

Starting a European Space Agency Sample Analogue Collection (ESA²C) and Curation Facility for Exploration Missions: CHEMICAL CHARACTERISATION

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Introduction

The Natural History Museum (NHM) and ESA have initiated a new sample analogue collection (ESA²C) to support current and future technology development activities that are required for human and robotic exploration missions. The goals for this project are described in abstract/poster 1218 [1] and includes an overview of the curatorial and chemical and physical characterisation procedures that will underpin the collection.

In 2016, 5 Basalt (aggregate and rock) and 6 Clay powder/granular sample analogues from the ESA²C were characterised in a round of chemical testing using the following techniques: XRD, SEM, EPMA, ICP-AES and ICP-MS

The analytical techniques and results are presented with an overview of the samples' chemical suitability as analogues of Mars, Phobos, Deimos, C-Type Asteroids and the Moon for ESA Exploration Missions



Analytical Techniques and Conditions - NHM Imaging and Analysis Centre

X-Ray Diffraction (XRD) - Whole Rock Mineralogy



- PANalytical X'Pert Pro Diffractometer - 45 kV and 40 mA
- Basalt and clay samples crushed and milled to fine powders
 - Oriented and Random Mounts (air-dried and EG Solvated)
 - Quantification of mineral phases
 - Rietveld Method for Basalts
 - Pattern Stripping for non-clay phases in clay-rich samples

ICP-AES/MS - Major, Trace and Rare Earth Elements



- Thermo scientific iCAP 6500 duo ICP-AES
- Major elements analysis: Al Ca Fe K Mg Mn Na P Si Ti Zr C H S
 - Fine aliquot powders fused with LiBO₂ flux
- Agilent 7700x ICP-MS
- Analysis of 37 Trace and REE including: Sc V Cr Co Ni Cu Zn Sr Zr Ba
 - Acid digestion of fine aliquot powders

Scanning Electron Microscopy (SEM) - Mineralogy



Zeiss EVO 15LS SEM (EDS) - 20 keV and 3 nA (5nA - mapping)

- Basalt samples only
- 5 aliquots of each basalt sample prepared as epoxy resin polished blocks
- Element Mapping, Backscattered Electron (BSE) Imaging
- Compositional Points Analysis

Electron Probe Micro-Analysis (EPMA) - Mineralogy



Cameca SX100 (WDX and EDX) - 20 keV and 20 nA

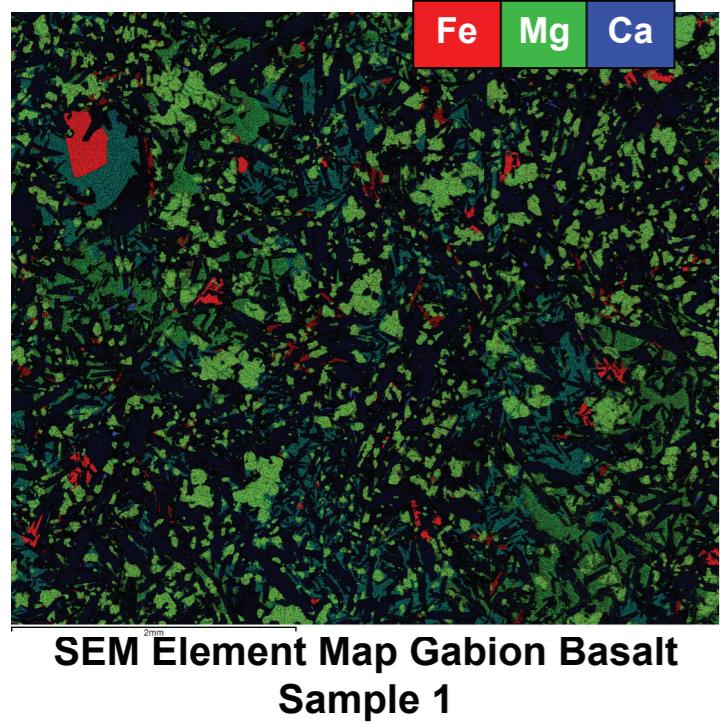
- Tested 3 aliquots basalt samples (polished blocks)
- Compositional Points Analysis and BSE Imaging only to validate SEM results

Results and Comparison with Extra-Terrestrial Chemical Data

Lunar and Martian Basalt Analogue

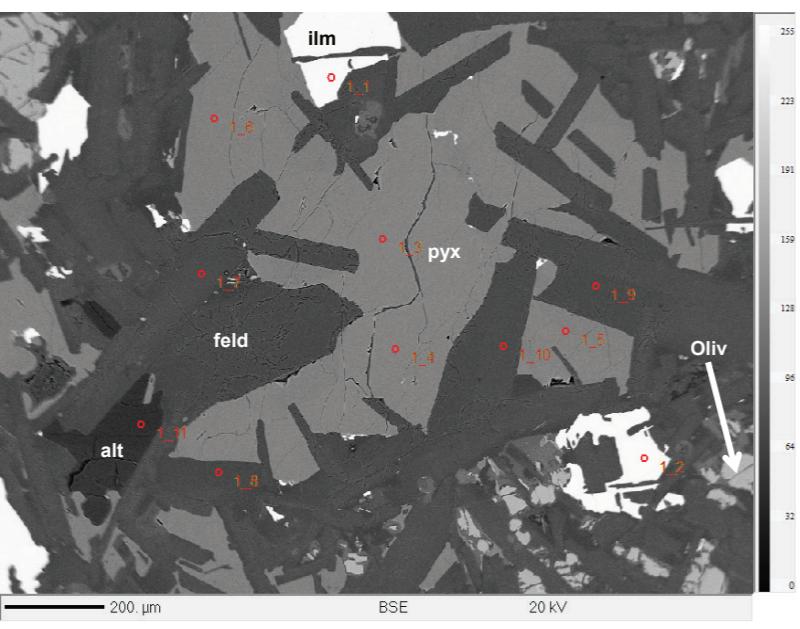
Results of the basalt aggregate and gabion samples using the XRD, SEM, EPMA and ICP techniques give an overview of the minerals and chemical signatures present. The dominant mineral phases identified are: Olivine, Pyroxene and Feldspar. Minor phases include clay/alteration and Ilmenite/titano-magnetite minerals.

In support of the analogue requirements, the mineral suite and chemistry as characterised show that the samples are indeed of basaltic igneous origin and are suitable mineralogical analogues for Martian and Lunar basalts.



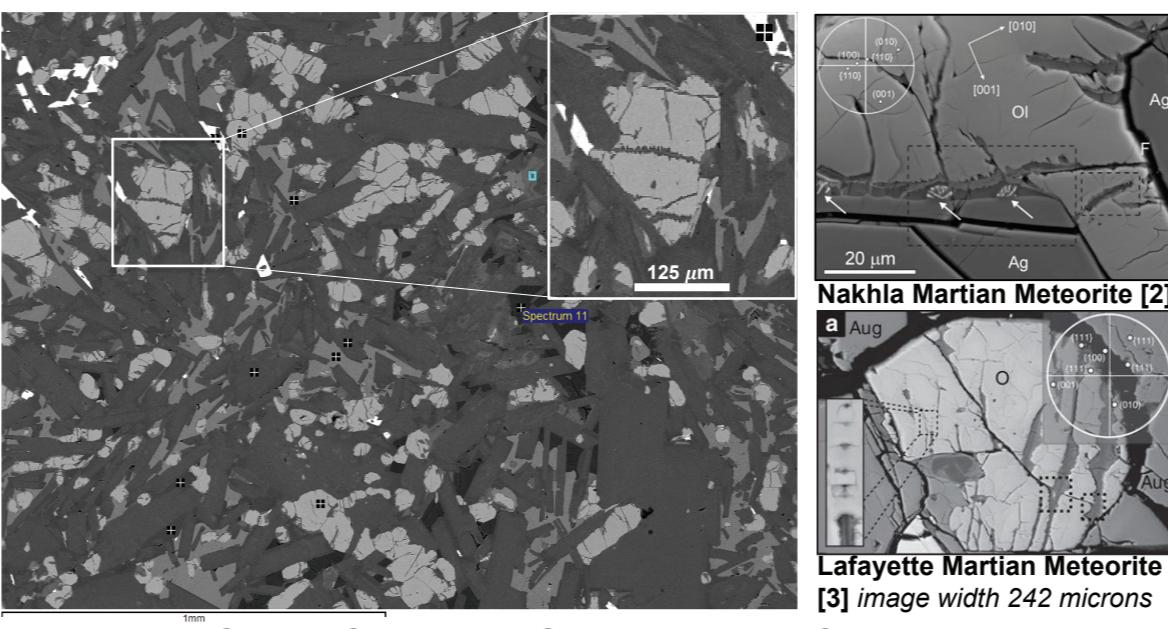
SEM Element Map Gabion Basalt Sample 1

Ilmenite/titano-magnetite - Red; Olivine - Bright Green; Pyroxene - Dark Green/Blue Green; Feldspar - Dark Blue



EPMA BSE image Gabion Basalt Sample 1

ilm = Ilmenite/titano-magnetite; oliv = olivine; pyx = pyroxene; feld = feldspar; alt = alteration

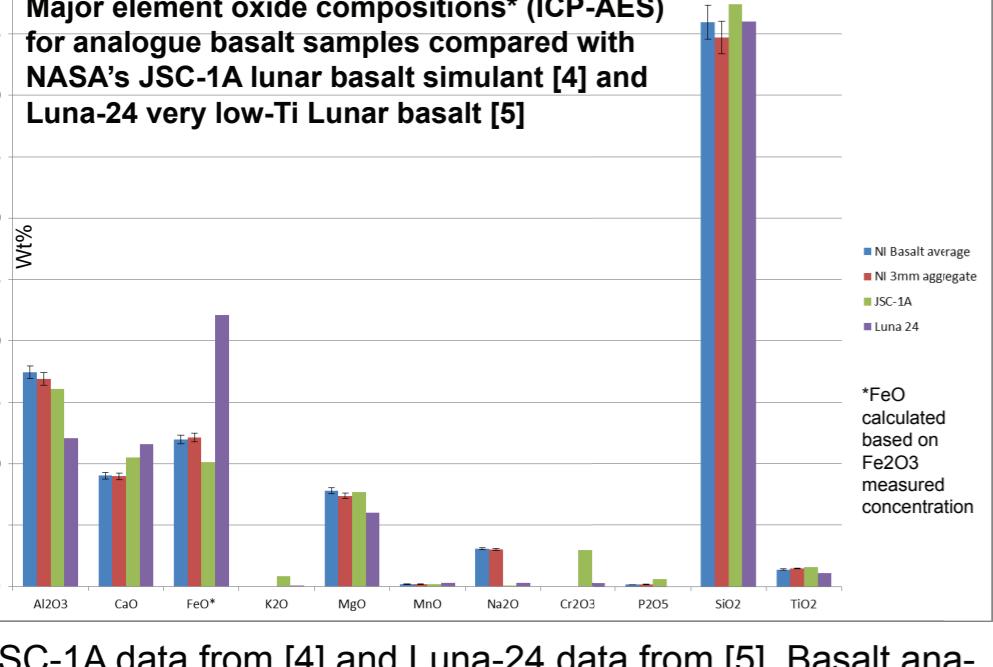


SEM BSE image Gabion Basalt Sample 4

Olivine alteration observed using SEM technique. Olivine crystal (inset) showing horizontal, linear alteration pattern with serrated, 'sawtooth' edges similar to those observed in Nakhla [2] and Lafayette [3] Martian meteorites.

Mineral Phase	Calculated amount with Estimated relative error (Wt.%)
Intermediate plagioclase/feldspar ($\text{Ca}_{50}\text{-Na}_{45}$)	65.9 ± 3.3
Olivine ($\text{Fe}_{80}\text{-Mg}_{20}$)	14.5 ± 0.7
Pyroxene (Augite-like match)	15.5 ± 0.8
Clay (Montmorillonite)	4.1 ± 0.4

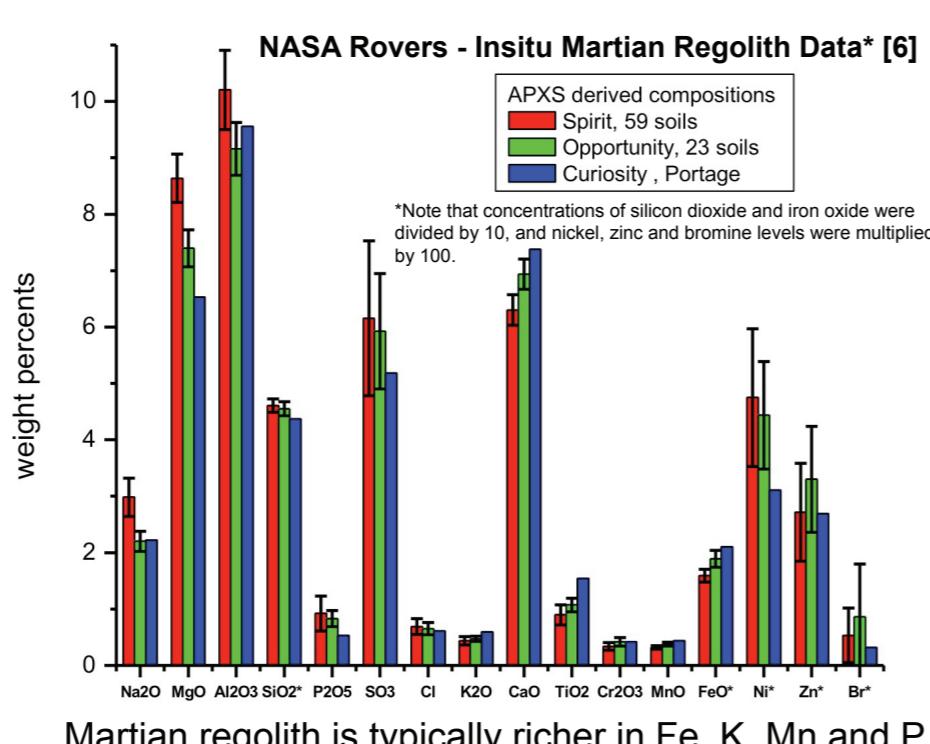
Table of results from XRD analysis of whole rock mineralogy of the basalt aggregates and gabion



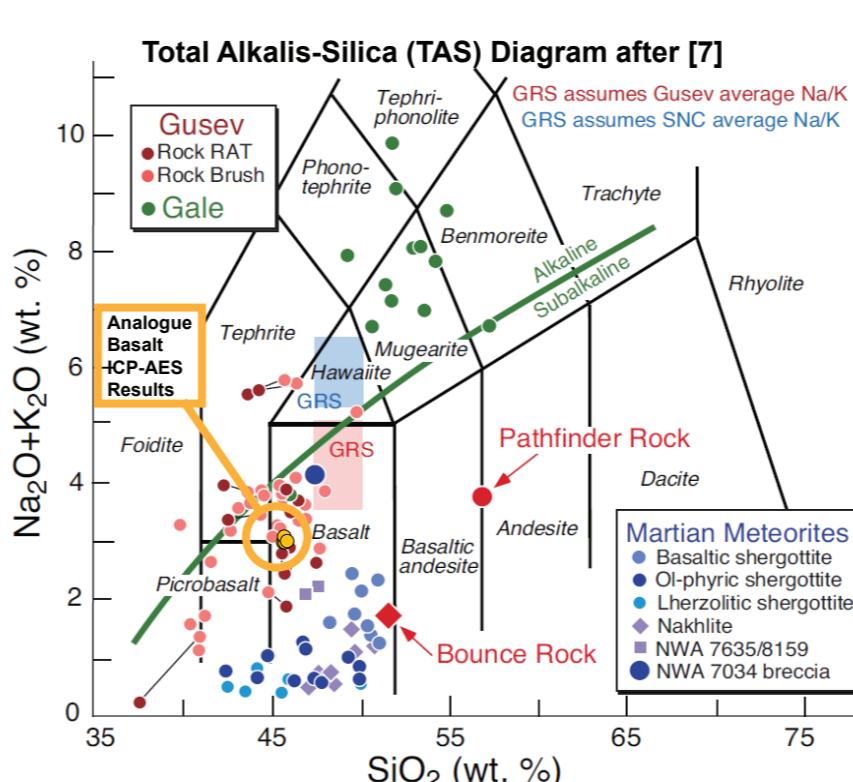
JSC-1A data from [4] and Luna-24 data from [5]. Basalt analogue samples have 3% error bars; average of 6mm, 10mm and 19mm aggregates and gabion stones. JSC-1A and our basalt analogues most chemically similar to very low-Ti Lunar basalt.

	Olivine		Pyroxene		Feldspar			
	Forsterite	Fayalite	Enstatite	Wollastonite	Ferroferrite	Albite	Orthoclase	Anorthite
SEM wt%	54-60	40-46	37-39	45-47	16-18	45-54	1-2.5	44-54
EPMA wt%	54-60	40-46	36-39	44-46	16-18	35-46	1-2	52-64

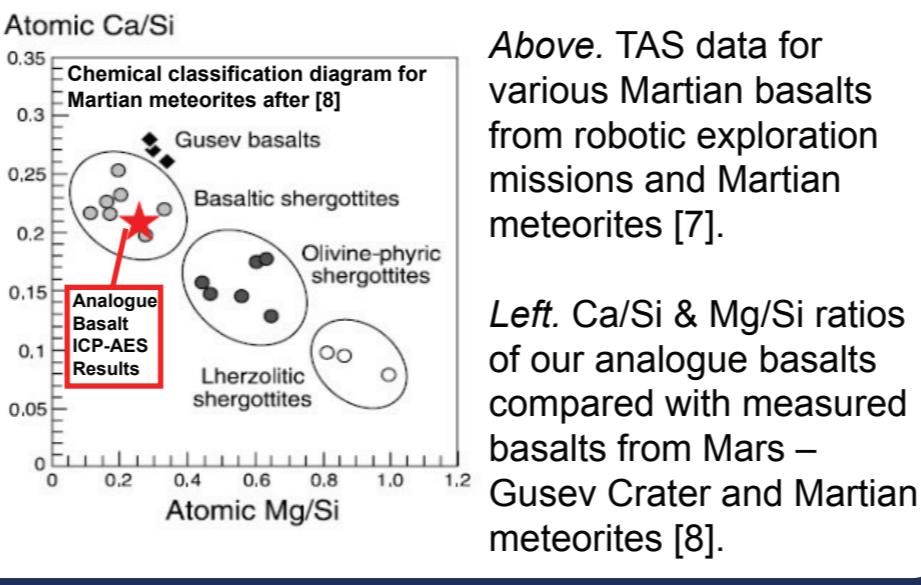
Comparison of SEM & EPMA Point analysis results showing the end member wt% in the dominant basalt minerals: olivine, pyroxene and feldspar



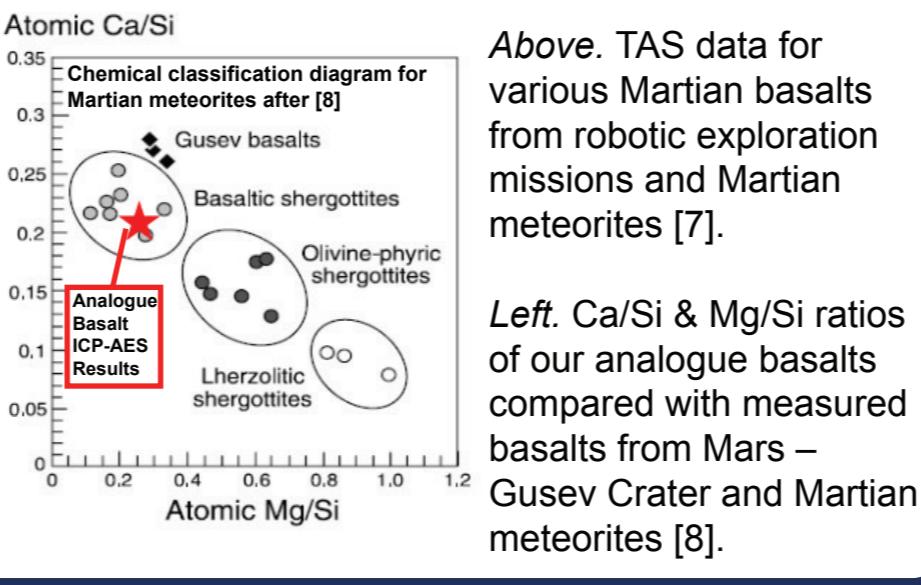
Martian regolith is typically richer in Fe, K, Mn and P but depleted in Al compared with the terrestrial basalt analogues. Al is higher in the terrestrial samples but for the other elements the abundances are similar.



Above. TAS data for various Martian basalts from robotic exploration missions and Martian meteorites [7].



Left. Ca/Si & Mg/Si ratios of our analogue basalts compared with measured basalts from Mars – Gusev Crater and Martian meteorites [8].



Mars, Phobos, Deimos, C-Type Asteroids Clay Analogues

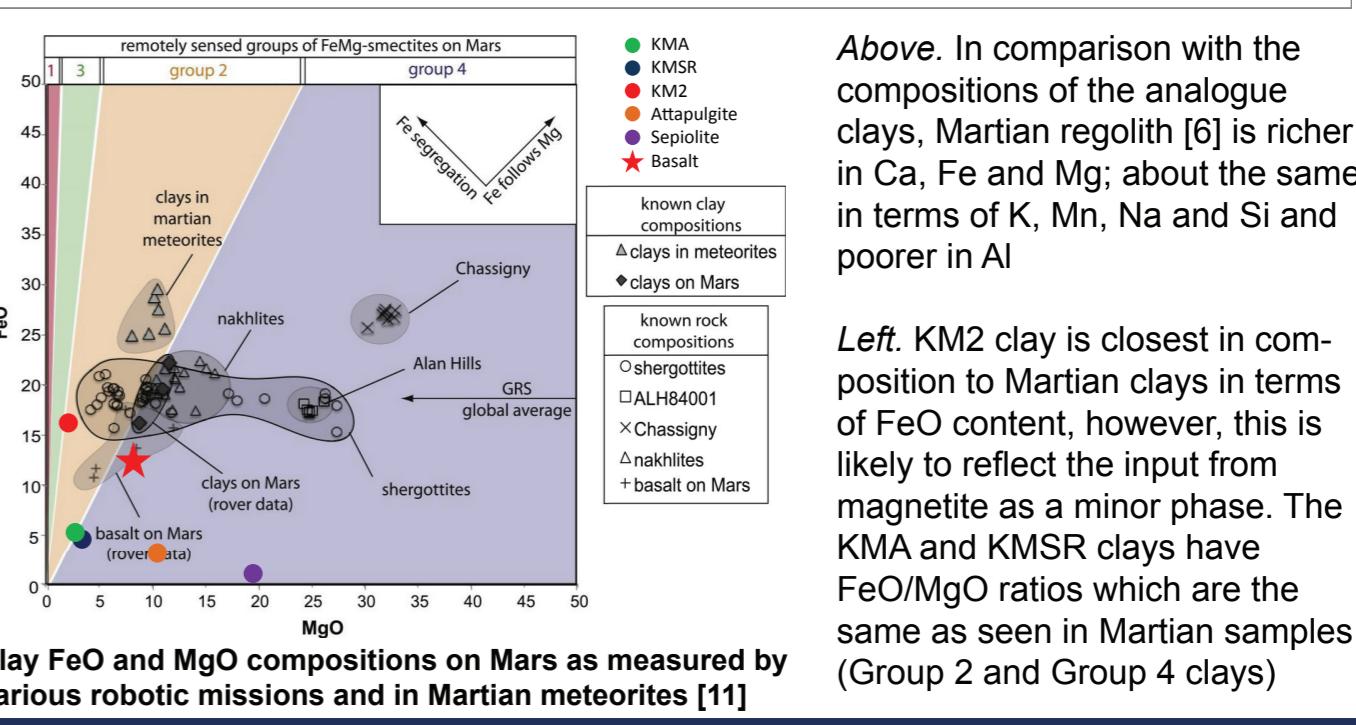
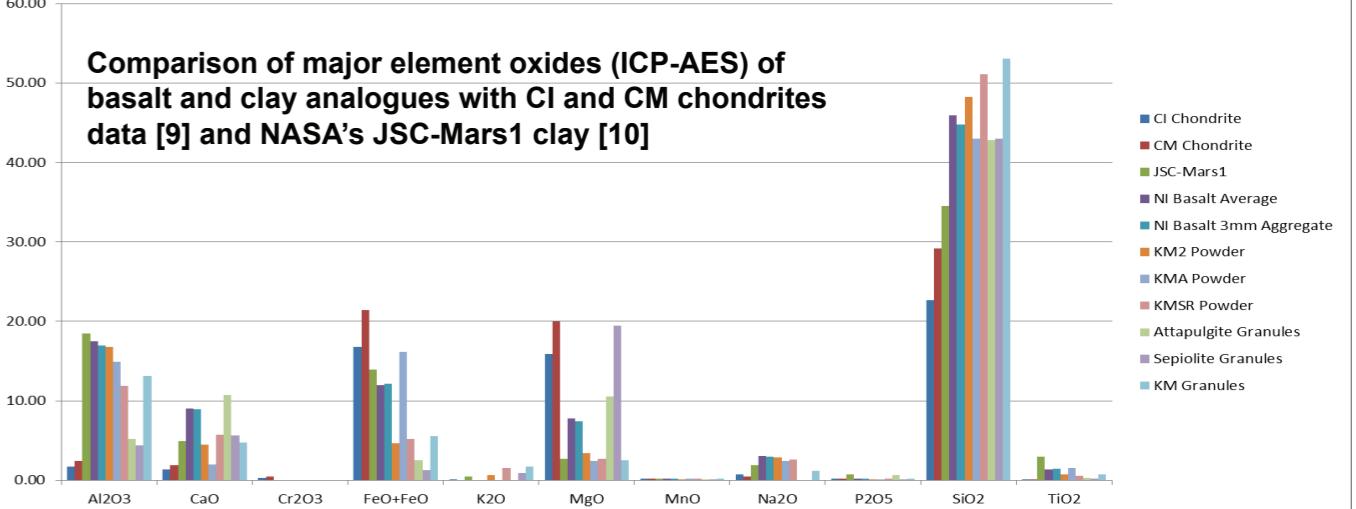
Results of the clay granular and powder samples using the XRD and ICP techniques are presented.

The dominant mineral phases identified are:

- Palygorskite-Sepiolite Group Minerals** in the granular Attapulgite and Sepiolite analogue samples
Smectite Group Minerals in the granular and powdered Bentonite Analogue Samples

Clay Sample	Main Phase(s)	Wt. %	Minor Phases	Wt. %
Attapulgite Granules	Palygorskite-Sepiolite Group Minerals (non-expandable clay) + Smectite Group Mineral component (expandable)	>90%	Ca plagioclase, quartz, dolomite	<10%
Sepiolite Granules		>95%	Calcite, dolomite, quartz, mica	<5%
KMA Bentonite Powder		>95%	Quartz, calcite, feldspar, magnetite	<5%
KMB Bentonite Powder	Smectite Group Mineral (expandable clay)	>95%	Quartz, calcite, dolomite, feldspar/plagioclase, magnetite	<5%
KMSR Bentonite Powder		>90%	Quartz, calcite, feldspar/plagioclase, mica, chlorite (?)	<10%
KM Bentonite Granules		>90%	Quartz, calcite, mica, chlorite (or kaolinite?)	<10%

Table of results from XRD analysis and quantification of the clay analogue powders and granules.



Above. In comparison with the compositions of the analogue clays, Martian regolith [6] is richer in Ca, Fe and Mg; about the same in terms of K, Mn, Na and Si and poorer in Al.

Left. KM clay is closest in composition to Martian clays in terms of FeO content, however, this is likely to reflect the input from magnetite as a minor phase. The KMA and KMSR clays have FeO/MgO ratios which are the same as seen in Martian samples (Group 2 and Group 4 clays).

Satisfying the Analogue Chemical Requirements

Following a detailed literature review in 2015, a list of analogue requirements were outlined. With the results of the analytical characterisation work, it was possible to assess that the requirements were mostly satisfied for the basalt and clay analogue chemical properties:

Requirements fully satisfied

- The Mars anhydrous silicates analogue regolith shall have a basaltic composition
- One of the Mars analogue rock analogues shall have a basaltic composition
- The lunar surface analogue regolith shall have a basaltic composition
- Lunar isolated surface rocks should have basaltic composition
- The Mars hydrated silicate analogue shall have a basaltic composition and contain clays
- The Mars hydrated silicate analogue should contain clay minerals similar to those observed on Mars

Requirements partially satisfied

- The surface regolith analogue selected for Phobos, Deimos and C-type asteroids shall have the same compositional properties
- The surface regolith and isolated surface rock analogues selected for Phobos, Deimos and C-type asteroids shall be composed of approximately 75% phyllosilicates, with approximately 25% of olivine, pyroxene and sulphides

Sample mixing would be required to meet these requirements

References

- [1] Smith et al. 2017. 48th LPSC, #1218 [2] Lee et al. 2013. Meteoritics & Planetary Science, 48(2), pp.224-240. [3] Tomkinson et al. 2013. Nature Communications, 4. [4] Taylor, 2005. sci.esa.int/Conferences/ILC2005/Presentations/Taylor-01-PPT.pdf [5] Taylor et al. 1991. Ch.6 in Lunar Sourcebook eds. Heiken et al. [6] NASA, JPL, 2012. [7] McSween, 2015. American Mineralogist, 100, 2380-2395 [8] McSween et al. 2006. Journal of Geophysical Research, 111, doi:10.1029/2005JE002477 [9] Jaroszewich, 1990. Meteoritics, 25, p. 323 [10] Allen et al. 1997. 29th LPSC, #1690 [11] 2015. Michalski et al. Earth and Planetary Science Letters, 427, 215-225
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