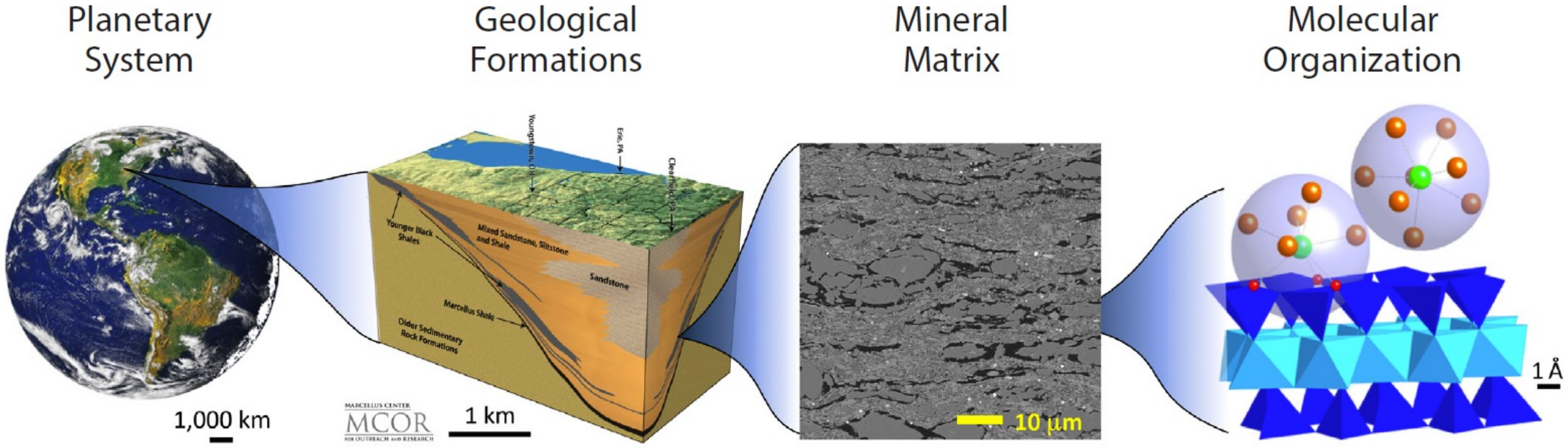
XPLAin: Compact, rapid elemental, mineralogical, and textural analysis of unprepared samples



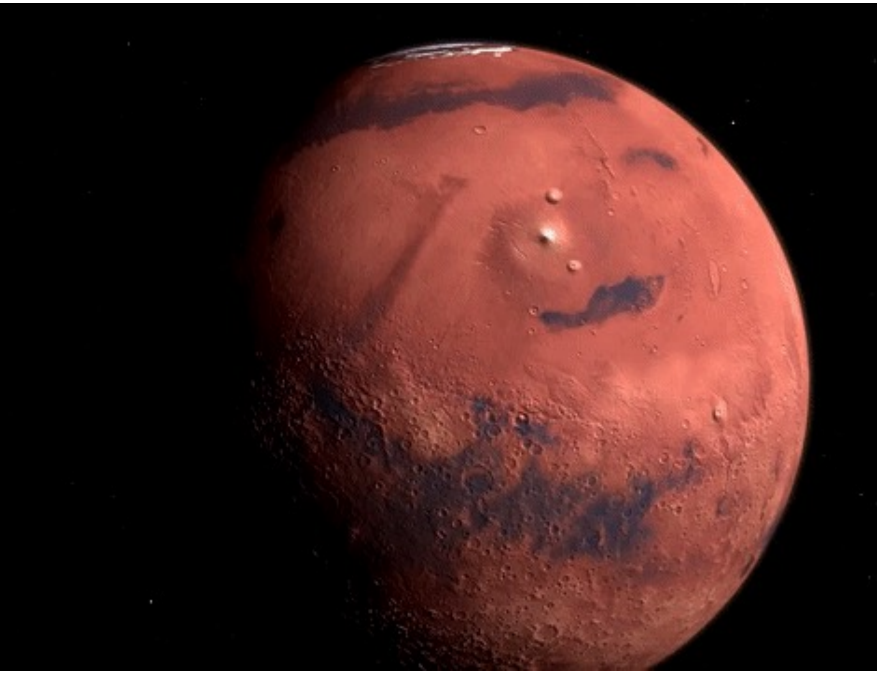
Interplanetary Small Satellite Conference
Pamela Clark, Przemyslaw Dera, Daniel Scheld and Christopher Dreyer
May 3, 2022

Earth is a complex and dynamic system.

Major geologic events have significant effect on the human civilization.

Human activity can significantly affect the stability of the Earth system in the long run and require geological resources, which form in the interior.

Understating of the structure, composition and properties of the Earth interior offers us a better chance to live in harmony with our planet (e.g. geo-hazards, mineral resources, etc.).



Motivation for understanding planetary mineralogy

- Geologic history
- Colonization resources
- Natural hazards

The Apollo Lunar Surface Experiment Packages (ALSEPs) were a unique series of in-situ geophysical experiments, which included seismic experiments. No seismic observations have been performed on the Moon since Apollo. The experiments included the Passive Seismic Experiment (PSE), the Active Seismic Experiment (ASE), and the Lunar Surface Profiling Experiment (LSPE). For decades, these data have been used to investigate the internal structure of the Moon

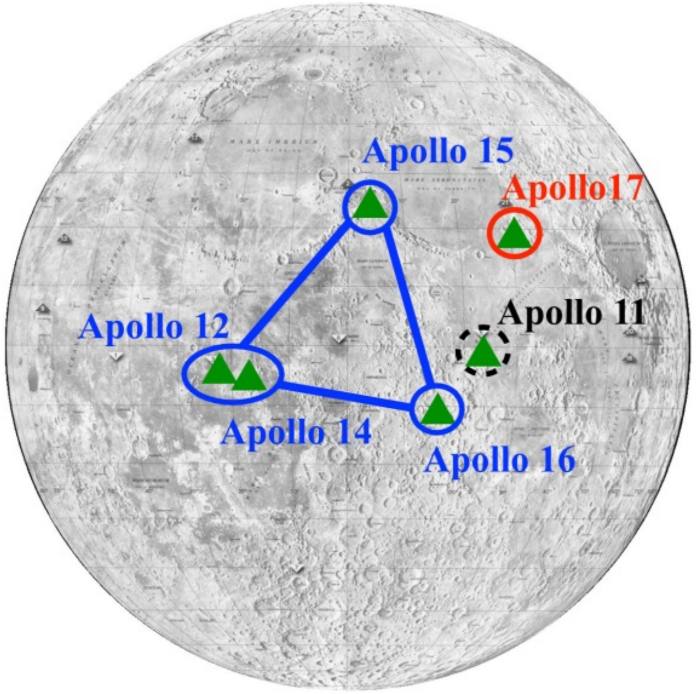
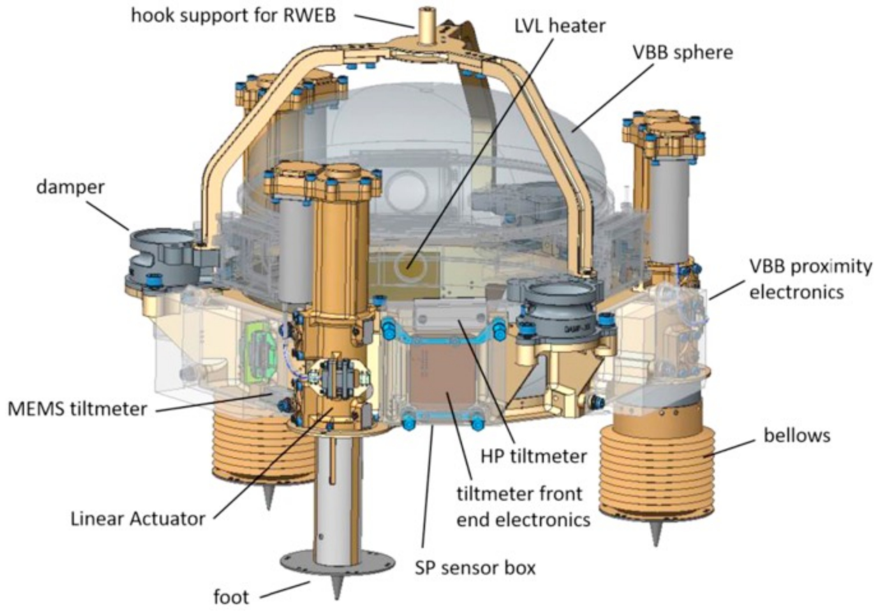


Fig. 1 Locations of the Apollo stations on the Moon. Passive Seismic Experiments (PSE) were based at Apollo 11, 12, 14, 15 and 16 (station 11 was only operational for one lunation). Active Seismic Experiments (ASE) were based at Stations 14 and 16. A second active experiment, known as the Lunar Seismic Profiling Experiment (LSPE) was based at station 17. Station 17 also included the Lunar Surface Gravimeter (LSG), which is a source of additional passive seismic information



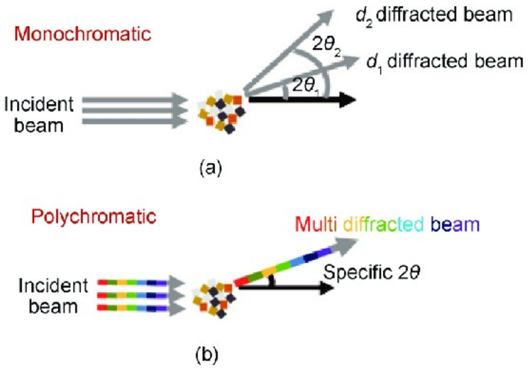
NASA's InSight spacecraft **touched down Nov. 26, 2018, on Mars to study the planet's deep interior**. A little more than one Martian year later, the stationary lander has detected more than 480 quakes and collected the most comprehensive weather data of any surface mission sent to Mars.

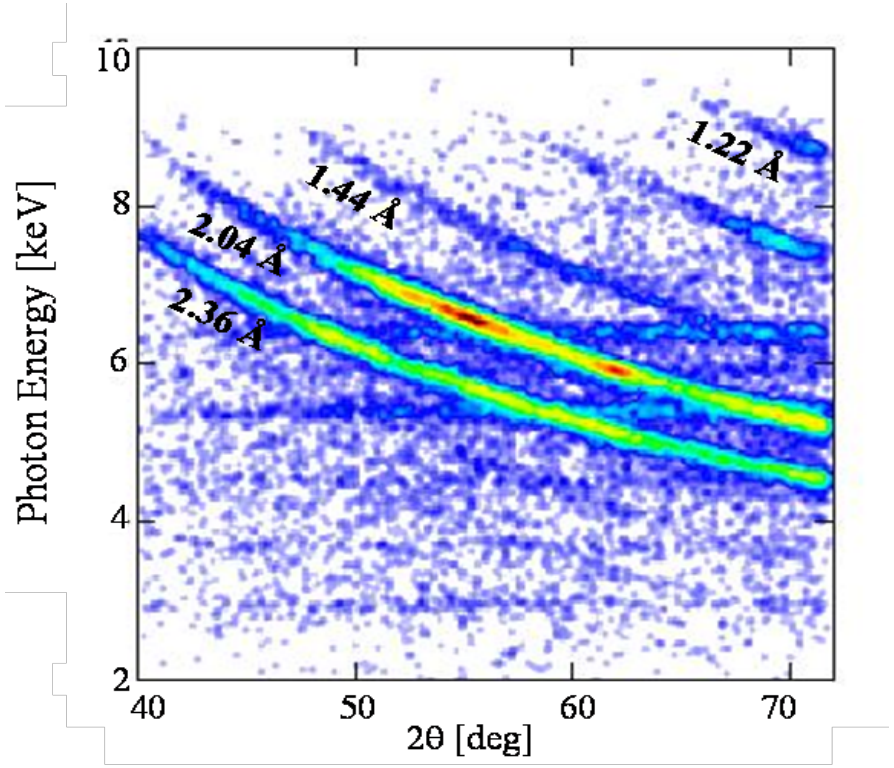
XRD

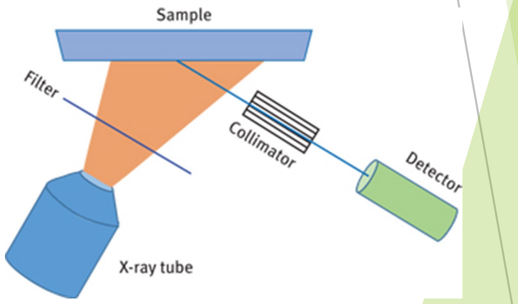
- Each mineral phase contributes a set of diffraction peaks
- Bulk phase composition can be refined by refining phase fraction
- Signal has a geometric distribution (directional peaks or cones of radiation)
- Peak intensities are sensitive to elemental composition

XRF

- Each element contributes a set of peaks
- Signal is isotropic
- There is no information about crystal structure
- Emission lines of light elements (below Al) are very difficult to detect







CheMin

A Historical Perspective of the Development of the CheMin Mineralogical Instrument for the Mars Science Laboratory Mission

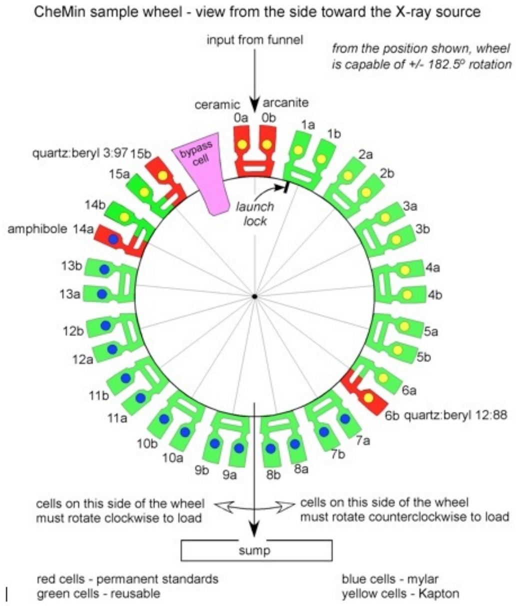
by David Blake, NASA Ames Research Center

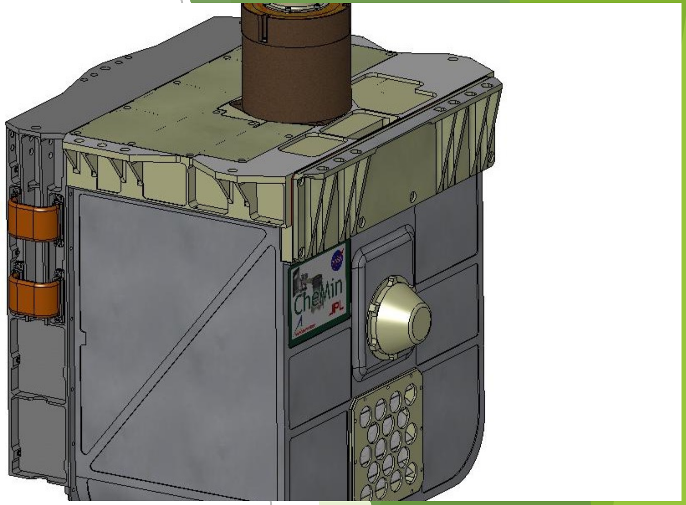
> ***Introduction by Paul Mahaffy (NASA Goddard Space Flight Center)***
> *Volumes of multispectral infrared imaging data presently flowing in from Mars orbiting spacecraft are giving us a new view of the planet (Ehlman, Geochemical News 142) and pointing toward candidate landing sites for surface rovers. Highly ruggedized and miniaturized instruments on future rovers will carry out an even more detailed exploration of the chemistry and mineralogy at the most interesting sites to elucidate geological and geochemical processes that may point toward habitable environments for past or present life. One such instrument planned for use on the Curiosity rover that is planned to land on Mars in 2012 is the x-ray fluorescence/x-ray diffraction instrument CheMin described in this contribution from the Principle Investigator for this investigation, David Blake. Some of the robust field-testing of this instrument on remote Mars analog sites in the Arctic Svalbard archipelago is also described.*

David Blake

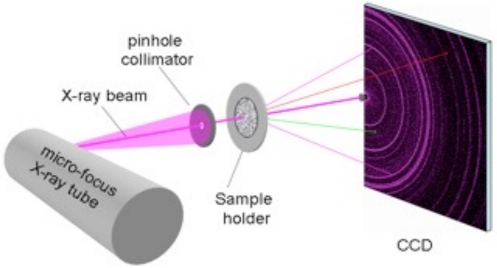
About the Author

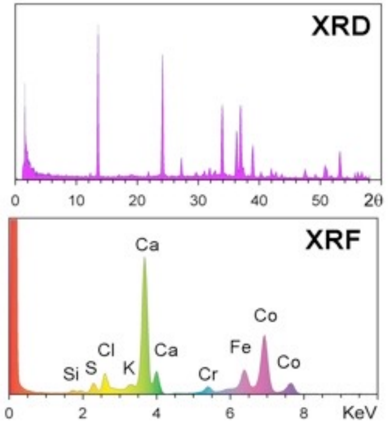
David F. Blake is co-investigator of Photosynthetic and Chemosynthetic Ecosystems for NASA's Space Science and Astrobiology at Ames.

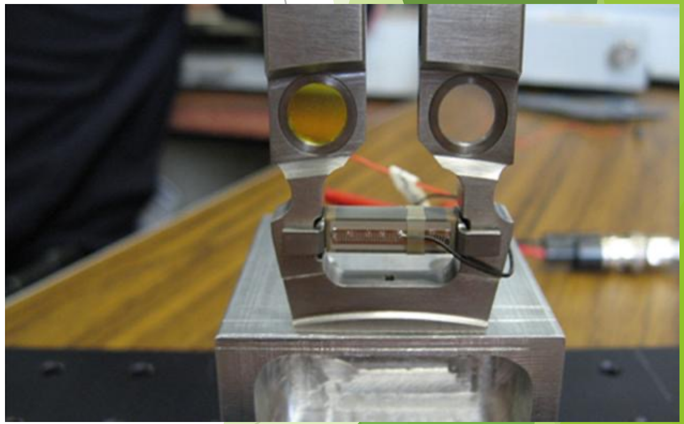

CheMin sample wheel - view from the side toward the X-ray source



The first sample of powdered rock extracted by the Curiosity rover's drill. The image was obtained by Curiosity's Mast Camera on Feb. 20, or Sol 193, Curiosity's 193rd Martian day of operations. (Image credit: NASA/JPL-Caltech/MSSS)
ttps://www.space.com/20182-ancient-mars-microbes-curiosity-rover.html

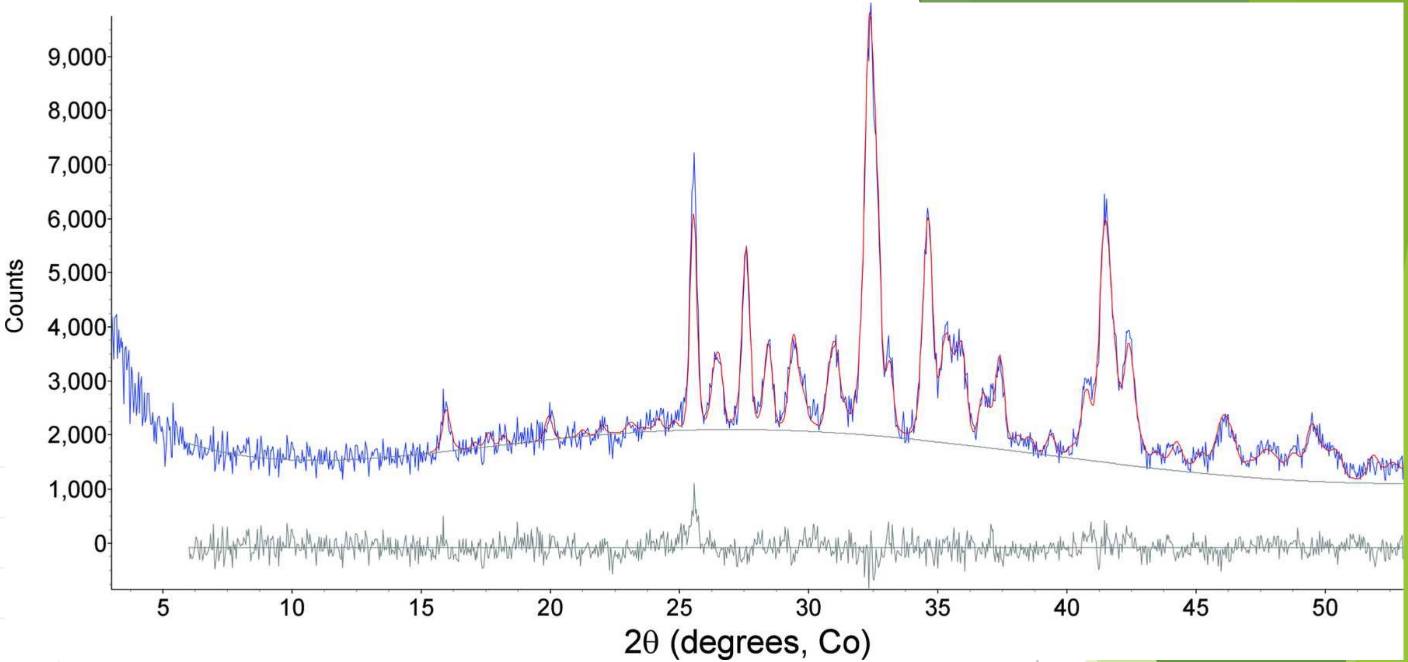






https://mars.nasa.gov/msl/spacecraft/instruments/chemin/for-scientists/

CheMin data

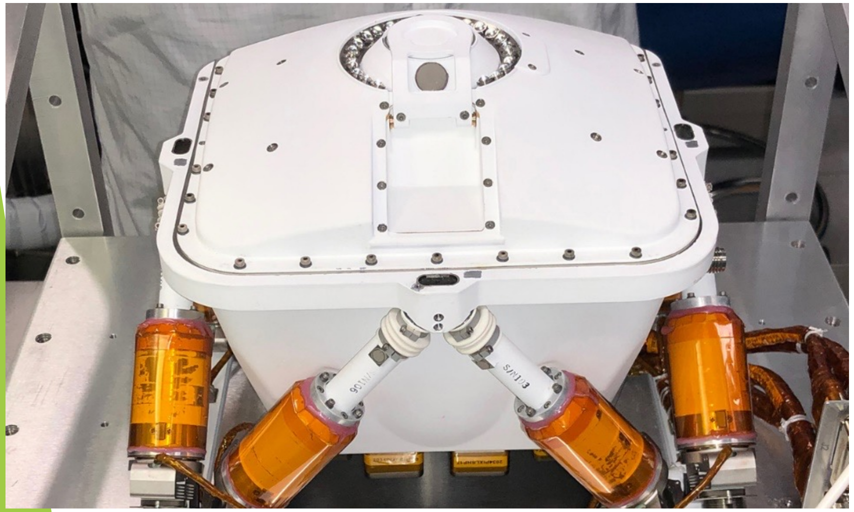


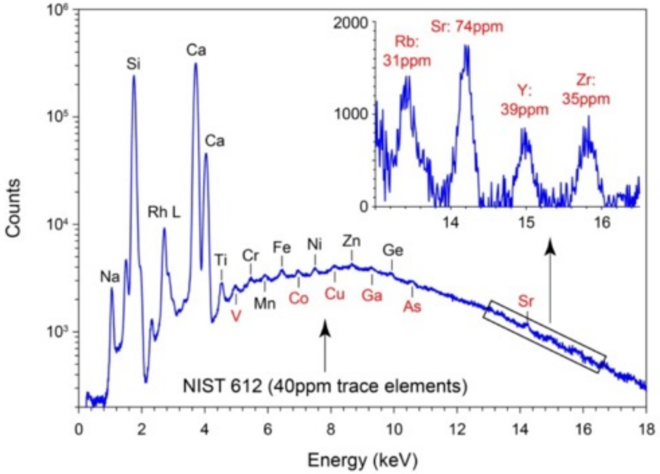
Bish et al. (2014) IUCrJ 1, 514-522

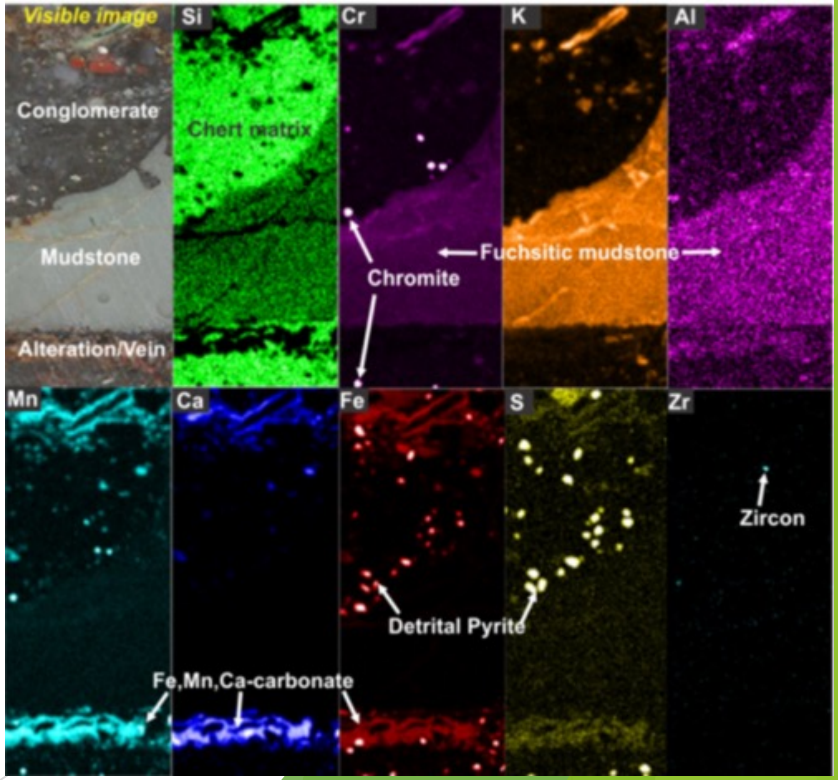
Plagioclase	22	22
Fe-forsterite	3	1
Augite	4	4
Pigeonite	6	8
Orthopyroxene	3	4
Magnetite	4	4
Anhydrite	3	1
Bassanite	1	1
Sanidine	1	2
Quartz	0.4†	0.1†
Hematite	0.6†	1
Ilmenite		0.5†
Akaganeite	1	2
Pyrite	0.3†	
Pyrrhotite	1	1
Phyllosilicate	22	18
Amorphous	28	31

NASA PIXL:(Planetary Instrument for X-ray Lithochemistry)

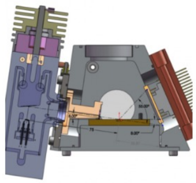
https://mars.nasa.gov/mars2020/spacecraft/instruments/pixl/for-scientists/

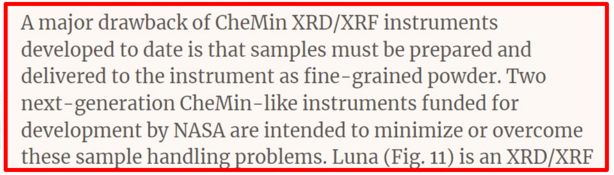






Future Versions of CheMin for Mars and Other Solar System Destinations





A major drawback of CheMin XRD/XRF instruments developed to date is that samples must be prepared and delivered to the instrument as fine-grained powder. Two next-generation CheMin-like instruments funded for development by NASA are intended to minimize or overcome these sample handling problems. Luna (Fig. 11) is an XRD/XRF

Figure 11a

Figure 11b

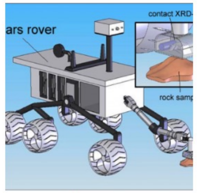


Figure 12a

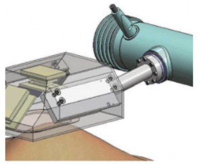


Figure 12b

CheMin -type instrument challenges

- Sample processing-induced alterations
  - Different phases grind with different ease
  - Mesh filtration can significant alter phase fractions (different phases have different grain sizes)
  - Mechanochemistry induced by sample grinding can decompose some minerals or cause other chemical reactions
  - Information about granularity and texture of rocks is valuable from petrological perspective
- Sample chamber contamination
- Cloggage of transport lines
- Signal absorption by heavy mineral phases
- Mineral phase-specific chemical information
- Quantitative information on amorphous phases

2008 NASA ASTID: Hybrid powder / single-crystal X-ray diffraction instrument for planetary mineralogical analysis of unprepared samples. PI S. Sarrazin
2014 NASA PICASSO: Miniature Guinier X-ray Diffraction Instrument for Planetary Exploration. PI S. Sarrazin
2018 NASA DALI: XTRA: An eXTraterrestrial Regolith Analyzer for Lunar Soil. PI D. Blake

Why mineralogy without chemistry is not enough

- Chemistry of amorphous component cannot be deduced by Rietveld Refinement.
  - Martial soil contains unusually high fraction of amorphous material
  - So far with CHEMIN data we assumed that there is single amorphous phase with chemical composition close to the average of
  - Amorphous phases can be important clues about mineral alteration processes

Bulk mineralogy vs. microanalysis/petrography

https://simulantdb.com/simulants/bp1.php

http://microckscopica.altervista.org/en/

- Only major and minor mineral fractions are determined.
- No information about grain orientations, sizes and contact is preserved.

- The mineral content and the textural relationships within the rock are described in detail.
- The classification of rocks is based on the information acquired during the petrographic analysis.
- Micro-texture and structure are critical to understanding the origin of the rock.

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ADVANCES IN
SPACE
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(a COSPAR publication)

www.elsevier.com/locate/asr

Development of a thin section device for space exploration: Overview and system performance estimates

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Received 22 May 2012; received in revised form 13 December 2012; accepted 17 December 2012
Available online 23 December 2012

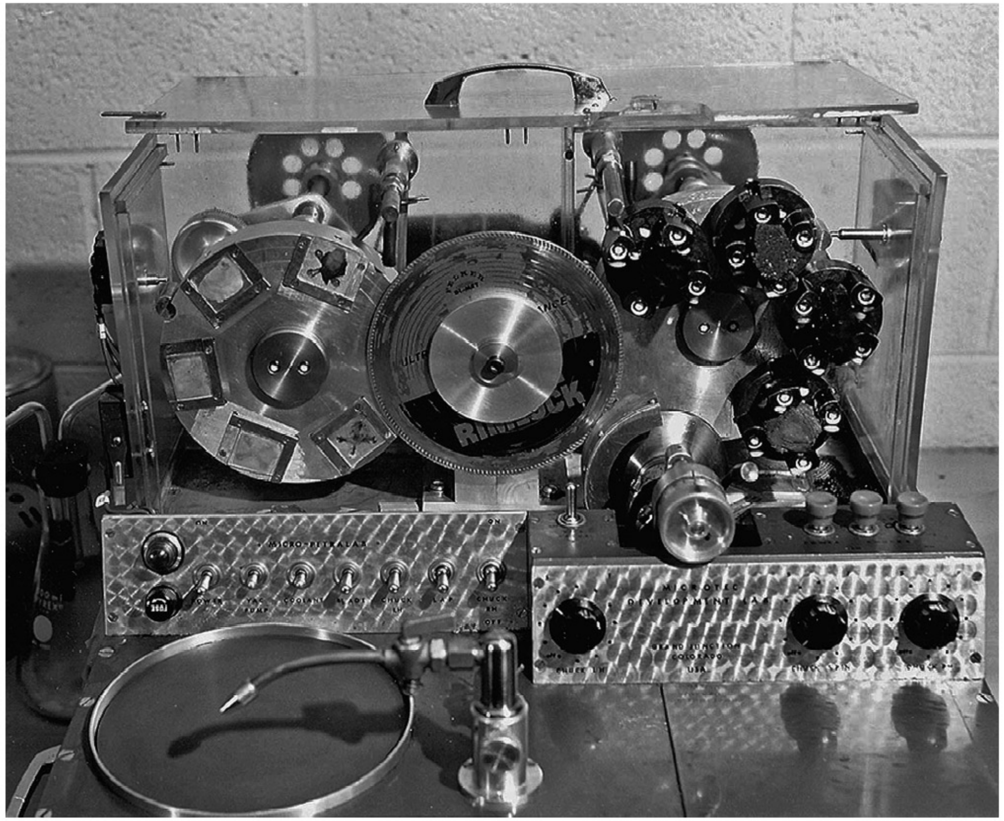
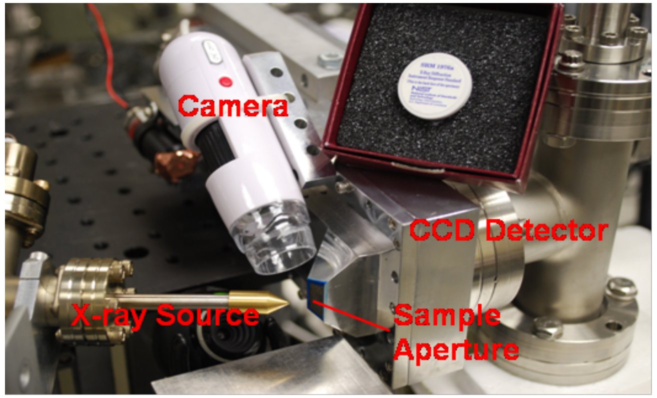


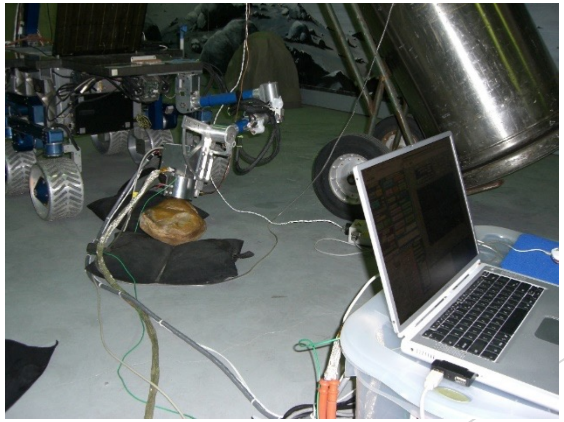
Figure 34- Prototype, semi-automatic rock cutting and thin-sectioning machine that was designed and built in 1965 for the Apollo Applications Program (AAP) and Advanced Lunar Programs Groups at the USGS in Flagstaff in 1965 by Paul Cary in Grand Junction, Colorado. Branch of Astrogeology personnel arranged for NASA to provide Paul with a $6,000 grant to hand-build the prototype machine for used in geologic laboratory and field training of the astronauts. Paul later formed "Petrolab", a very successful company that produced automatic, rock thin-section machines; USGS photo F12653.

XPlAin design principles

- Design principles
  - Reflection-geometry “camera” that can be deployed on a rover arm
  - No sample processing required
  - Functional complementarity & redundancy (multiple detectors, multiple sources)
  - Micro analysis on planetary surface (mineralogy and chemistry from a single grain)
  - Quantitative characterization of amorphous material (chemistry and pdf)
  - Texture analysis

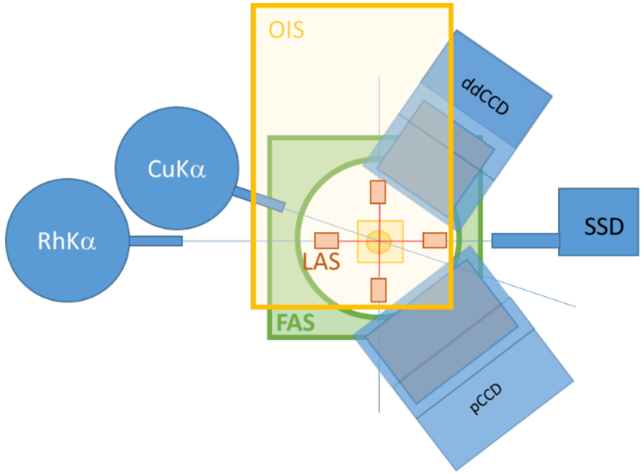


CMIST (PI K. Gendreau) 2012
Chromatic Mineral Identification and Surface Texture

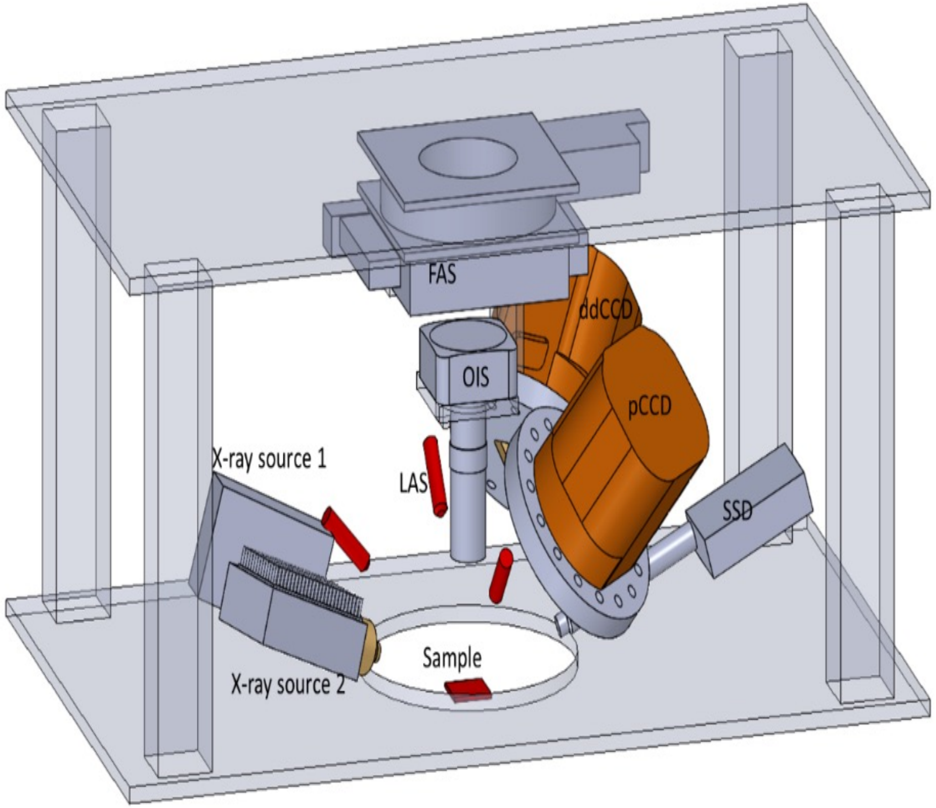


MICA (PI J. Marshall) 2005
Mineral Identification and Composition Analyzer

XPlaIn design concept

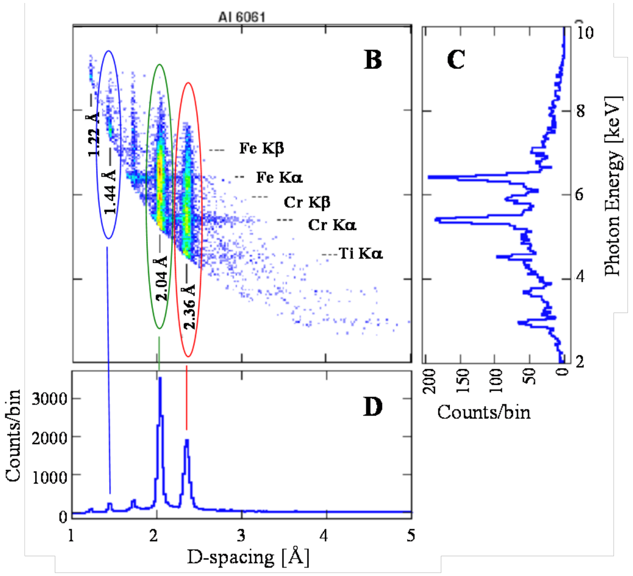


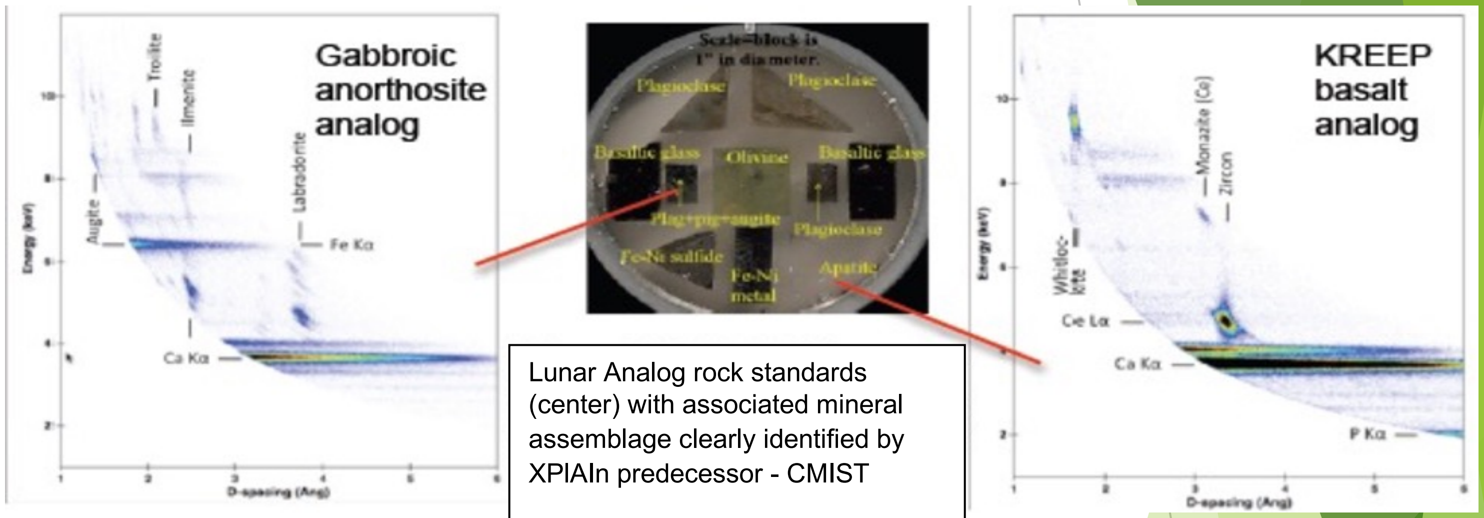
Notional diagram of XPlAIn components viewed from the top of the instrument.



Notional diagram of XPLAIn components. FAS: Fine Actuation System, OIS: Optical Imaging System, LAS: Laser Alignment System, pCCD: phosphor CCD, ddCC: direct detection CCD, SSD: solid-state detector.

Preliminary data from previous NASA projects





Lunar Analog rock standards (center) with associated mineral assemblage clearly identified by XPIAIn predecessor - CMIST

Preliminary data from previous NASA projects

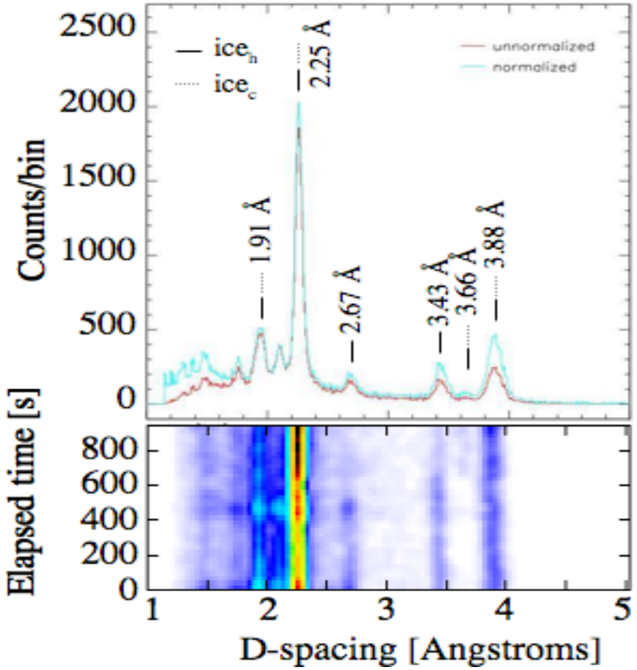


Figure 6. *CMIST* Time-dependent XRD revealing changes in frost consistent with a crystal phase transition

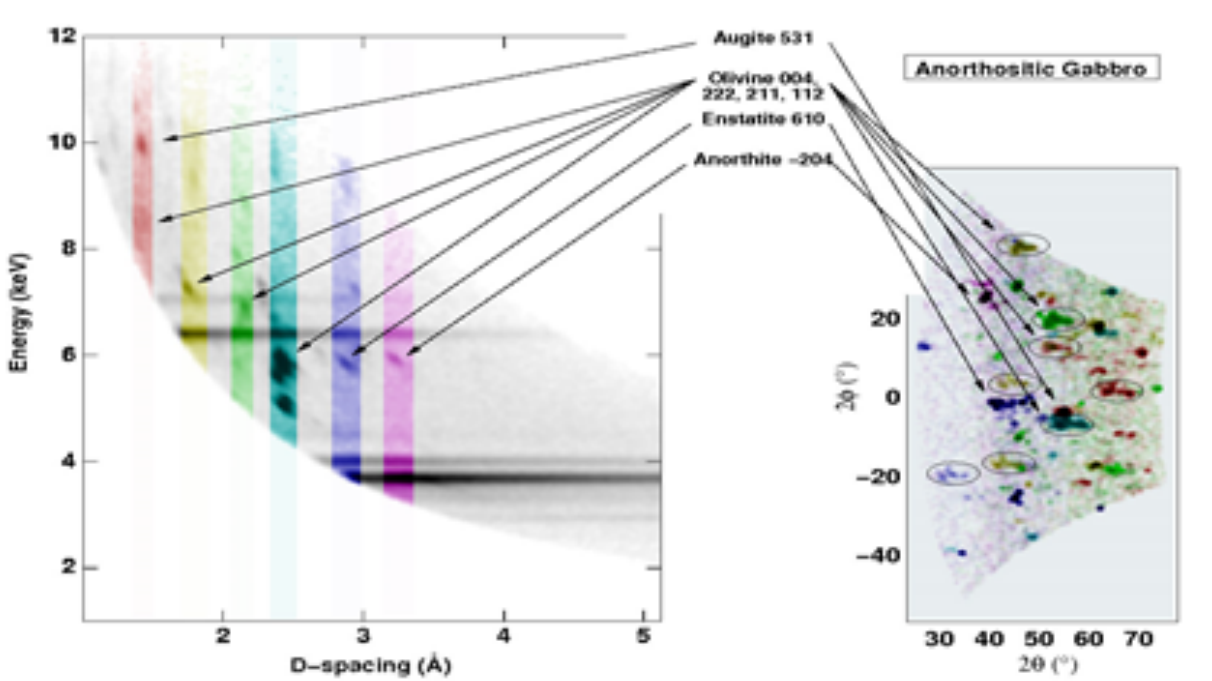
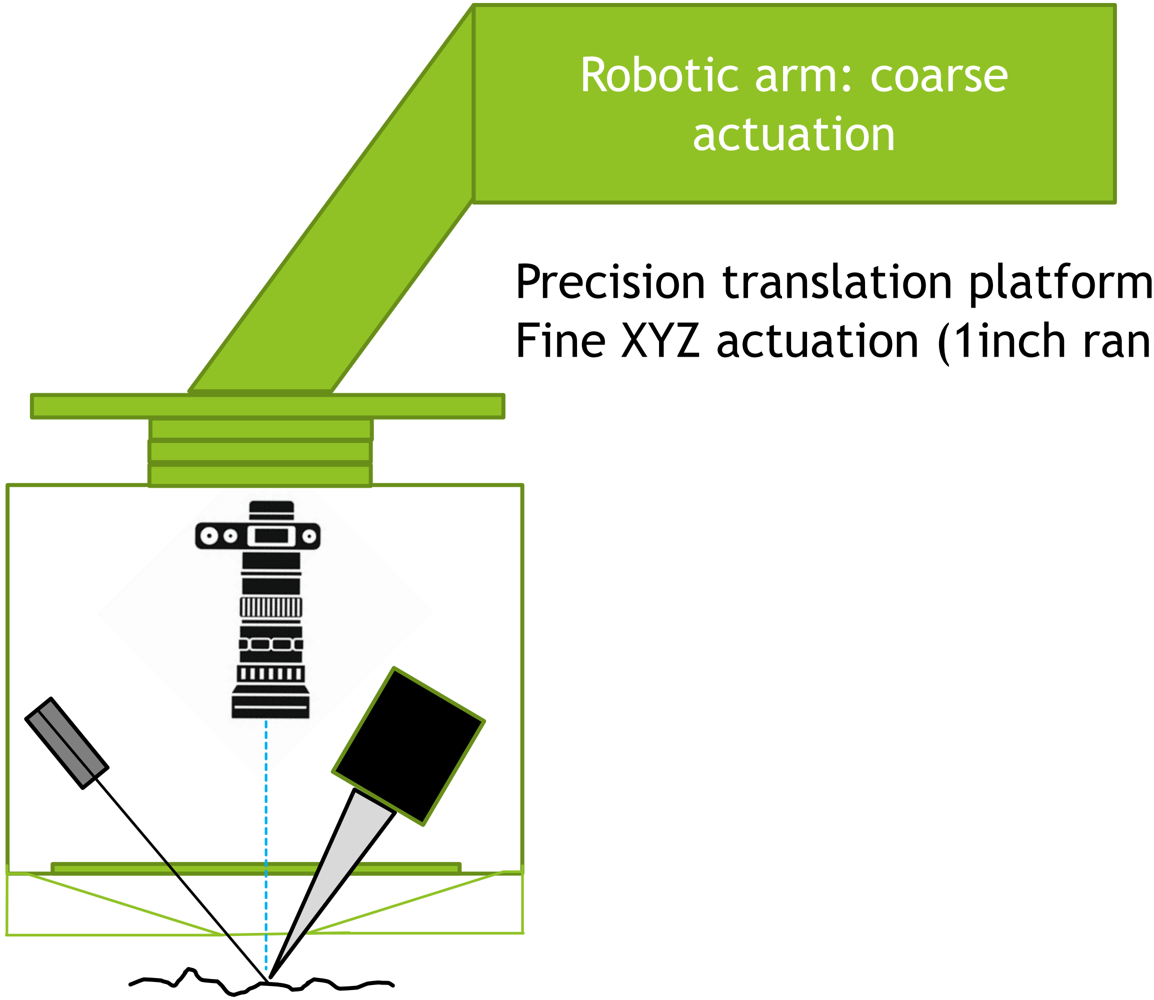


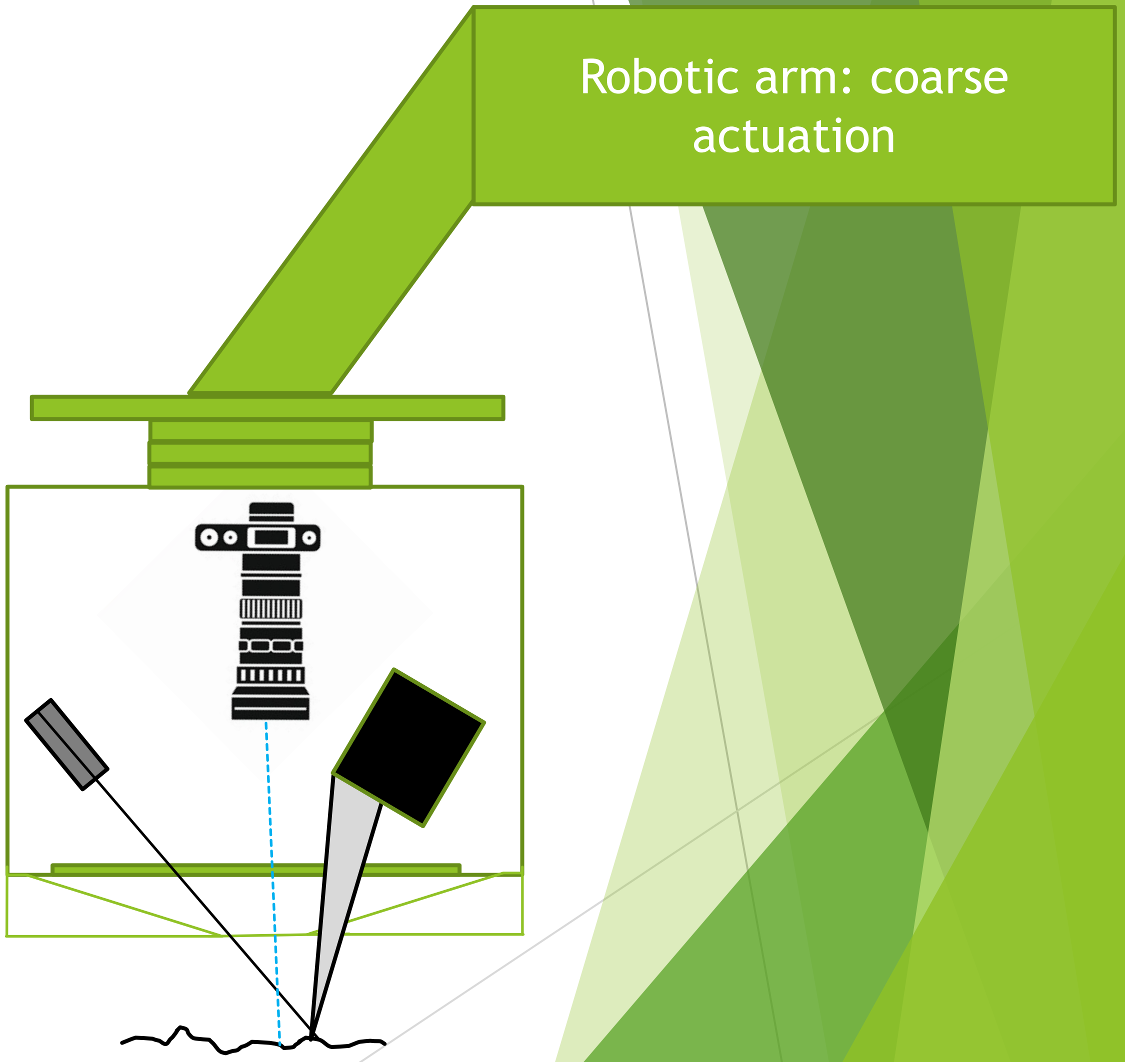
Figure 7. Data from an unprepared sample of lunar-analogue anorthositic gabbro. Selected d-spacings are color-coded (*left*) to highlight major minerals by Miller index. A map of the crystallite orientations (*right*) shows repeating Laue spots of the mineral grains, revealing the morphology of an olivine crystal (*circled*)

Accurate positioning challenge



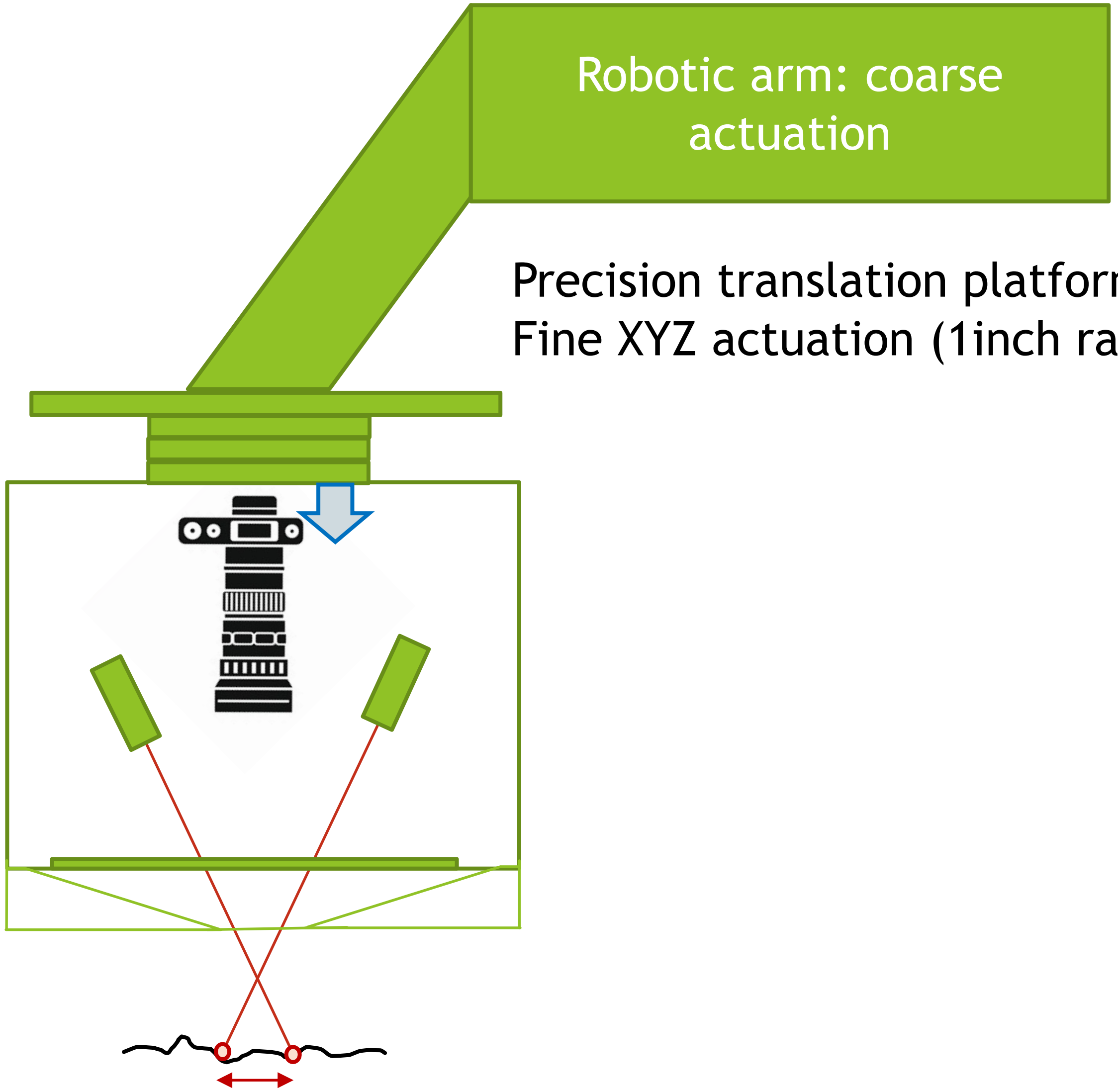
Robotic arm: coarse actuation

Precision translation platform
Fine XYZ actuation (1inch range)

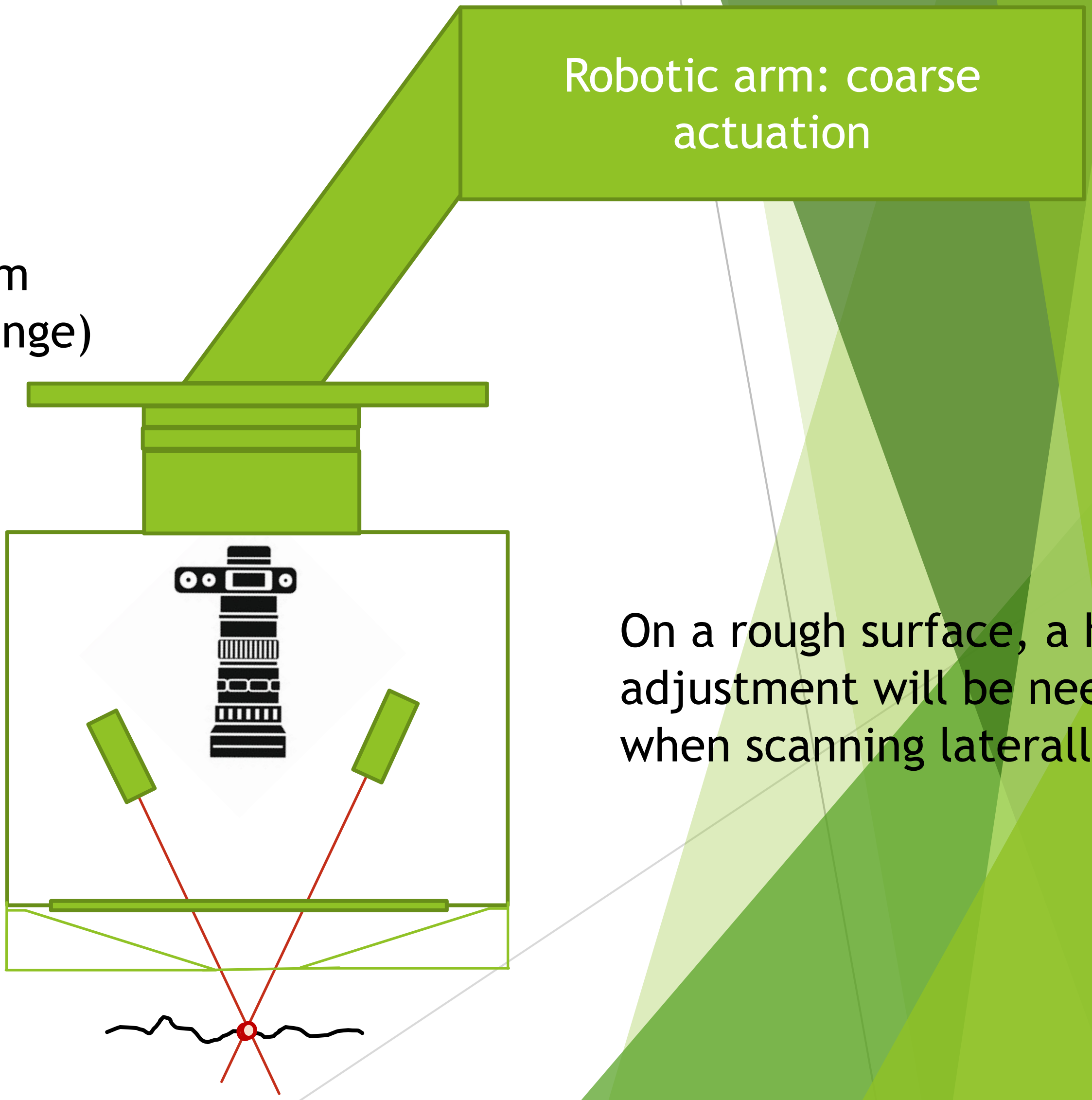


Robotic arm: coarse actuation

Accurate positioning challenge



Precision translation platform
Fine XYZ actuation (1inch range)



On a rough surface, a hight adjustment will be needed when scanning laterally.

XPLAIN proposal team

Member	**Org.**	**Role/responsibilities**	**Relevant experience**
PI P. Dera	*UH*	Project Manager Software development Lead scXRD development lead XRD SME	Crystallographer and mineral physicist Synchrotron XRD program manager Co-I on multiple NASA instrument projects MSL CheMin team software developer
Co-I W. Farrand	*SSI*	OIS developmen lead Planetary geology SME	Planetary geoscientist, MER Science Team, PI, Mars fundamental research and data analysis program PI
Co-I C. Shearer	*UNM*	Standards, calibration, and metrology lead Planetary petrology and mineralogy SME	Geochemical lab, analysis and sample handling specialist, Manager Institute of Meteoritics, planetary geochemistry, data analysis, and instrument developer PI
Co-I C. Dreyer	*CSM*	Instrument Manager Intrument design lead System testing lead Engineering SME	Engineer, instrument and tool developer, NASA instrument development programs PI
Co-I P.E. Clark	*JPL*	Lab Methodology development lead Sample Interface Methodology Lead Systems engineering support lead Lunar Science, Instrumentation, and Environment SME	Planetary geoscience, compact instrument systems, and formulation for robotic and human surface exploration, NASA mission Science PI
Co-I L. Ehm	*SBU*	Glass analysis lead Total scattering SME	Crystallographer and mineral physicist Synchrotron XRD program manager PI on multiple funded NASA projects
Co-I D. Sheld	*N-Science Inc.*	Instrument design lead Instrument manufacturing lead Engineering SME	Engineer, instrument and tool developer, NASA instrument development programs PI
Collaborator Col.: S. Speakman	*Panalytical Inc.*	Advisor on analysis algorithm and software development XRD SME XRF SME	XRD and XRF analytical software and lab technique developer, former head MIT PRISM lab.

Conclusions

- XPLAin will enhance analytical capabilities for mineralogical and petrological analysis of rocks and soil on planetary surfaces enabling single gain characterization.
- Transmission geometry design will remove the necessity to process the sample material.
- Functional redundancy and complementarity of the different detectors and X-ray sources will make the instrument more fail-safe and accurate.
- XPLAin will enable quantitative analysis of amorphous materials which might be critical for understanding of the nature of mineral alteration processes.
- Textural information for rock sample will provide valuable petrological insights.