desk with your three mass spectra and proceed with Part Two of the activity.

PART 2: Data Interpretation

5. Interpretation of mass spectra:

a) First, turn to the spectrum obtained with the shortest pulse time and determine the masses of the particles recorded by the mass spectrometer, assuming that the observed particles carried a single negative charge. Write the observed masses near the top of the mass spectrum that you printed out. Based on the observed masses of the particles, identify them, and then write the name of the element adjacent to where you recorded the observed masses. To aid in doing this, ask your teacher for a table of isotopic masses. Next show the chemical symbol of the isotopes that you have identified, complete with the correct mass number label. Finally indicate which substances were present in the highest and lowest amounts, respectively.

- b) Next turn to the spectrum obtained with the intermediate pulse time and repeat the instructions provided in part a).
- c) Finally, turn to the spectrum obtained with the longest pulse time and repeat the instructions provided in part a).
- 6. **Questions**—Write your answers to the following questions in the space provided.
 - a) Which of the particles do the blue, red, and yellow circles represent, respectively, in the simulation?

b) If one of the colored circles represented ³²S, would you have seen it in the mass spectrum of the simulation? Why or why not?

c) Why were the sputtered particles of matrix material (silicon) not recorded on the mass spectrum (as presented in the simulation)?

d) Hydrogen is expected to be a component of the solar wind. If the yellow circle represented hydrogen, discuss how the spectrum obtained with the longest pulse time would be changed in appearance.

e) Might an even longer pulse time (or perhaps a higher primary gun energy for the same time) provide additional information about the solar wind? Why or why not?

f) Why would diamond not be a good choice for the wafer material if one were interested in collecting the isotopes of carbon for detailed analysis?

7. Put your name on your spectra and answer sheet and hand them in to your teacher.

GENESIS SEARCH FOR ORIGIN

Genesis Sample Analysis: The Bottom Line

Magic Bullets for Elemental Analysis

Education

STUDENT TEXT

Historical Background for Neutron Activation Analysis

The early years of the 20th century saw unparalleled developments in physics that continue to fuel scientific advances in this new century. In the student text entitled "<u>Mass Spectrometry—A Historic</u> <u>Technique of Great Importance to Genesis</u>," we learned that the fundamental idea, which is going to be exploited extensively for the analysis of Genesis samples, was recognized with the award of a Nobel Prize in 1922. Ten years later another immensely important discovery was made that also will play a key role in the analysis of Genesis samples. This second discovery also led to the award of a Nobel Prize, this time to the English physicist James Chadwick in 1935.

What was this discovery? Before answering the question let us backtrack a bit. Growing out of the work of Madam Curie and her husband Pierre, was the discovery of the alpha particle, which was shown by Earnest Rutherford in 1909 to be a doubly ionized atom of helium. Once the positively charged alpha particle was identified, physicists immediately visualized it as an "atomic sized" bullet and busied themselves with experiments in which they fired alpha particles at other atoms in an effort to break things or change one atom into another. The experiments met with limited success. The problem with using alpha particles as atomic bullets was their positive charge—at close distances they were strongly repelled by the positive charge of target atomic nuclei and were unable to breach the electrostatic defenses of heavy atoms. Nevertheless, Rutherford did succeed in transforming nitrogen into oxygen in 1919 by alpha particle bombardment. This now brings us back to Chadwick.

What Chadwick discovered in the early 1930's was another atomic bullet. But this one did not carry a charge. He discovered the neutron and this particle indeed proved to be a truly magic bullet.

Scientific Development

Physicists immediately recognized that in the neutron they had a wonderful new key for unlocking atomic secrets, since an *uncharged* neutron bullet, unlike the positively charged alpha particle, should easily penetrate a positively charged atomic nucleus. Enrico Fermi, an Italian physicist, soon became an enthusiastic atomic marksman and systematically started to work his way through the periodic table, shooting neutrons at larger and larger atoms. Fermi understood that when a nucleus absorbs a neutron its atomic number remains unchanged (the nuclear *charge* is not altered), but its mass number goes up by one unit, corresponding to the mass of the absorbed neutron. Thus, oxygen-17 becomes oxygen-18 and so on. Typically the neutron also delivers energy to the target nucleus and "excites" it. The excited nucleus of the new isotope usually reaches a more stable state by emitting energy in the form of one or more gamma rays (a so-called "prompt" gamma ray). However this is not the end of the story.

The overall process is diagrammed below.



In 1936 the scientists Hevesy and Levi realized the potential of neutron bombardment for qualitative and quantitative identification of elements present in unknown samples. That is, they saw the analytical potential of carrying out nuclear reactions on samples followed by measurement of the induced radioactivity. This realization was the birth of Neutron Activation Analysis (NAA), a technique that may be used in one form or another for quantitative multi-element analysis of some of the Genesis samples.

Neutron Activation Analysis Technology

In practice, NAA begins with neutron bombardment of a sample to convert stable isotopes in the sample to radioactive isotopes. The neutrons are usually obtained from nuclear reactors (a very specialized technique). Following removal of the sample from the reactor, the radioisotopes created during the irradiation process decay with the passage of time. The resultant gamma rays are emitted at characteristic energies and are indicative of specific radioactive isotopes. This is made clear by choosing sodium as an example. Natural sodium, sodium-23, is converted first to radioactive sodium-24 in a beam of neutrons. The sodium-24 then ultimately decays to stable magnesium-24, with the emission of <u>characteristic</u> delayed gamma rays having energies of 1368.53 and 2754.09 kilo-electron volts. Thus, the observation of delayed gamma rays coming from an irradiated sample at these specific energies is indicative of the presence of sodium. Quantitative measurements by gamma ray spectroscopy, followed by data reduction of the spectra, provide the concentration of the various elements in the samples under study. Below is an example of a gamma ray spectrum from an irradiated pottery sample (irradiation time, 5 seconds). Note the wide variety of elements detected from a single measurement.



NAA is a versatile technique that offers high sensitivity on the order of parts per billion or better and only small samples are required. It is not suited for the analysis of all of the elements, but approximately 60 of them can be analyzed quantitatively. It basically is a non-destructive technique and the sample may be used for other purposes after NAA. NAA clearly offers many advantages.

A less frequently applied technique called Radiochemical Neutron Activation Analysis (RNAA) may be used in the Genesis project. This technique employs chemical separations after the irradiation step to remove interferences or to concentrate isotopes of interest. However, the basic principles outlined above still apply.