In Situ Compositional Measurements of Rocks and Soils with the Alpha Particle X-ray Spectrometer on NASA's Mars Rovers

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The Alpha Particle X-Ray Spectrometer (APXS) on Curiosity with the Martian landscape in the background. Image courtesy of NASA/JPL-Caltech/MSSS

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The Alpha Particle X-ray Spectrometer (APXS) is a soda can–sized, arm-mounted instrument that measures the chemical composition of rocks and soils using X-ray spectroscopy. It has been part of the science payload of the four rovers that NASA has landed on Mars. It uses ^{244}Cm sources for a combination of PIXE and XRF to quantify 16 elements. So far, about 700 Martian samples from about 50 km of combined traverses at the four landing sites have been documented. The compositions encountered range from unaltered basaltic rocks and extensive salty sandstones to nearly pure hydrated ferric sulfates and silica-rich subsurface soils. The APXS is used for geochemical reconnaissance, identification of rock and soil types, and sample triage. It provides crucial constraints for use with the mineralogical instruments. The APXS data set allows the four landing sites to be compared with each other and with Martian meteorites, and it provides ground truth measurements for comparison with orbital observations.

Keywords: Mars geochemistry, XRF, PIXE, APXS, MER, MSL, chemostratigraphy

INTRODUCTION

The alpha particle X-ray spectrometer (APXS) in the Mars Science Laboratory (MSL) is the fourth instrument of its kind that has landed on Mars. After Pathfinder (1997) and the twin Mars Exploration Rovers (MERs) Spirit (2004–2011) and Opportunity (2004–ongoing), the MSL APXS was competitively selected by NASA in 2004 to be part of the scientific payload of the rover Curiosity. It was contributed by the Canadian Space Agency (CSA). The APXS is an arm-mounted, contact instrument that measures the chemical composition of rocks and soils using X-ray spectroscopy. Bulk geochemistry is a key geological tool that complements mineralogy and imaging. It delivers crucial constraints for the interpretation of available scientific data. APXS compositional data from four landing sites, covering more than 50 km of traverse on diverse terrain, allow detailed assessment of the geology of Mars. Here we discuss the APXS method and instrument as well as some of the results from the current MER and MSL missions.

THE APXS MEASURING METHOD AND RESULTS

The APXS irradiates a sample with alpha particles and X-rays and measures the radiation emitted from the sample atoms. While it shares many principles with the standard X-ray fluorescence (XRF) technique used on Earth, it is a highly optimized version specifically adapted for the requirements of space exploration—light, robust, and easy to operate. Once the instrument has been deployed, the energy of each registered X-ray signal is determined and the spectrum is accumulated over a given time. The improvements and design changes in the MSL version are based on the experience gained in using the method during the development and operations of the Pathfinder and MER APXS instruments. Highlights of the instrument design, its evolution, and the basic physics principles underlying the method are summarized below.

The APXS has undergone significant changes from Pathfinder through MER to MSL. The Pathfinder instrument (Rieder et al. 1997) was mainly optimized for Rutherford backscattering (RBS) and was equipped with a thin alpha detector in the center of a ring of ^{244}Cm sources (Radchenko et al. 2000), which emitted 5.5 megaelectron volt alpha particles and plutonium X-rays with energies of 14.3 and 17.6 thousand electron volts onto the sample. The Si-PIN detector for X-rays was attached to the side. The MER instruments, designed and developed at the Max Planck Institute for Chemistry (Rieder et al. 2003), introduced the then new silicon drift detector (SDD), which was placed in the center of the ring of sources. However, the RBS method was found to be seriously degraded by the ~6 millibar CO_2 atmosphere on Mars, which resulted in a very high detection limit for carbon in the sample.

For MSL the alpha particle detectors were abandoned. This allowed a more compact geometry, resulting in a counting rate about three times higher than in the MER APXS.

The next design change concerned the ^{244}Cm sources. The alpha particles best excite the lower atomic number (Z) elements sodium to calcium by particle-induced X-ray emission (PIXE), while XRF excites better the higher-Z elements. The additional PIXE excitation gives the APXS a significant advantage over the pure XRF instruments on Viking, Beagle, and the Chinese lunar rover and over the CheMin XRF mode in the MSL. To essentially double the sensitivity for important high-Z trace elements, the ^{244}Cm sources, each emitting alpha particles and X-rays, were augmented by sealed ^{244}Cm sources to pass only X-rays.

With these improvements, the MSL APXS can acquire a good spectrum within 20 minutes, as shown in Figure 1; the composition extracted from this spectrum and the associated errors are shown in Table 1. A comparable analysis on MER took several hours at night. The activation of a

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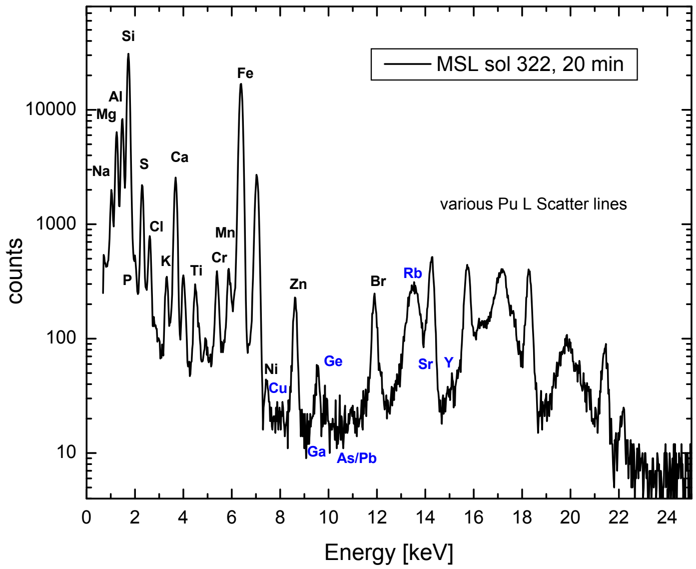

Figure 1 X-ray spectrum of the rock Ailik measured in Gale Crater on sol 322 over a 20-minute period. Plotted is the logarithm of the X-ray counts versus X-ray energy (in thousands of electron volts). The peaks labeled in black are for the 16 standard elements. The blue labels indicate elements quantifiable when in elevated abundance. The analysis procedure transforms the peak areas into concentrations of the respective element oxides.

Peltier cooler for the SDD allows acquiring several spectra during the Martian day with centimeter-scale offsets to raster the sample. This technique reveals the degree of homogeneity of the sample and can indicate the presence of certain minerals through correlated changes of the elements measured.

Compared to terrestrial XRF analyses, there are important limitations on the surface of another planet. On Earth, samples are usually melted into borate pellets to make them compositionally homogeneous. Preparing such pellets is infeasible on Mars and would also result in the loss of many geochemically important volatiles. The APXS measures a sample about 1.7 cm across—the same as the diameter of the MSL drill—and averages over all mineral grains in its field of view. While in homogeneous glasses X-ray self-absorption can be modeled very well, the APXS samples on Mars have different absorption depending on the minerals present. This is the underlying reason for minor imperfections in accuracy in the calibration with ~100 terrestrial reference materials (Table 1). Additional details of the analysis methods can be found in Gellert et al. (2006) and Campbell et al. (2012).

Table 1 COMPOSITION EXTRACTED FROM THE SPECTRUM SHOWN IN FIGURE 1. The statistical error represents precision, or repeatability. The already very precise data can be improved by overnight measurements lasting several hours. Detection limits and average relative accuracies determined during the calibration are given in the table. The last column is the composition of Portage, a soil measured on sol 89 in Gale Crater.

	Weight %	Stat error	Accuracy %	Limit of detection	Portage soil
Na_2O	2.87	0.14	11	1	2.70
MgO	9.08	0.25	14	1	8.69
Al_2O_3	8.34	0.19	7	1	9.37
SiO_2	41.6	0.54	3	1	43.0
P_2O_5	0.65	0.05	15	0.3	0.95
SO_3	6.29	0.15	15	0.2	5.47
Cl	1.11	0.04	30	0.2	0.69
K_2O	0.43	0.02	15	0.2	0.49
CaO	6.73	0.08	7	0.2	7.26
TiO_2	0.76	0.03	20	0.2	1.19
Cr_2O_3	0.79	0.03	19	0.05	0.49
MnO	0.40	0.03	8	0.05	04.1
FeO	20.7	0.26	7	0.03	19.2
Ni/ppm	208	30	16	50	456
Zn/ppm	1010	35	16	30	327
Br/ppm	393	15	20	20	34

On Mars the rovers do have some means of sample preparation. Besides measuring the sample as is, a brush can remove dust, soil, and unconsolidated alteration materials. MER has a rock abrasion tool (RAT) used to expose the interior; MSL also has a drill that delivers fines from an ~5 cm deep hole to the analytical instruments. Both the excavated fines and the aliquots of unused fines can be measured by the APXS. The results for drill cuttings typically agree well with results from the brushed surface. Systematic comparisons indicate that even an unbrushed spot typically shows clearly the underlying rock characteristics. For example, the unbrushed Jake_M rock in Gale Crater has 7% Na_2O, which is about three times more than the sodium content in the dust, indicating that either the dust coating is patchy or that the surface measurement is representative of the altered rock.

APXS RESULTS FROM THE MARS ROVERS

The scientific objective of the twin MER rovers Spirit and Opportunity (Squyres et al. 2003) was to "follow the water" by finding evidence that water interacted with the rocks and soils in the past. After achieving that by identifying many different alteration products, the MSL mission (Grotzinger et al. 2012) continued to the next step of investigating the past and present habitability in detail. The fact that APXS instruments are on all rovers makes it possible to compare and interrelate all landing sites. This part of the article will discuss only the highlights of the APXS findings. Detailed mission overviews can be found in Arvidson et al. (2010, 2011), including maps and results of the overall investigations. We will refer to the samples (rocks or soils) using the names given by the mission and discuss the first encountered rock that is usually the namesake for a class of rocks with similar chemical composition. The APXS data, spectra, and extracted compositions can be found on the NASA Planetary Data System website, http://pds-geosciences.wustl.edu/.

Spirit

Spirit landed on the plains of Gusev Crater in January 2004. APXS measurements of 6 float (non-outcrop) rocks revealed that they are all similar, primitive, olivine-bearing basalts; they are named after Adirondack, the first ever rock investigated in situ on Mars. The major elements of Adirondack basalts are somewhat similar to the global basaltic soil found at all landing sites, but they differ in many minor elements, like K, Ni, S, and Cl (Fig. 2, right). Some rocks had, on their surfaces, alteration rinds or exogenous coatings with elevated soluble elements, especially Cl (Gellert et al. 2006). Finding no evidence for extensive altered sediments, Spirit drove ~2 km towards the Columbia Hills. Already outlasting the primary mission duration of 90 sols (Martian days), Spirit began around sol 200 to explore the ~100-meter-tall hills.

There, Spirit encountered an astonishing diversity of rocks and outcrops, some of which were altered to various degrees. Relative to the global basaltic soil, the Wishstone class of

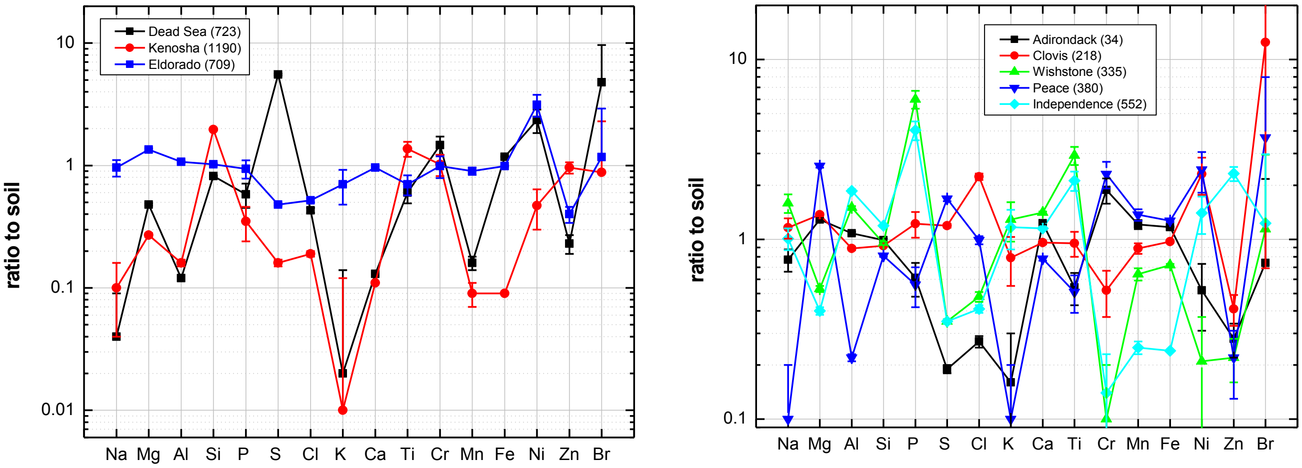

FIGURE 2 Logarithmic ratio of the standard suite of elements relative to the global basaltic soil composition (Grande Flats, sol 47) for various soils (LEFT) and rocks (RIGHT) encountered in Gusev Crater. Numbers in parentheses indicate the sol number. Similarities in elemental patterns allow one to group samples into classes. The samples shown are representative of some of the diverse compositions found. One feature of the plot is that dust or basaltic soil on rocks or admixed into the subsurface soils will skew the ratio towards 1.0.

rocks and outcrops are rich in feldspars, with elevated Na, K, Al, and Ca, low Mg and Fe, and very low Cr and Ni. A peculiar trait is their high 5 wt% P_2O_5 and nearly 3 wt% TiO_2 (Gellert et al. 2006); they are interpreted as alkaline volcanic rocks with ~10% merrillite (Usui et al. 2008). One outcrop, Independence, has very high Al and Si and lower Fe, and this composition is possibly indicative of montmorillonite (Clark et al. 2007). Clovis-class outcrops are very soft and have high levels of S, Cl, and Br, even in their abraded interiors (Ming et al. 2008). The rock called Peace has high Mg, Fe, and S and extremely low alkalis within the abraded rock, and it is interpreted as being composed of mafic minerals cemented by sulfates.

Spirit then descended the opposite side of the Columbia Hills towards a structure named Home Plate, where the first in situ detection of carbonates on Mars was made with the Mössbauer spectrometer (Morris et al. 2010). The outcrop Comanche contains about one-third Fe–Mg carbonates. The APXS suggested ~10% undetected light elements (carbon, oxygen) (Campbell et al. 2008) and facilitated quantification of the carbonates.

Due to a dragged frozen wheel, two very distinct types of bright subsurface soils were serendipitously discovered. FIGURE 2, TOP, shows that the Dead Sea soil is very rich in S, accompanied mostly by Fe and Mg. APXS scatter peaks indicated about 20% bound water (Campbell et al. 2008). These hydrated ferric sulfate–rich soils were found at several locations. A second type of whitish subsurface soil was exposed close to Home Plate (Squyres et al. 2008). Kenosha consists of ~90% SiO_2, with all other elements diminished except Ti and Cr. Virtually all these cases of aqueous alteration point towards habitable environments. The eolian dune field sample, Eldorado, is coarse-grained, mafic sand and contains lower amounts of S, Cl, and Zn. In all measurements of fine-grained soils, these elements correlate well with each other, and they are enriched in the brighter surface dust, which is interpreted to be the result of volcanic exhalations.

Opportunity

Opportunity landed 2 weeks after Spirit on Meridiani Planum. The landscape is dominated by a vast, fine-grained sandstone, which is partially covered by windblown soil, dust, and millimeter-sized spherical concretions (blueberries), released during the erosion of the soft, sulfate-rich bedrock in which they form. Blueberries contain the hematite that had been detected from orbit. The plains are punctuated by impact craters, which expose deeper parts of the layered sedimentary bedrock. This bedrock, dubbed the Burns formation, contains about 25 wt% SO_3 (FIG. 3). The APXS documented precisely the continuation of Burns formation material along the ~30 km long traverse. FIGURE 3 is a plot of elemental ratios, relative to Martian basaltic soil, of various rocks found on Meridiani. Guadeloupe, one of ~100 APXS spots in the Burns formation measured so far, is depleted in most elements compared to soil by ~20%, simply because of dilution through the addition of SO_3. Only Mg, Ni, and possibly P have higher values, indicating that Mg sulfate could be part of the salt assemblage. As Opportunity drove downhill into the ~100 m diameter Endurance Crater, it recorded a one-third drop in Mg and S and a threefold increase in Cl in abraded samples (FIG. 4),

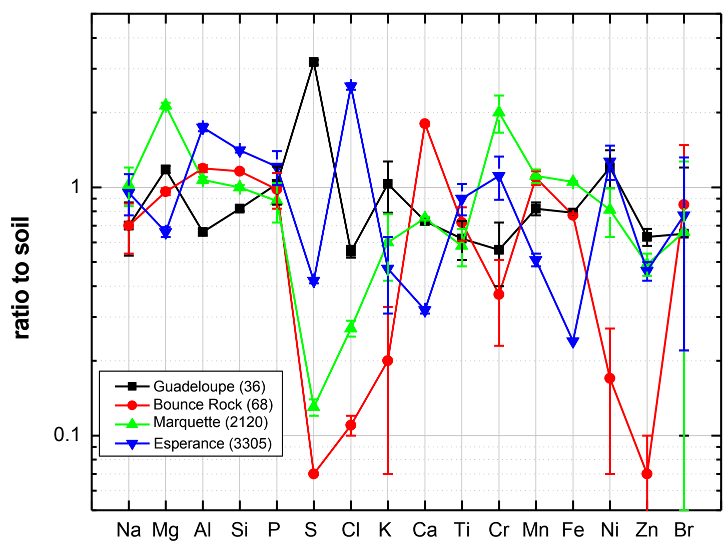

FIGURE 3 Logarithmic ratio of the standard suite of elements relative to the global basaltic soil for various rock types found on Meridiani Planum. Numbers in parentheses indicate the sol number. Guadeloupe is a rock from the Burns formation with up to 25 wt% SO_3 and elevated Mg and Ni. Esperance is an altered rock at the rim of Endeavour Crater and is rich in Al and Si. Bounce Rock and Marquette are basaltic floats, likely representing ejecta from a distant crater.

again indicating that Mg sulfate is present. The depletion in Mg sulfate is compensated by an increase in an Al silicate phase, which was interpreted as due to groundwater percolating through the bedrock, dissolving and depositing minerals (Grotzinger et al. 2005). This conclusion was later confirmed when the same chemostratigraphy was found about 8 km to the south in the much larger Victoria Crater (Squyres et al. 2009).

Iron meteorites and ejected basaltic blocks are readily identified in the bland landscape on Meridiani. Bounce Rock and Marquette, likely ejected from a crater elsewhere on Mars, plot as distinct compositions in Figure 3, extending our knowledge of basaltic rocks on Mars. Bounce Rock, a pyroxene-bearing rock, was the first rock measured on Mars that resembles a Martian meteorite in composition and mineralogy, specifically EETA79001B (Zipfel et al. 2011).

Around sol 2700, the mission entered an exciting new phase when Opportunity reached the 15 km wide crater Endeavour. The rim, which predates the Burns formation bedrock, shows evidence of the presence of clays (Arvidson et al. 2014). Clay formation could indicate a changing environment, from neutral to acidic conditions. At the rim, low-sulfur rocks with compositions similar to Martian soil were found. Two kinds of white veins—a vein 3 cm thick and protruding (Homestake) and thinner fracture fills in bedrock (Ortiz) (Squyres et al. 2012)—have high Ca and S in molar proportions corresponding to Ca sulfate. Some rocks around Endeavour show signs of alteration, possibly due to hydrothermal activity. Tisdale is high in P_2O_5 (3 wt%), with Ni, Zn, and Br up to several thousand parts per million. Esperance shows elevated Al_2O_3 and SiO_2 and very little SO_3, a composition consistent with montmorillonite. The APXS is the only functional spectrometer remaining on Opportunity. Composition alone is not sufficient for identifying clay, especially clay in small amounts. But the correlation with orbital data, local geological setting, and elemental trends provide valuable evidence for alteration.

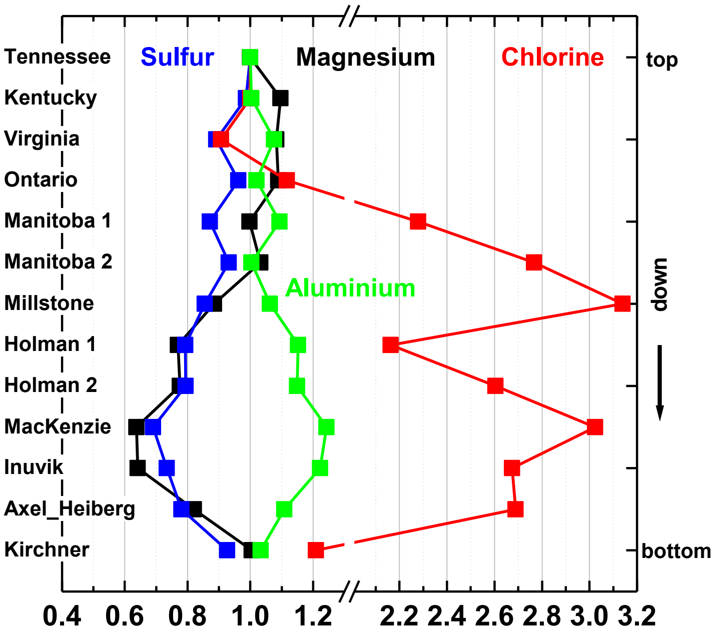

Figure 4 Compositional changes with depth in Endurance Crater; this is the first chemostratigraphic study on Mars. The elements shown are normalized to their content at the highest elevation (Tennessee). The changes indicate a decrease in Mg sulfate in deep layers and a simultaneous threefold increase in Cl.

Curiosity

The MSL rover Curiosity (Grotzinger et al. 2012) landed in August 2012 in Gale Crater, where it was to search for previously habitable environments. Gale Crater was selected because clay, hematite, and sulfate deposits had been detected in the central mound, called Mount Sharp, from orbit.

Curiosity's traverse so far is shown in Figure 5, together with the locations of samples investigated with the APXS. While the soil in Gale is very similar to the soil at all other landing sites, the first rock analyzed was a surprise. Jake_M (Fig. 6) is a feldspar-rich rock and is characteristically high in Al, Na, and K but low in Mg, Fe, Ni, and Cr; its composition resembles that of terrestrial mugearites (Stolper et al. 2013). Jake_M-like compositions were found repeatedly in float rocks, as well as in sedimentary bedrock composed of conglomerate, sandstone, and mudstone/siltstone. Overall, the Gale Crater lithologies are significantly more felsic than those from all previous landing sites (possibly excepting Pathfinder's site), with over half of all APXS measurements recording >0.7 wt% K_2O. In contrast, only one-tenth of the measurements in Gusev Crater have potassium at this level, and Meridiani has even less potassium; the global soils rarely exceed 0.5 wt% K_2O. The rock-forming particles in the sedimentary rocks are assumed to have been derived from alkaline igneous rocks located in the Gale Crater rim (Grotzinger et al. 2014; McLennan et al. 2014).

Curiosity has stopped four times at bedrock to drill samples for the SAM and CheMin instruments (see Grotzinger et al. 2015 this issue for a description of the MSL instrument package). The first two drill holes were located a few meters apart in the Sheepbed mudstone, whereas the third drill hole was located ~5 km away at Kimberley (Fig. 5). Figure 6 shows the composition of the brushed rock called Wernecke, which is part of the Sheepbed. It has one of the lowest SO_3 values (1%) measured on Mars, while the concentrations of major elements are soil-like. CheMin found about 20% clay in this mudstone (Vaniman et al. 2014). An analysis of early diagenetic, cement-filled cracks showed Fe, Mg, and Cl in proportions that are consistent with the mineral akaganeite, detected by CheMin. The mudstone at Yellowknife Bay has postlithification fractures that are filled with white minerals. The APXS raster analysis for Sajunei and the ~2 cm wide white rock Mavor indicate the presence of Ca sulfate. Two examples of dark, fine-grained sandstone, a common rock in the sedimentary record in Gale, were investigated in situ. Secure and Et Then, which are ~5 km apart, have very similar characteristics: high FeO (~26 wt %) and K, but low Mg, Ca, and Al. Both share a peculiar elevated Ga content of ~70 ppm but no significant Ge, which is usually present at levels of 50–100 ppm in the sandstone.

The third drill target, named Windjana, is a sandstone that forms part of the Kimberley outcrop. As Figure 6 shows, it is distinct from Yellowknife Bay rocks, being low in Na and Al and high in Fe (~26 wt% FeO), Mg, and especially K (>3 wt% K_2O). Windjana is also highly enriched in Zn (~4000 ppm) and Br, and overall is similar to a class of sandstones, named Bathurst, encountered around Yellowknife Bay. ChemCam LIBS shots exposed a greyish, shiny patch on a rock named Stephen with a consistently high Mn signal (Wiens et al. 2015 this issue). An APXS raster of Stephen revealed high MnO (~4 wt%), in addition to high Mg, Cl, K, Ni, Zn, Br, Cu, Ge, and Co (~300 ppm Co). A four-spot raster of Stephen shows a good correlation of Mn with Ni, Zn, and Cu. All spots have 3–3.5% Cl, the

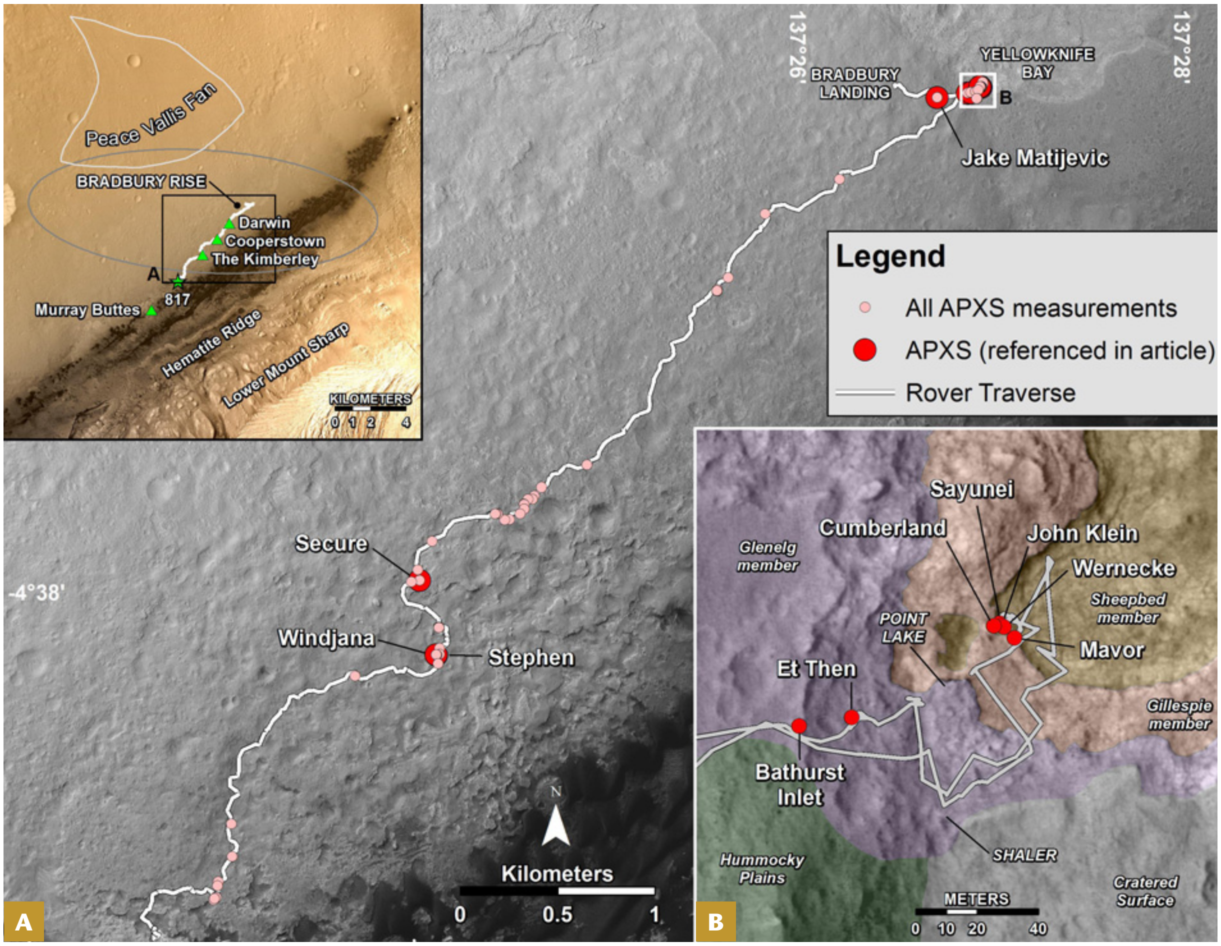

Figure 5 Locations of samples studied using the Mars Science Laboratory APXS instrument in the Aeolis Palus region. The location of panel A is shown in the inset map in the top left corner of the figure; the inset also serves to situate the rover traverse in relation to certain features on Mars, such as Mt. Sharp. The location of inset map B is shown in the top right corner of panel A.

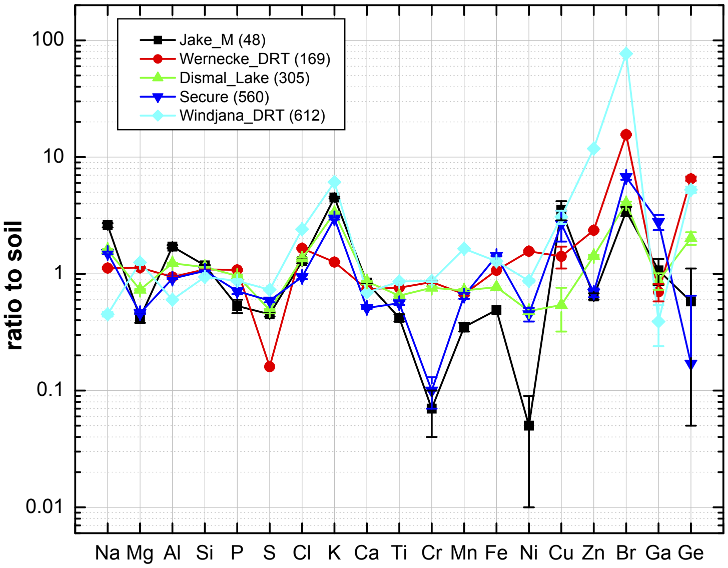

Figure 6 Logarithmic ratio of a suite of elements relative to the global basaltic soil for several rocks in Gale Crater. Several floats of the feldspar-rich Jake_M type were found with compositions nearly identical to the original Jake_M composition. Outcrops and conglomerates like Dismal_Lake show similar compositions, possibly indicating a mixing or common source region. Secure and Et Then are possibly part of the dark Capping unit. Wernecke is representative of the Yellowknife Bay mudstone that contains 30% clay. Note the general similarity in the rock compositions to soil composition, indicating isochemical weathering. The Windjana drill target at Kimberley is poorer in Na and Al but richer in K, Mg and Fe. Potassium is variably enriched in Gale Crater compared to other landing sites.

highest values measured yet on Mars. The similarity of the APXS data from Stephen to the trace metal enrichments in deep-sea Mn nodules is striking (Gellert et al. 2014).

The APXS is used for reconnaissance, drill-site selection, and many other investigations by the MSL rover. The data from the drill fines are used for XRD Rietveld analysis and to constrain the X-ray-amorphous component (Vaniman et al. 2014); the potassium measurement can be used for K–Ar dating of the rocks (Farley et al. 2014); and the contents of volatile and soluble elements, such as P, S, Cl, and Br, allow estimates of the composition of the water from which the rocks were deposited. In the future investigation of Mount Sharp, the capability of the APXS to quantify key elements will be of great importance for testing observations made by orbiter instruments and for making new, detailed, in situ discoveries of sulfates, clays, and other evidence of aqueous activity.

SUMMARY

The APXS has been used successfully on four NASA rovers. The compositional analyses of ~15 samples from Pathfinder, 220 samples from Spirit, 350 samples from Opportunity, and 150 samples from Curiosity have supported the rover science investigations and allowed detailed comparisons of the landing sites with the global soils investigated at the two Viking sites (Clark 1982). The capability to quantify bulk abundances of at least 16 geochemically important elements enables planetologists to relate new meteorites from Mars (e.g. Agee 2013) and orbiter data to all landing sites. APXS data are used to ground truth elemental and mineralogical measurements obtained from orbit.

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