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Materials Characterization Activities for "Take Our Sons & Daughters to Work Day" 2013

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Materials Characterization Activities for "Take Our Daughters & Sons to Work Day" 2013

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Abstract

We created interactive demonstration activities for Take Our Daughters & Sons to Work Day (TODSTWD) 2013 in order to promote general interest in chemistry and also generate awareness of the type of work our laboratories can perform. "Curious about Mars Rover Curiosity?" performed an elemental analysis on rocks brought to our lab using the same technique utilized on the planet Mars by the NASA robotic explorer Curiosity. "Food is Chemistry?" utilized a mass spectrometer to measure, in seconds, each participant's breath in order to identify the food item consumed for the activity. A total of over 130 children participated in these activities over a 3 hour block, and feedback was positive. This document reports the materials (including handouts), experimental procedures, and lessons learned so that future demonstrations can benefit from the baseline work performed. We also present example results used to prepare the Food activity and example results collected during the Curiosity demo.

ACKNOWLEDGMENTS

The authors would like to acknowledge Dora Wiemann for her kind suggestions and thoughtful reviews of our procedures and handouts; and Richard Harrison for helping run the LIBS instrument during TODSTWD. We would also like to thank all the kids and parents that attended and note that we appreciated the constructive feedback provided by several parents.

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NOMENCLATURE

DART	Direct Analysis in Real Time
JPL	Jet Propulsion Laboratories
LIBS	Laser Induced Breakdown Spectroscopy
MSL	Mars Science Laboratory
MS	mass spectrometry
NASA	National Aeronautics and Space Administration
SNL	Sandia National Laboratories
TODSTWD	Take Our Daughters & Sons to Work Day

1 INTRODUCTION

Every year Sandia National Laboratories hosts a day for employees to bring children (“sons and daughters”) to the labs for demonstrations and learning activities. In order to promote general interest in chemistry and also generate awareness of the type of work our laboratories can perform; we created two demonstrations called “Curious about Mars Rover Curiosity?” and “Food is Chemistry?” A schedule of events is published prior to the event by the organizers with short descriptors along with location and time. Our summary texts were as follows:

> *1. Curious about Mars Rover Curiosity? Bring us a rock (smaller than your fist) and we’ll show you how Curiosity is testing for minerals and chemical elements on Mars! Watch a ChemCam-like analysis of your rock.*
>
> *2. Food is Chemistry? Come see how a mass spectrometer in our analytical chemistry lab can be used to solve a mystery, just like on TV. So did you eat any molecules today?*

Our goal was to have demonstrations that would be fun and engaging for both the children and their parents or hosts and spark curiosity for self-study afterward. With success it would allow for multiple levels of discussion during the activity and be interesting to a wide variety of visitors (age, background, science education, etc.). Participation for the activities was approximately 85 children (Curiosity) and 50 children (Food). Based upon feedback and our observations during the event we believe these demonstrations achieved most of our goals.

This document reports the experimental procedures, the handouts, and lessons learned so that future activities can benefit from the baseline work performed and therefore also be improved.

2 CURIOUS ABOUT MARS ROVER CURIOSITY?

2.1 Background

The Mars Robotic explorer (Rover) named Curiosity launched from earth November 26, 2011 and landed on Mars August 6, 2012. A variety of chemical analysis tools are on board with capabilities to test the rocks and soil of Mars. The concept of LIBS for space exploration was published as early as 2000 (1).

One of the tools on Curiosity is called the ChemCam, which uses a technique called Laser Induced Breakdown Spectroscopy (LIBS) to test the elemental composition of rocks and soil. The general operation is shown schematically in Figure 1. A focused laser pulse is directed at the object, creating a spark plasma that emits wavelengths of light characteristic of the elements. A spectrometer separates the light so that the elements can be identified.

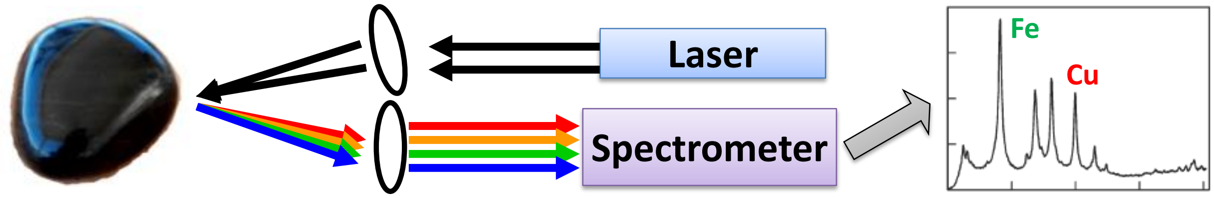


Figure 1: General schematic of a LIBS experiment.

LIBS has several advantages for chemical analysis – there is no sample preparation required and the analysis can be performed at a distance and in seconds. For example, Curiosity is performing LIBS on rocks that are 7 meters away!

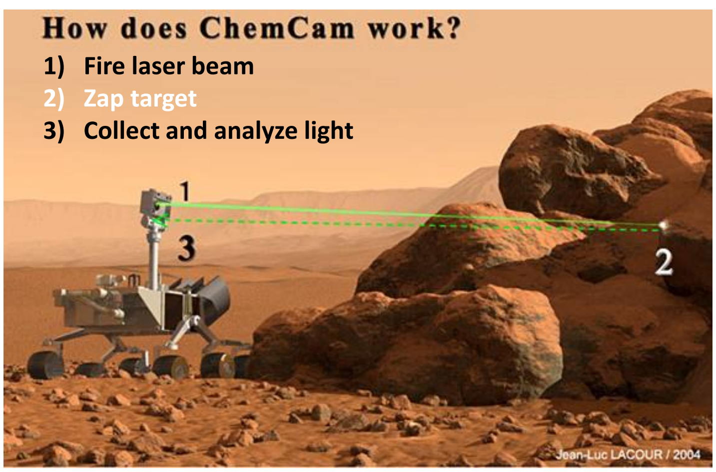
With the exception of distance, these are the same advantages that make LIBS an excellent tool for a science demonstration. We have performed demonstrations using coins, jewelry, and powders such as sand. For powders the sample is held via double sided tape to a disposable sample carrier (Si or Al wafer).

2.2 Door & Hallway Sign

The figure below was printed on a full page and was posted on exterior building doors and outside the laboratory. In order to convey the demonstration in a simple manner, we labeled an image obtained from the JPL website (2).

TODSTWD: Curious About Curiosity?

1-3 p.m., Bldg. 701 Room 2307 (south side, 2nd floor)



Curious about Mars Rover Curiosity? Bring us a rock (smaller than your fist) and we'll show you how Curiosity is testing for minerals and chemical elements on Mars! Watch a ChemCam-like analysis of your rock. Only takes 10 minutes.

Brought to you by Materials Characterization & Performance Dept. 1819

Figure 2: Door and hallway sign for "Curiosity" activity.

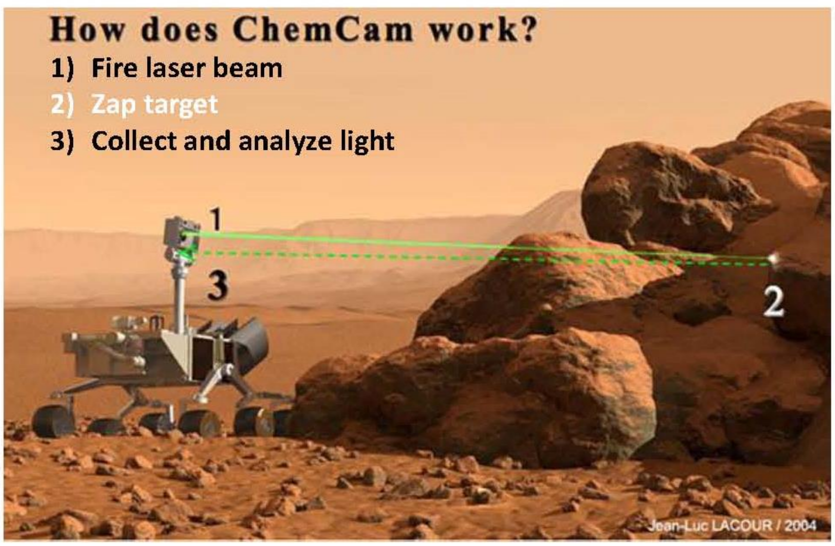
2.3 Handout

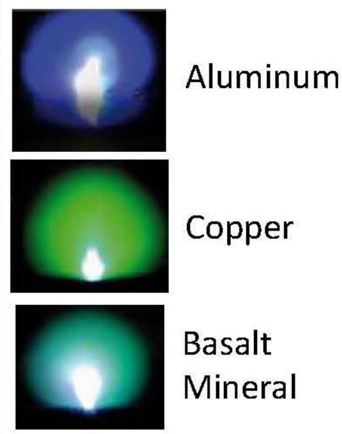
The following pages contain images of the handout created for this activity. It was printed double sided and in color and with 0.5 inch top and bottom margins. The ChemCam spectra were obtained via the JPL photo archive and modified by cropping and labeling to meet the needs of a short handout (3, 4).

Curious about Curiosity?

Welcome to Sandia's Materials Characterization and Performance Department. We test gases, liquids, and solids for Sandia, looking for chemical elements and compounds. One of our pieces of equipment is called a LIBS (Laser Ionization Breakdown Spectroscopy) analyzer.

NASA's Mars robotic explorer "Curiosity" also has a LIBS analyzer named ChemCam. NASA uses the LIBS for detecting chemical elements in Martian rocks and soil – without touching them! The process is illustrated below: 1) ChemCam shoots a high energy laser (~1 million light bulbs!) at the target that 2) creates a spark that emits light which 3) is collected and analyzed. The light tells scientists what chemical elements are present. Learn more about Curiosity and ChemCam at these websites: http://mars.jpl.nasa.gov/msl/ and www.msl-chemcam.com/index.php.





Curiosity's cameras look for mineral features and then test them with ChemCam, which has detected the elements Si, Al, Mg, Ca, and others. The elements detected help NASA scientists figure out the Martian minerals. Some of these minerals are similar to those seen on Earth. Elements were also detected on Mars by the rovers Spirit and Opportunity (using other instruments). These include Na, K, Ti, Cr, Mn, Fe, Ni, and Zn.

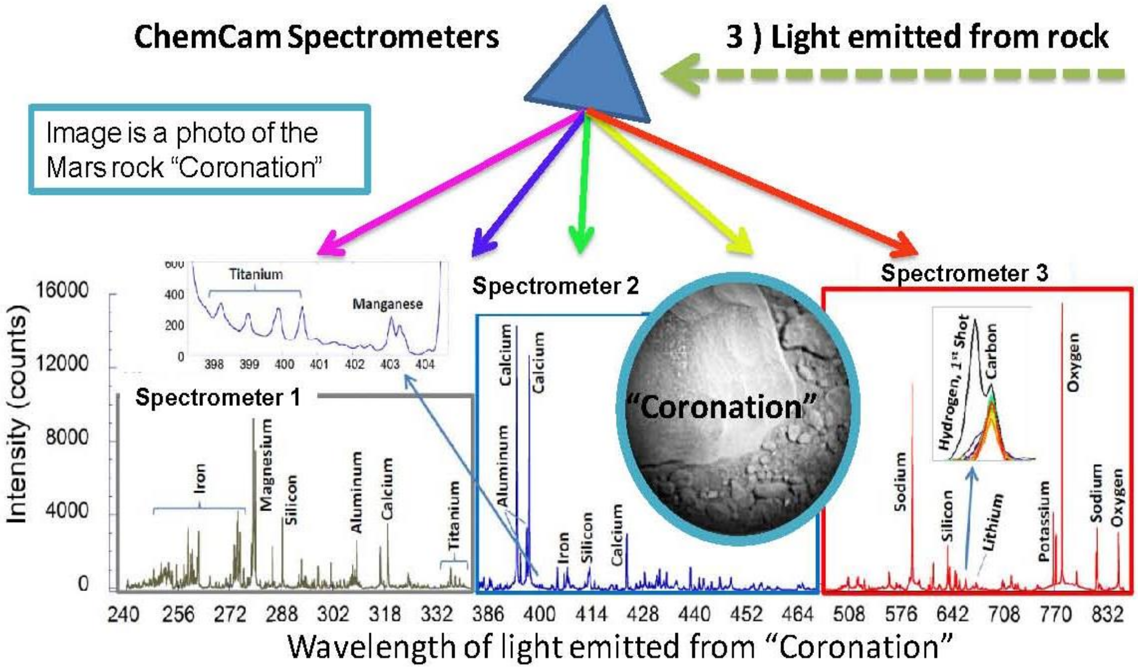
When Curiosity tests a Martian rock, NASA gives it a name. Data from the rocks named "Coronation" and "Crest" are shown on the next page.

Si = Silicon	K = potassium
Al = Aluminum	Ti = Titanium
Mg = Magnesium	Cr = Chromium
Ca = Calcium	Mn = Manganese
Na = Sodium	Fe = Iron

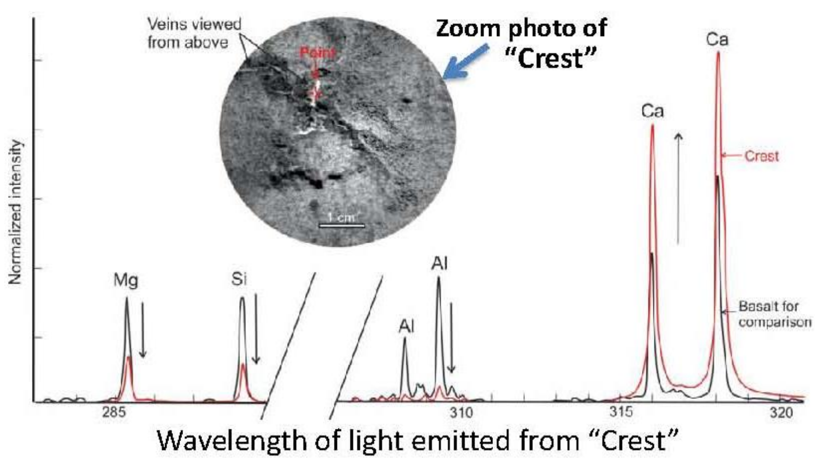
ChemCam uses three spectrometers to separate the light emitted from a rock into its individual wavelengths (called a spectrum).

Image credits: J-L Lacour/CEA/French Space Agency (CNES) and Sirven et al, JAAS.

Each element emits very specific wavelengths of light. The elements found in "Coronation" are labeled.



This a small section of the spectrum of the Martian rock "Crest" (red) compared with the Earth mineral basalt (black). Both have Ca, Al, Si, and Mg but in different amounts.



In our Laboratory on Earth

Can you see the spark when the laser fires?

What elements were detected in your rock?

Does your rock have any of the same elements found on Mars?

Can you find your rock's elements on the periodic table?

Can you find the spot where the laser blasted (melted and ablated) your rock?

Data/Image Credits: NASA/JPL-Caltech/LANL/CNES/IRAP

2.4 Logistics and Procedure(s)

As soon as the site-wide schedule was posted, the location and time was verified. The afternoon before the scheduled time, door signs were posted on the exterior building doors. The main entry of building 701 has a two-stage doorway such that signs were posted on the second door and were out of the weather. People started lining up in the hall approximately a half hour early.

Approximately six children at a time (and their accompanying adults) were brought into the laboratory and positioned so that they could see the chamber (see photos below). Some additional background about LIBS and the Mars rover, not detailed on the handout, was discussed. Children were asked to focus on the rock rather than the computer monitor in order to see the spark. Additional “lab” rocks were available, consisting of a variety of colors and rock types. The activity announcement worked well from the standpoint that children brought in rocks of appropriate size to fit in the chamber.

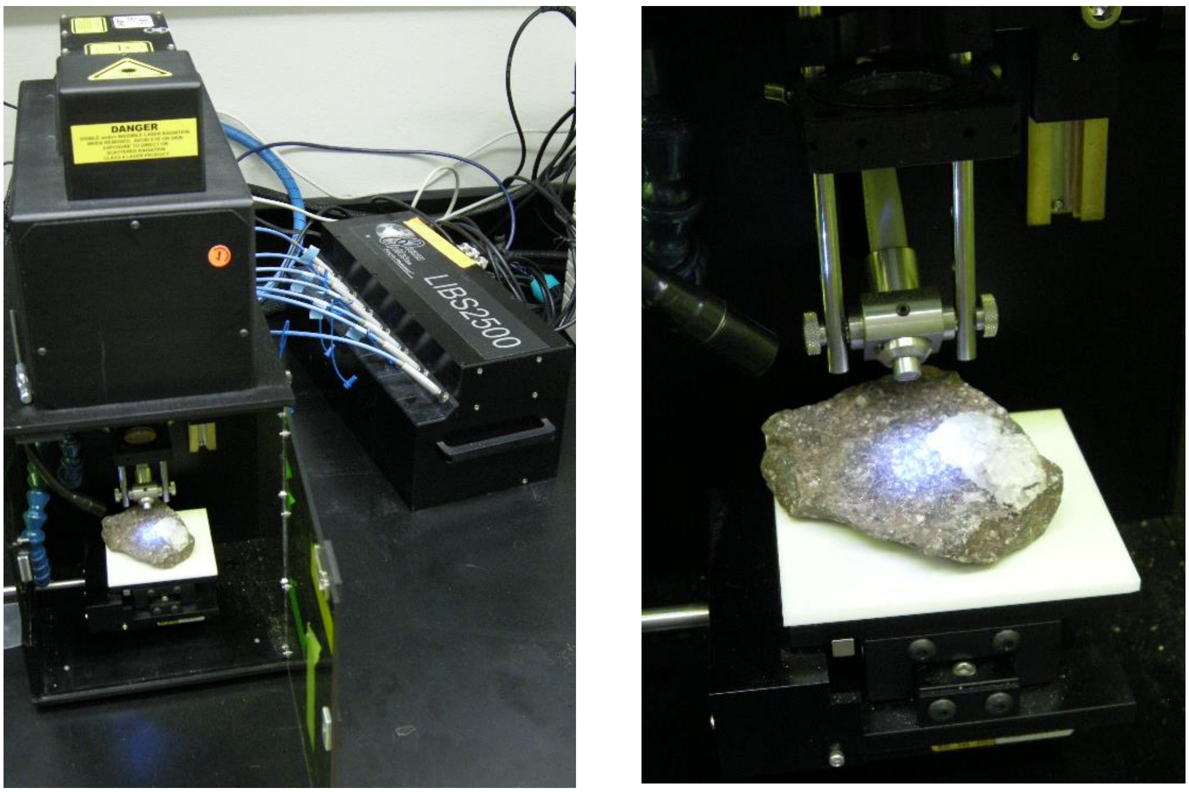


Figure 3: Photo of LIBS chamber (left image), laser is on top while spectrometer is box with blue fiber optic cables connected), and close-up photo of rock inside (right image).

Each child’s rock was subsequently placed in the chamber and the laser lens was adjusted in order to obtain a good spark and data. A height guide inside the chamber was used as an aid for this purpose. Once the chamber was closed and the interlock clear, the shutter was opened and the children were asked to watch the rock. Our system is considered a Class 1 laser system; therefore laser goggles are not required because the laser radiation is completely contained. Each rock was “zapped” two or three times, with focal adjustment as needed for good emission signal. In some cases with rocks that had clear distinctive areas, two areas were tested. Once a “good” spectrum (not necessarily dominated by Ca or Na) was obtained, the data file was saved as the [child's name.spc] using the LIBS system (LIBS2500 sample chamber and spectrometer, Ocean Optics, Dunedin, Fl). The spectrum was then opened using Grams spectral software (Thermo Scientific, San Jose, CA) and printed out using a template that would include the filename (and thus the child's name) on the printout. The laser was set for 95% power and a 2 microsecond Q-switch delay; no bath gas was used.

In some cases unusual peaks recognized by the operator were mentioned or pointed out; likewise if an unusual rock was tested, for example one with blue crystals, the operator would attempt an "on-the-fly" detection of elements.

After the whole group had printouts, they were sent to the "data processing center" in the chemical-free zone (office) adjacent to the lab. Pre-printed transparencies were available with reference spectra printed for the following elements: Zn, S, Si, Fe, Cu, Cr, K, Al. Spectral lines were highlighted in color to help them stand out, and the children were instructed to place the transparencies over their own data and any overlap between their data and the reference indicated that element was present in their rock.

Data processing was intended as an activity that would allow those with the interest and energy to spend more time and the remainder could move on to another activity. The average time spent on analysis per person is not known. Two 10 year-old helpers coached in the process provided assistance to the visitors.

2.5 Results

Approximately 85 children came through the laboratory. Approximately 75% brought their own rock. The activity seemed to keep the interest of a wide range of children, and many of the adults as well. The figure below illustrates the data obtained for "Cole's Rock" and is approximately what his printout contained. The ubiquitous sodium peak is labeled here. The figures that follow zoom in on the spectral ranges and example peaks that were in the printed library spectra for children to match.

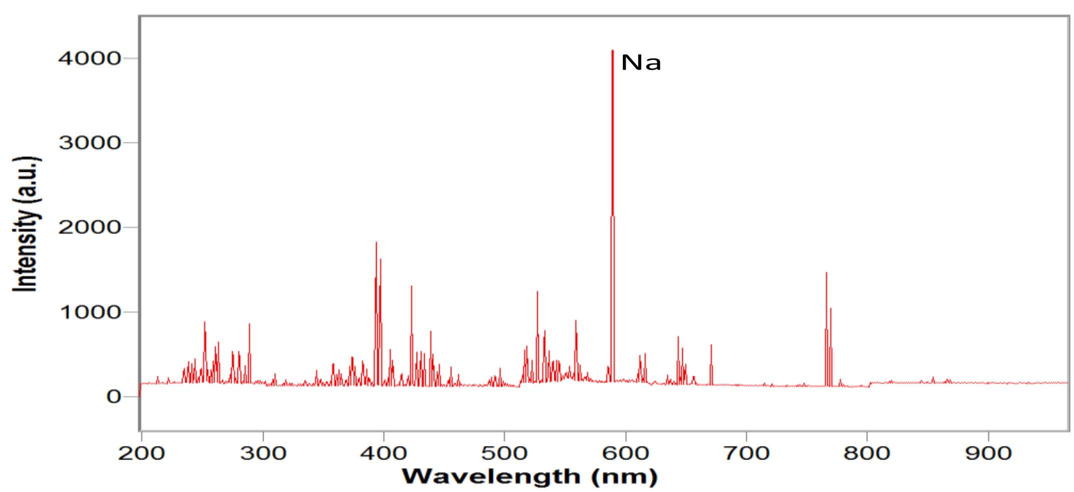
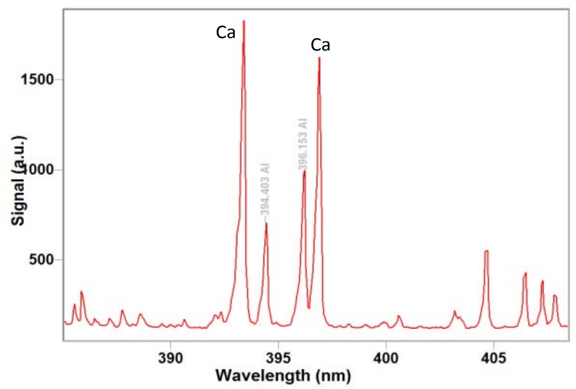
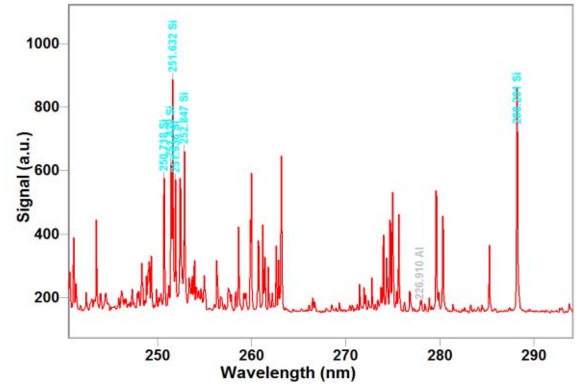


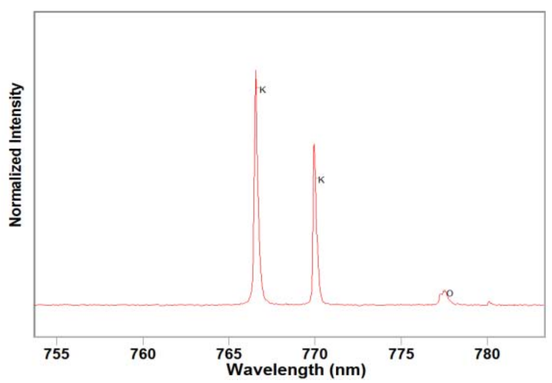
Figure 4: LIBS spectrum of "Cole's Rock" showing full wavelength range.



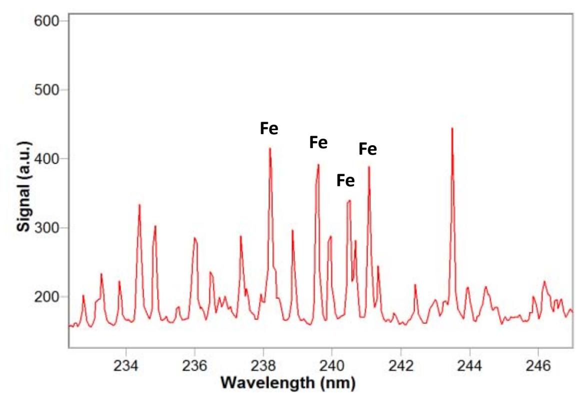
Aluminum wavelengths: 394.40, 396.15 nm
Calcium wavelengths: 393.39, 396.93 nm



Silicon wavelengths: 250.72, 251.43, 251.63, 251.94, 252.85, 288.20 nm



Potassium wavelengths: 766.54, 769.97 nm



Iron wavelengths: 238.20, 239.56, 240.49, 241.05 nm

Figure 5: LIBS data from "Cole's Rock", showing example regions used to detect common elements and specific wavelengths for Al, Ca, Si, K, and Fe.

As expected, a wide variety of spectra were obtained. Several examples that seemed unusual, given the rocks tested, are shown below. Barium was observed in about 10 rocks, chromium was only observed in 1 or 2, silicon was not typically observed alone, and copper was only observed in one rock.

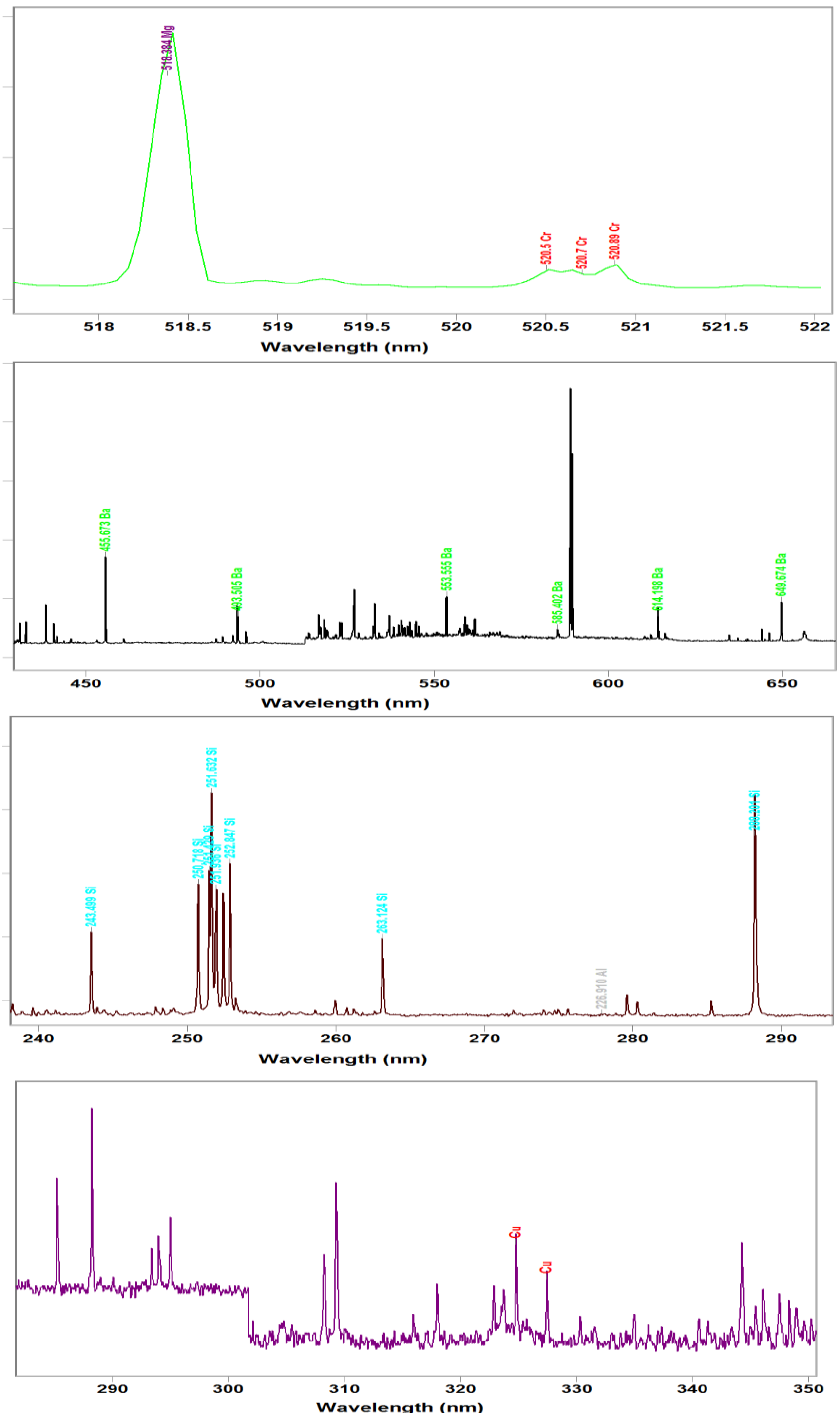


Figure 6: LIBS spectra (intensity versus emission wavelength) of four rocks that showed uncommon features or elements, top to bottom: chromium (Conner's), barium (Emmaline's), silicon without other elements (Gwen's), and copper (Logan's).

2.6 Lessons Learned

Performing the demo in groups worked well, as that kept the waiting time down and allowed each child to observe multiple tests (sparks!). Six (not including parents) was a good number with respect to the ability of everyone to see the demo. Printing the data worked well as they could then have a souvenir of the event. A fast printer was important to high throughput.

The sound and visual of the laser spark were the favorite part of the activity.

While the overall demo seemed to generate lots of excitement and discussion, there were things "behind the scenes" that could be improved. A stronger connection between the spectra in the handout sheet and the spectra obtained on their rock could be made. Some parents commented that this was not clear. This could be achieved by pausing and pointing to the just-generated peaks on screen and identical ones in the handout (for example Ca, Na, O, Fe would be common). A bigger computer screen would be useful (a laptop was used). Viewing issues should already be alleviated as it was determined that the OOLIBS software would operate under Windows 7 (32 bit mode) thus allowing a full-sized desktop computer to be used.

The biggest lessons learned were that crowd management and the data analysis process need improvement. Wait times of approximately 20-30 minutes were experienced and based upon queries on TODSTWD and parent emails later; we don't believe wait times longer than 30 minutes were experienced. This wait time is not excessive compared to other TODSTWD activities but improving the "boredom" factor while waiting is desirable. The following ideas are suggested as improvements:

1. Video or computer video set up in hallway with Mars Rover or Chemcam videos (available on the internet).
2. Handouts should be located and visible in the hallway so that they can be picked up and read prior to lab entry (rather than handed out).
3. Enlist additional help for discussion(s) in the hallway prior to lab time.
4. Use argon bath gas for more intense spectra.
5. Use OOLIBS software feature that shoots the rock several times to "clean" surface grime from rock – this will allow more rock-specific elements to be observed and would provide more sparks to observe.
6. Create a small specialized library for OOLIBS to use that will label a short list of peaks and/or labels them as an element that is available in the transparencies so they know that a particular line can be found in the "analysis library". This could be achieved using an elemental label "L" for "library". And use thicker lines on transparencies.
7. Create a better library (with fewer lines to compare) or perhaps remove the participant search from the activity.
8. Post instructions for adults to read/follow to assist their children in this activity, or recruit additional adults. The crowd and throughput was too much for younger helpers. Perhaps this activity could be moved to the hallway or a conference room (or both) with more tables.
9. Rehearse a semi-scripted presentation for the laboratory to keep a consistent flow. This script would be useful for any help recruited and would also provide a mechanism for improvements to be made.

3 FOOD IS CHEMISTRY?

3.1 Background

A mass spectrometer (MS) is used by all sorts of chemistry and testing labs in a variety of applications from water testing or drug enforcement to food manufacturing. All molecules have their own unique mass, and mass spectrometers can measure them and even break them apart to determine their structure. All mass spectrometers manipulate molecules as charged particles and thus every mass spectrometer has a mechanism to create charged molecules (ions). For this demonstration, we use a technique called Direct Analysis in Real Time (DART). DART uses a Helium plasma to generate ions which are then pulled by suction and electric fields into the mass spectrometer. The figure below shows a picture of the DART along with a schematic of our ion trap mass spectrometer from vendor literature (Velos, Thermo Corp., San Jose, CA). The low pressure cell (LPC) at the right is where the mass analysis occurs.

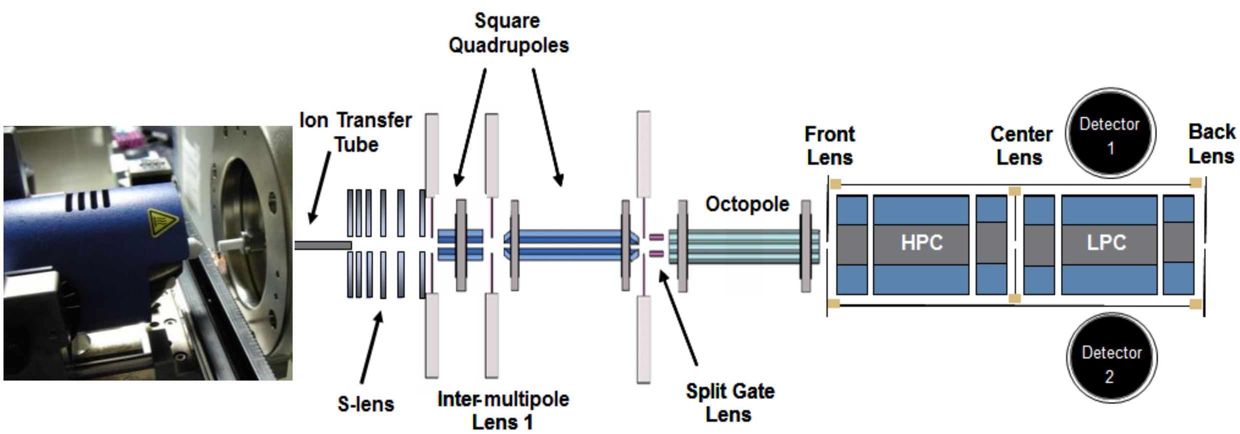


Figure 7: Photo of DART interface and schematic of mass spectrometer (HPC: high pressure cell, LPC: low pressure cell).

3.2 Door & Hallway Sign

The figure below was printed on a full page and was posted on exterior building doors and outside the laboratory. This sign was made using chemical structures of alar (a pesticide used for apples), sucrose, vitamin C, water, and clipart from Microsoft Powerpoint.

Figure 8: Door and hallway sign for "Food" activity.

3.3 Handout

The following pages contain images of the handout created for this activity. It was printed double sided and in color and with 0.5 inch top and bottom margins.

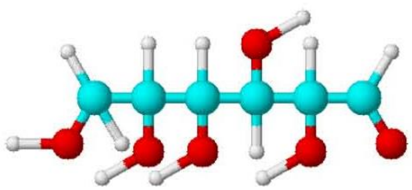
Food is Chemistry?

Welcome to Sandia's Materials Characterization and Performance Department. We test gases, liquids, and solids for Sandia, looking for chemical elements (atoms) and compounds (molecules). One tool we use is called a mass spectrometer. It "weighs" molecules and will help us solve the mystery, "Did you eat any molecules today?"

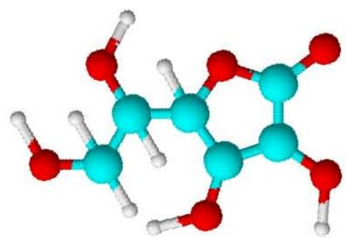
Atoms are the building blocks of everything around us – including us! Atoms in turn are the building blocks of molecules. You may have heard of some of them:

Deoxyribonucleic acid (DNA), keratin, water, salt, proteins, oxygen (O2).....

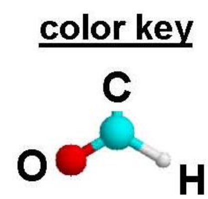
If we were to shrink ourselves down to the size of this dot . we would still be more than 3 *million* times bigger than a sugar molecule. From chemical measurements, we know how the atoms of many molecules are connected together. Here's how the atoms in molecules you might know are connected; each "ball" represents an atom of Carbon (C), Oxygen (O), or Hydrogen (H).



Glucose (sugar)



Ascorbic acid (Vitamin C)



Because we know how much each atom weighs, we can figure out the weight, or mass, of a molecule. For example, Vitamin C has 6 carbons, 8 oxygens, and 6 hydrogens which equals a mass of 176 atomic mass units (amu). Glucose weighs 180 amu.

You can find mass spectrometers in places like hospitals, factories, Army vehicles, and even on the Mars Rover Curiosity! They help us with important jobs like newborn health screening, Olympic athlete drug testing, food manufacturing, and space station air testing.

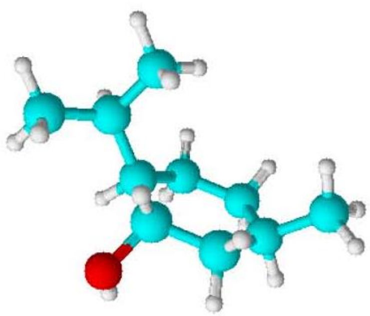
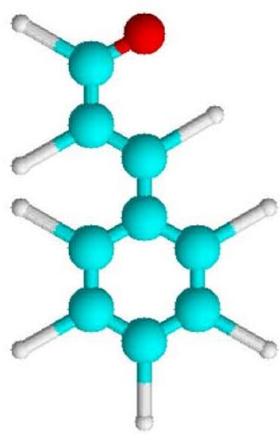
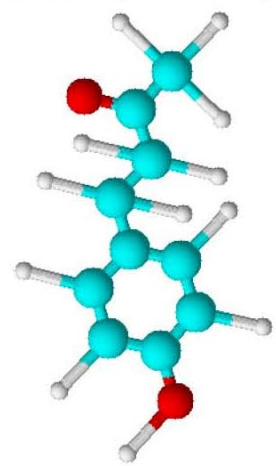
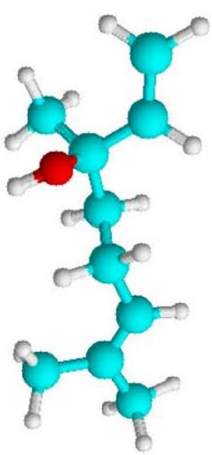
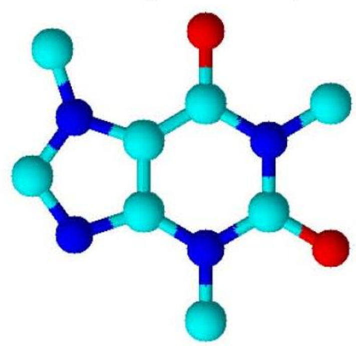
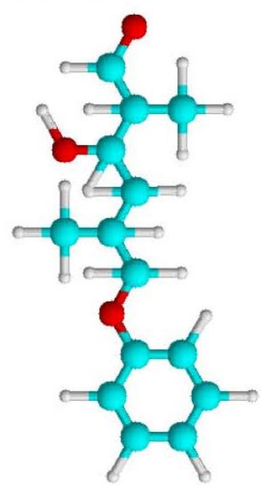
Let's solve the mystery, "Did you eat any molecules today?"

1) Choose a food item at our "Chemistry is Food?" table; chew or eat it.
2) What do your senses tell you about this food?
   a. How does it get its color?
   b. Does it have any smells?
   c. How many different molecules do you think are inside?
   d. Can your Sandia host detect which food you ate?
3) While you chew, think about whether you are eating any molecules.
4) Do you think that if you ate a molecule that you could breathe it out?
5) Come into the lab and maybe we can "see" your molecules.
6) P.S. – NO food in the lab!!

Inside our Laboratory

We have a mass spectrometer that weighs molecules. We're not allowed to put you inside or let you spit into it, but maybe we can check your breath….

Follow our scientist's instructions (BUT don't tell him which item you ate!). Our mass spectrometer is able to measure many different types of molecules. Here's some of the molecules you might have eaten.

Menthol $C_{10}H_{20}O$ (156.2 amu) 	Cinnamaldehyde C_9H_8O (132.1 amu) 	Raspberry ketone $C_{10}H_{12}O_2$ (164.2 amu) 
Linalool $C_{10}H_{18}O$ (154.2 amu) 	Caffeine $C_8H_{10}N_4O_2$ (194.1 amu) 	Bubble gum molecule (proposed) $C_{14}H_{20}O_3$ 

Did any molecules travel in your breath into the mass spectrometer?

3.4 Logistics and Procedure(s)

A table in a discussion area of the building hallway near the laboratory was set up with food items for consumption prior to the demonstration. The available items were based upon preliminary work performed to determine foods with distinctive spectra, that gave good signal, did not pose any allergy hazards, and were not too expensive. Several items that were tested but rejected included: “cutie” oranges, Capri Sun juice boxes (kiwi or fruit punch flavored), orange- and apple-flavored gum. Coffee was provided (for adults), although it seemed like those adults that participated consumed the other items.

Handouts were placed on the table along with the food items. A trash can was provided to discard (as needed) any food before entering the lab, however it was determined that better signal was obtained for items that were still in the mouth.

Using the spectral “key” for identification and watching the real-time data display, the operator would ask the participant to lean toward the DART and provide a long exhale. The operator would then attempt to determine what had been ingested.

3.5 Results

Both adults and children participated in this activity, and active response and later feedback was positive. Participant flow overall was rapid even though it was just one person at a time (as opposed to groups for the Curiosity demo). We provided less background narrative because we thought it would be difficult to succinctly explain the instrumentation. The short timeframe was a nice contrast to many of the TODSTWD demos that had long lines and long demonstrations. We estimate that we had about a 75% success rate in identifying what each person ate. Data was not saved or collected during the demo. Interestingly, a couple kids tried to “spoof” the analyst by eating two different items. The analyst (Adam) correctly detected each attempt!

During preliminary testing, several items were tested directly (i.e. not consumed and then breathed into the instrument). While the spectra were generally similar, a breath sample spectrum was sufficiently different and it is therefore recommended for future screening of new or additional food items to just perform the breath analysis. Examples are shown below of a direct analysis of an Altoids® mint and corresponding breath analysis.

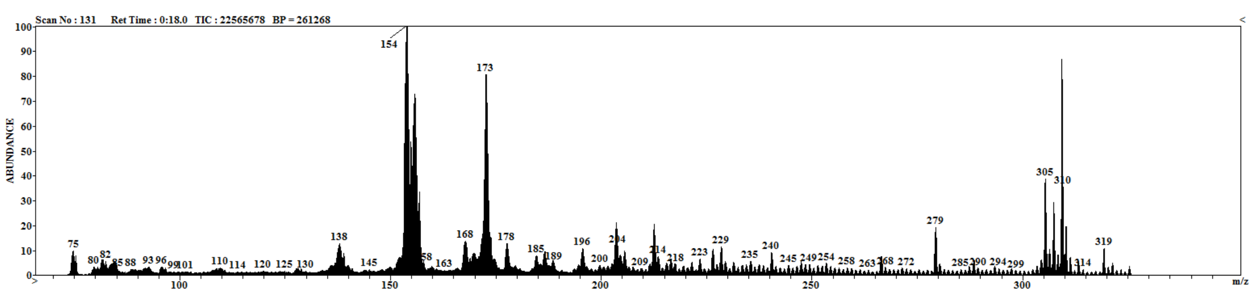


Figure 9: DART/MS spectrum of solid Altoids® sample.

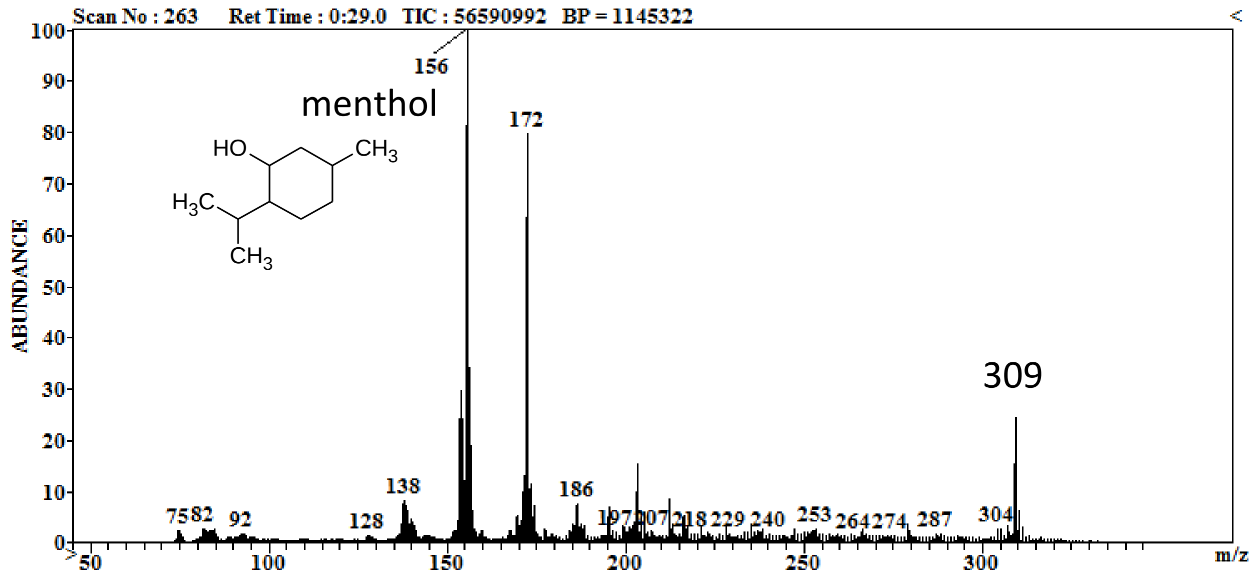


Figure 10: DART/MS spectrum of breath after Altoids® consumption.

Additional spectra are included for future reference in Appendix A. The following table is a listing of key ions that were observed for each type of food item along with additional ions observed. These were the "target" ions that were unique and allowed identification of the food item consumed by participants.

Table 1: key ions and molecules determined for test items.

x	Sample	Key ions (large to small)	Additional ions	key molecule
1	Altoids®	156, 154, 172	309	menthol
2	cherry cough drop	165, 138, 156	108, 228	raspberry ketone
3	Big Red® gum	134, 153, 138	265	cinnamaldehyde
4	bubble gum	236	138	unknown
5	coffee	195		caffeine
6	cutie orange	138, 170, 235	154	linalool

The following spectra were also collected prior to the demo and this data was utilized in an effort to identify the "bubble bum molecule" that was a strong and unique signal of bubble gum in breath. The first spectrum is an isolation of m/z 236 followed by a fragmentation sequence to determine how m/z 236 would break apart. This is a typical procedure for determining the structure and identity of an unknown molecule.

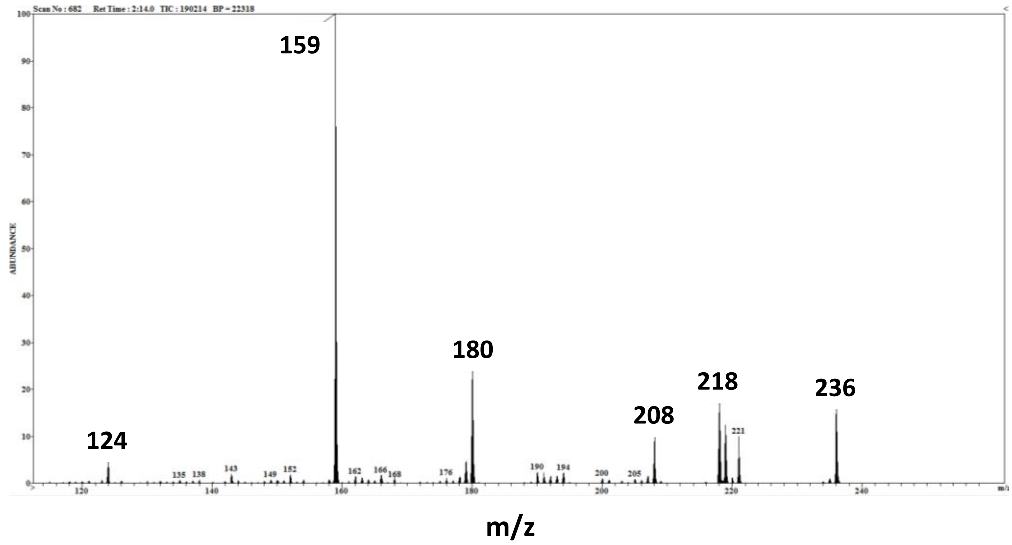


Figure 11: DART/MS^2 of "bubble gum molecule", isolate and fragment m/z 236, scan for products.

In an additional experiment, m/z 236 was isolated and fragmented. The fragment at m/z 159 was then itself isolated and fragmented. Because of the multiple stages of isolation, fragmentation, and analysis, this process is designated as MS^3. The spectrum below is a result of MS^3 analysis of the "bubble gum molecule". This data allowed us to estimate the structure shown in the handout. We have been unable to find this molecule or any similar flavor or fragrance molecule in our literature and World Wide Web searches. Since this is a unique molecule for bubble gum in our tests and thus would be an excellent candidate for future demos, we will continue to investigate the identity of this species.

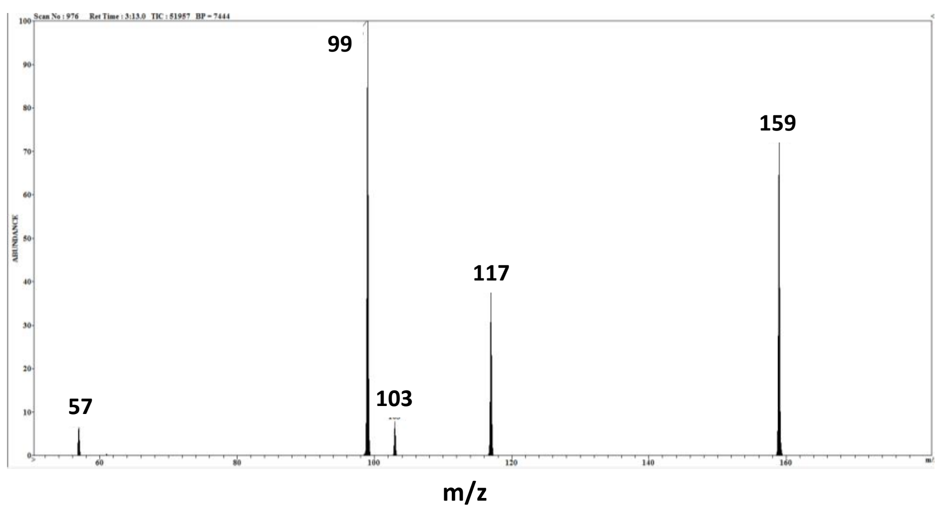


Figure 12: DART/MS^3 of "bubble gum molecule", isolate and fragment m/z 236 followed by isolate and fragment m/z 159, scan for products.

3.6 Lessons Learned

People were lining up without seeing the handout, and/or the food table – and were thus unaware that they should choose and partake of a food item before coming in the lab. Additional personnel were thus needed for "traffic control" and for discussing the process with those in line. There were several children and parents that attempted the demo without eating. This used up time and reduced throughput, as they had to be sent out to the table for a food item.

A stronger tie-in with current events or some other use of a mass spectrometer would be useful. Schematics or a video in the hallway might provide additional information for people to view while waiting, or if they become more interested, would serve as background information following the demo.

4 CONCLUSIONS

We created interactive demonstration activities for Take Our Daughters & Sons to Work Day (TODSTWD) 2013 in order to promote general interest in chemistry and also generate awareness of the type of work our laboratories can perform. A total of over 130 children participated in these activities over a 3 hour block, and feedback was positive. We generated handouts and experimental procedures to facilitate the demonstrations and provide something for children to take home. We've reported here several lessons learned so that future demonstrations can benefit from the baseline work performed. Children and adults alike seemed to enjoy these demonstration activities, and we believe that we achieved our goals of promoting chemistry and our laboratory capabilities in an interesting and fun way. Breathing into an instrument and having something in your breath identified immediately, and seeing the spark as your rock is zapped by a laser, were activities enjoyed by all who attended.

5 REFERENCES

1. Knight, A. K.; Scherbarth, N. L.; Cremers, D. A.; Ferris, M. J., Characterization of laser-induced breakdown spectroscopy (LIBS) for application to space exploration. Appl. Spectrosc. 2000, 54, 331-340.

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3. PIA16089: Coronation's Chemicals. http://photojournal.jpl.nasa.gov/catalog/PIA16089 (accessed 2013).

4. PIA16615: Calcium-Rich Veins in Martian Rocks. http://photojournal.jpl.nasa.gov/catalog/PIA16615 (accessed 2013).

APPENDIX A: REFERENCE SPECTRA FOR DART/MS DEMONSTRATION

These mass spectra were collected to generate the reference table that enabled the operator to identify which food item had been consumed.

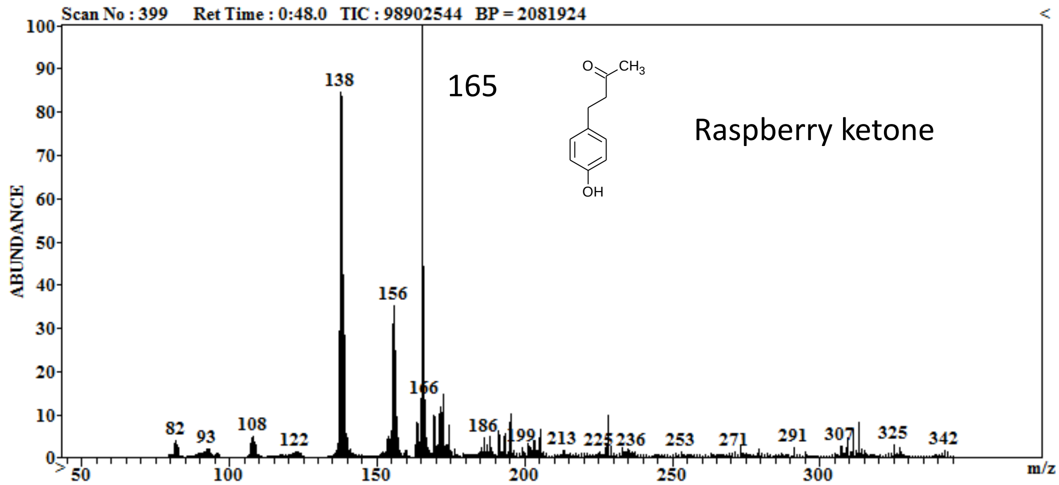

Figure 13: DART/MS spectrum of breath after cherry cough drop.

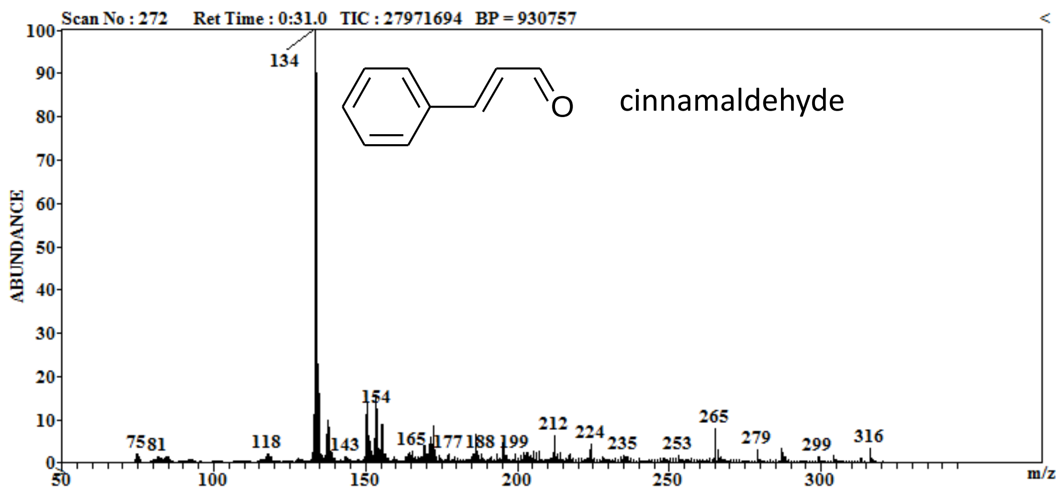

Figure 14: DART/MS spectrum of breath after Big Red® gum

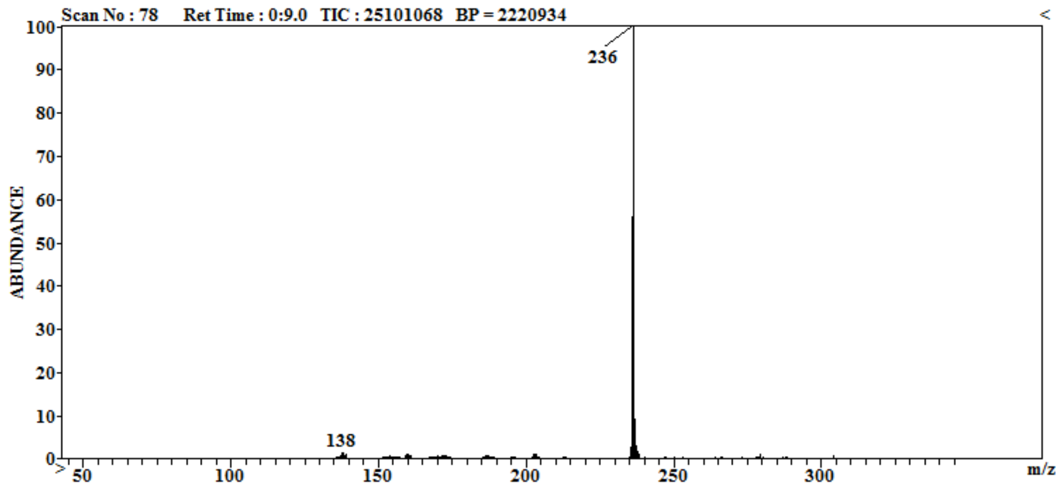

Figure 15: DART/MS spectrum of breath after bubble gum

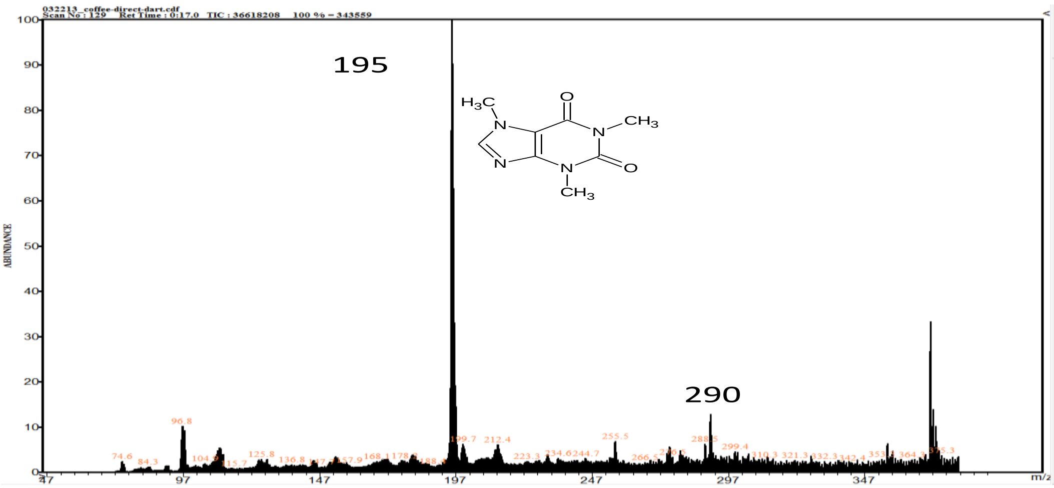

Figure 16: DART/MS spectrum of breath after coffee.

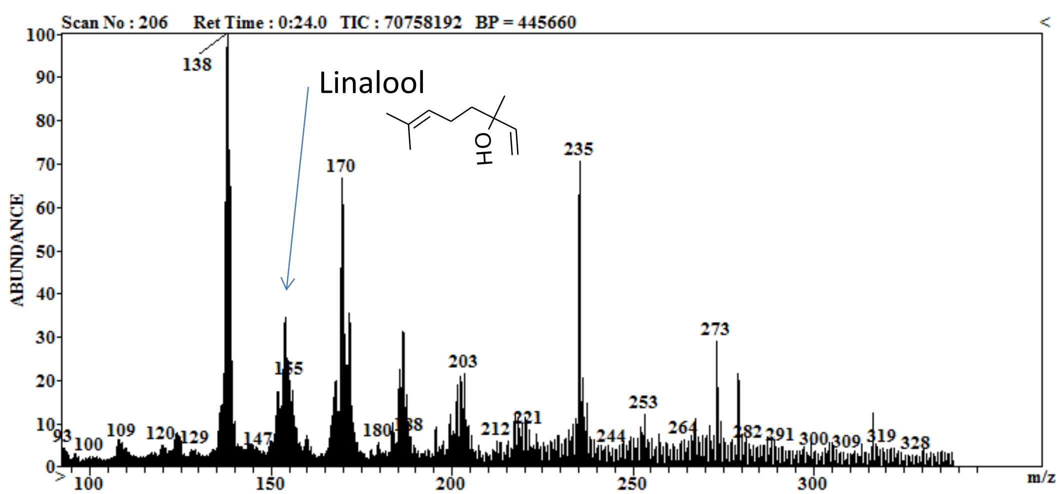

Figure 17: DART/MS spectrum of breath after "cutie" orange.

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