

Update to Mars-based major element quantification accuracies from calibration targets of ChemCam at 3013 sols and SuperCam at 527 sols

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Key points:

- Calibration targets do not degrade over time, as evidenced by dozens of analyses over more than 3000 sols for ChemCam and 500 for SuperCam.
- Earlier conclusions about ChemCam calibration targets lack of precision and scope are supported after more than 3000 sols on Mars.
- SuperCam Mars-based errors are worse than lab results but from diverse calibration targets so may be more appropriate to use with Mars data.

Plain language summary (200 word limit)

As laser-induced breakdown spectroscopy (LIBS) becomes a common analytical tool for remote planetary surfaces, it is important to understand how performance in Earth-based calibrations relates to results obtained on planetary surfaces. Calibration targets containing geological samples/analogs with known compositions are used on the *Curiosity* and *Perseverance* rovers to test the accuracy and precision of analyses on Mars via repeated analyses. These data have been used in the past to assess spectral reproducibility and to update and optimize calibrations post-landing. This study reports test errors using Mars-based analyses and compares them to the laboratory-based errors over the lengths of the missions. The two sets of values are not the same: Mars-based accuracies for *Curiosity* are better than lab values but are from a small number of onboard samples with limited scope; *Perseverance* has poorer Mars-based accuracies than expected in the lab, but onboard standards are diverse and representative of the Mars surface. Results also establish that the onboard calibration target samples do not appear to degrade over time.

Index terms

6225 Mars, 1009 Geochemical modeling, 1060 Planetary geochemistry, 1982 Standards, 1990 Uncertainty

Keywords

ChemCam, SuperCam, calibration, Mars, LIBS, accuracy

Abstract (150 word limit)

The *Curiosity* and *Perseverance* Mars rovers include the laser-induced breakdown spectroscopy (LIBS) instruments ChemCam and SuperCam, respectively, to assess the chemistry of Mars surface materials. Onboard calibration targets (CT) are frequently analyzed to check for system health. Their spectra and predicted compositions are uploaded to the NASA Planetary Data System (PDS). The ten ChemCam CTs cover limited compositions, while the 23 SuperCam CTs are more representative of Mars. This study compares and contextualizes predicted versus actual abundances of major elements (SiO_2 , TiO_2 , Al_2O_3 , FeO_T , MgO , CaO , Na_2O , and K_2O) in the CTs. For both instruments, Mars-based calibration accuracies differ from PDS-reported values: *Curiosity* on-Mars errors are equivalent or better than lab values but from CTs with limited scope. *Perseverance* has poorer Mars-based accuracies than expected in the lab, but CTs are diverse. Results over time reveal that CT surfaces have not degraded and Mars-based calibration accuracies are mostly reproducible.

1. Background

Nearly all types of analytical instruments require calibration. Those operating remotely on planetary bodies need special care to relate performance during Earth-based testing to extraterrestrial conditions, with regular monitoring to maintain quality. Calibration targets (CT) are often included onboard remote instruments to enable evaluation of instrument performance, analytical conditions, drift over time, and to check the quality of laboratory-based predictive

models. For laser-induced breakdown spectroscopy (LIBS) instruments, CTs provide the ability to evaluate and update in-situ quantitative accuracies.

LIBS instruments on the *Curiosity* (Mars Science Laboratory, 2012) and *Perseverance* (Mars 2020) rovers use onboard CTs to measure the major element chemistry on the surface of Mars. ChemCam (Maurice et al., 2012 and Wiens et al., 2012) and SuperCam (Maurice et al., 2021 and Wiens et al., 2021), respectively, frequently analyze a subset of the CTs to evaluate ongoing instrument and calibration performance. LIBS spectra and predicted abundances of the CTs are posted on the Planetary Data System (PDS) archive alongside other analyses from Mars. These data have been used to assess ChemCam precision from sols 0-360 (Blaney et al., 2014) and to inform and update the optimal SuperCam calibration model parameters post-landing. This paper updates those findings and compiles all available Mars-based ChemCam and SuperCam major element (SiO_2 , TiO_2 , Al_2O_3 , FeO_T , MgO , CaO , Na_2O , and K_2O) calibration test accuracies using data from the onboard CTs acquired throughout the missions to date.

2. Onboard calibration targets

2.1. ChemCam

Ten calibration targets are mounted onboard the *Curiosity* rover used by the ChemCam instrument (Fabre et al., 2011). Samples were chosen with the goal of being homogeneous, long-lasting disks that are compositionally representative of expected Martian surface materials. They include graphite, Ti metal, a natural volcanic glass (macusanite), three igneous glasses (norite, picrite, and shergottite), and four sulfate-bearing ceramics (nontronite “NAU2” with high-, medium-, and low-S, and kaolinite “KGA” with medium-S; Vaniman et al., 2012). The latter eight standards are examined in this study. Compositions are given in Table 3 of Wiens et al. (2013) and **Figure 1**.

The small number of CTs and their narrow compositional range (Figure 1) relative to samples from the Martian surface is acknowledged by Clegg et al. (2017). It is also known that the S-doped ceramic CTs are heterogeneous at the scale of the laser beam (Vaniman et al., 2012), potentially leading to inconsistent spectral reproducibility.

2.2. SuperCam

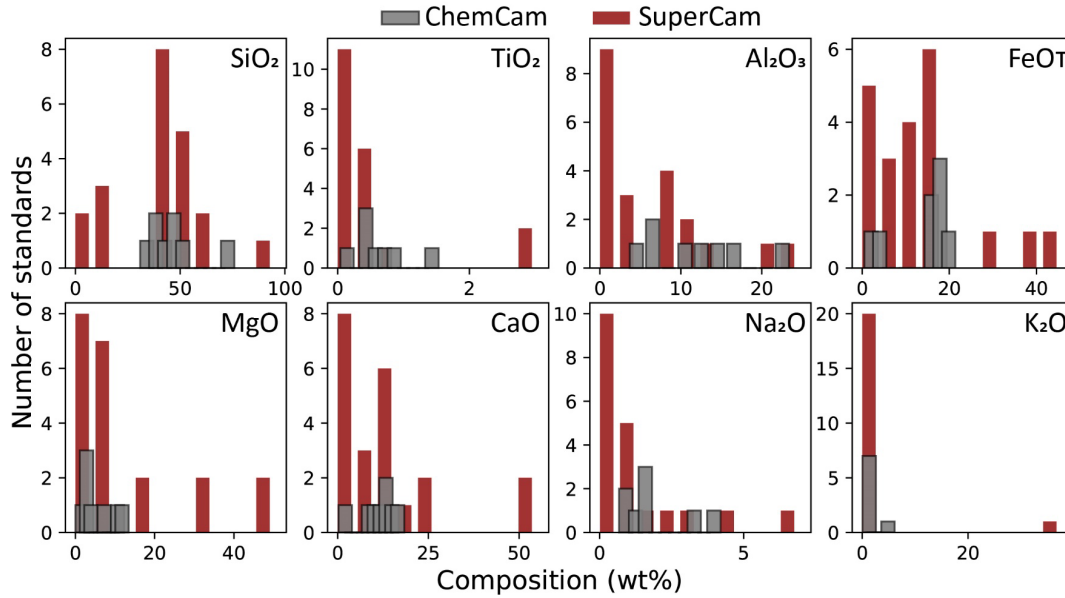


Figure 1. Histograms of the distributions of reported chemical compositions in calibration targets for ChemCam (gray, from Wiens et al., 2013) and SuperCam (red, from PDS at https://pds-geosciences.wustl.edu/m2020/urn-nasa-pds-mars2020_supercam/data_derived_spectra/supercam_libs_moc.csv).

In addition to a Ti metal plate, there are 22 calibration targets onboard the *Perseverance* rover for use with the LIBS instrument on SuperCam (see Tables 1 in Manrique et al., 2020 and Cousin et al., 2022). These homogeneous (Madariaga et al. 2022) samples include rocks, minerals, sulfate mixtures, and soil analogs, some doped with varying minor elements. Scientific rationales for calibration target choice include species detection, cross-calibration with ChemCam, weathering identification, and mineralogy (e.g., phases with known stoichiometry for elemental ratios). Compositions are found within the calibration metadata file on PDS (https://pds-geosciences.wustl.edu/m2020/urn-nasa-pds-mars2020_supercam/calibration_supercam/lib_spectral_library_reference.csv) and Table 3 of Cousin et al. (2022). All standards except Ti are examined in this study (Figure 1).

3. Data studied

3.1. Accession

NASA publicly distributes its mission data on the PDS online archive. The Geosciences Node (pds-geosciences.wustl.edu) contains datasets collected by the ChemCam and SuperCam instruments within the MSL and Mars 2020 nodes, respectively. These archives are updated in thrice-yearly releases as data continue to be collected from Mars.

Clean, calibrated LIBS data are used to generate predicted major element (expressed as SiO₂, TiO₂, Al₂O₃, FeO_T, MgO, CaO, Na₂O, and K₂O) abundances using the laboratory-developed calibration models. Predictions for each Mars analysis are included on PDS in Multivariate Prediction of Oxide Composition (MOC) tables. Though this study only uses results from the MOC tables, Python scripts that automatically extract and update the calibrated LIBS spectra datasets and MOC tables from PDS have been generated by the authors (Ytsma, 2023).

3.1.1. ChemCam data

Locations of relevant ChemCam LIBS spectra and prediction (MOC) data are described in the Data Availability Statement in the Open Research section. The current major element ChemCam calibration models are based on 408 standards and described in Clegg et al. (2017), which replaced the initial models from a smaller dataset (Wiens et al., 2013).

3.1.2. SuperCam data

Locations of relevant SuperCam LIBS spectra and prediction (MOC) data are described in the Data Availability Statement in the Open Research section. SuperCam major element calibration models are derived from 334 standards and described in Anderson et al. (2022).

3.2. Calibration errors

LIBS calibration uncertainties listed on PDS and in the literature are calculated as root-mean-squared errors (RMSE). This method of error calculation increasingly penalizes predictions the farther away they are from the true values and is intuitive because it is in the same units as the analyte (wt%, in this case). Uncertainties of measurements made in-situ are taken from test errors, or RMSEs of prediction (RMSEP), which are calculated from the error in predictions made on data unseen by the calibration model. All errors referenced in this paper are RMSEPs but are referred to as RMSEs.

Reported RMSEs for the ChemCam calibrations are variable – each measurement within the MOC table has its own RMSE value for each major oxide, calculated as a function of element composition. Derivation of this sample-specific uncertainty is described in Section 5.12 of Clegg et al. (2017), where Table 1 also gives overall MOC test values per major oxide. To generate an uncertainty for these models in the context of this study, the average posted RMSE values from PDS are used as the overall laboratory-based error and the standard deviation provides its range.

Table 1. Laboratory-based RMSEs for ChemCam and SuperCam from PDS versus RMSEs observed on Mars from predictions of calibration target compositions.¹

	ChemCam RMSE (wt%)		ChemCam RMSE (% of median CT composition)		SuperCam RMSE (wt%)		SuperCam RMSE (% of median CT composition)	
	PDS ²	Mars	PDS	Mars	PDS ³	Mars	PDS	Mars
SiO₂	5.26 ± 0.24	1.30 ± 0.65	12% ± 1%	3% ± 1%	6.1	11.30 ± 2.86	13%	24% ± 6%
TiO₂	0.40 ± 0.06	0.36 ± 0.54	78% ± 12%	70% ± 105%	0.3	0.45 ± 0.05	214%	321% ± 36%
Al₂O₃	3.42 ± 0.20	5.36 ± 0.69	30% ± 2%	46% ± 6%	1.8	6.69 ± 0.48	44%	165% ± 12%
FeO_T	3.36 ± 0.51	0.98 ± 0.27	21% ± 3%	6% ± 2%	3.1	4.70 ± 1.23	24%	36% ± 9%
MgO	1.74 ± 0.15	0.06 ± 0.04	69% ± 6%	2% ± 2%	1.1	7.33 ± 1.15	17%	112% ± 18%
CaO	3.12 ± 0.53	1.52 ± 0.54	26% ± 4%	13% ± 5%	1.3	2.38 ± 0.20	11%	21% ± 2%
Na₂O	0.59 ± 0.04	1.65 ± 0.65	40% ± 3%	112% ± 44%	0.5	2.88 ± 1.11	52%	298% ± 115%
K₂O	0.60 ± 0.09	0.55 ± 0.31	286% ± 43%	262% ± 148%	0.6	0.68 ± 0.03	353%	400% ± 18%

¹ Values provided in abundance (wt%) and in context of the standards' compositions (% of median).

² Average ± standard deviation of all RMSEs reported in MOC table. Also see reported MOC test values from Table 1 of Clegg et al. (2017): ±5.30 wt% SiO₂, ±1.03 wt% TiO₂, ±3.47 wt% Al₂O₃, ±2.31 wt% FeO_T, ±2.21 wt% MgO, ±2.72 wt% CaO, ±0.62 wt% Na₂O, ±0.82 wt% K₂O.

³ Agree with RMSEP values in Table 5 of Anderson et al. (2022).

For SuperCam, reported laboratory-based RMSE values are published in Table 5 of Anderson et al. (2022) and duplicated in the PDS MOC.

Mars-based calibration test errors for both instruments are calculated here by sorting analyses into 100-sol groups, calculating the RMSE for each CT from analyses within that time period, and averaging those values for each element. Mars-based RMSEs are averages of these 100-sol results. The time-resolved errors are used to observe potential changes in model accuracy over the lifetime of the instrument as well as to investigate the robustness of target-specific predictions, using ChemCam targets Shergottite and NAu2 Hi-S as examples. Mars-predicted values for each CT and element are calculated average values per target from all available analyses. These predicted compositions are compared to the true abundances to provide R² correlation metrics.

4. Results and discussion

4.1. Laboratory- versus Mars-based accuracies

Table 1 contains cumulative Mars-based calibration errors of major oxides versus those reported for ChemCam and SuperCam from laboratory calibrations. These results are also contextualized to the CT compositional ranges by dividing the RMSE by the median concentration for each major oxide. Laboratory- and Mars-based errors vary widely by element for both instruments.

Figure 2 shows the averages and standard deviations of Mars-predicted major oxide concentrations of each CT versus their actual compositions (also found in **Supplemental Table**

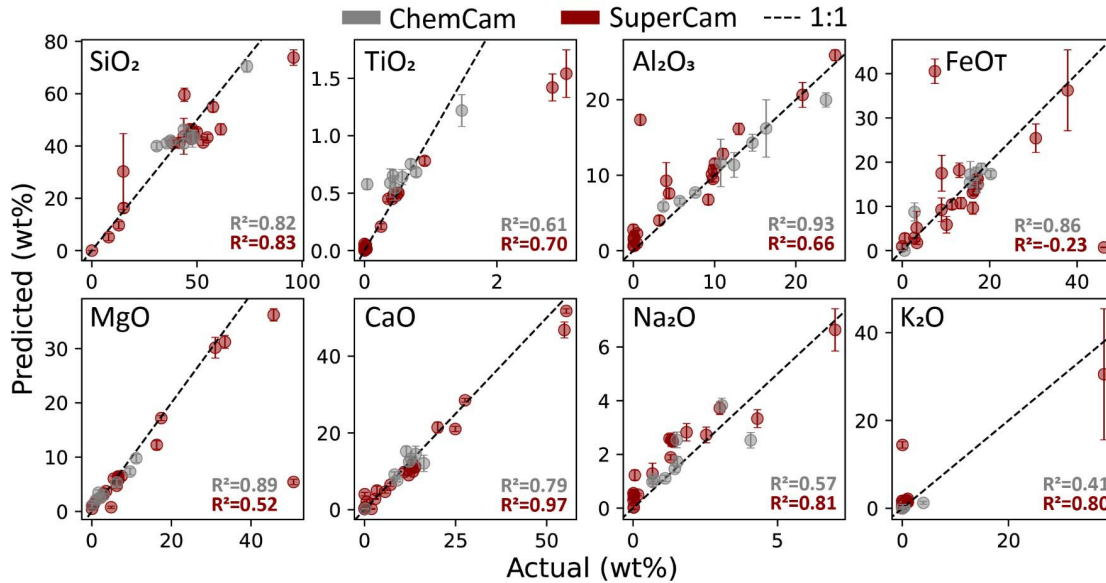


Figure 2. Overall model-predicted versus true abundances of ChemCam (gray) and SuperCam (red) calibration targets for each major oxide. The x-axis values come from Wiens et al. (2013) for ChemCam and PDS for SuperCam. The y-error bars are standard deviations of the predictions.

1). As seen in Figures 1 and 2, the number and range of CT compositions is much larger for SuperCam than ChemCam for all elements. These differences contribute to consistently higher Mars-based RMSE values reported for SuperCam in Table 1 (greater median concentrations) and also for the differences in R^2 correlations (more samples means individual outliers have less impact) shown in Figure 2. The more diverse SuperCam CTs are consequently better exemplars of prediction quality for unknowns across Mars and a more robust test dataset for calibration optimization. This comes at the expense of deceptively low errors as observed in the ChemCam CT results.

4.2. Changes over time

Because of their importance, the CTs on both Mars missions have been analyzed dozens of times, raising the concern that their surfaces might degrade over time. It also is possible that heavy elements might be preferentially re-deposited over volatiles on the surfaces of each CT as the plasma dissipates, causing changes in CT composition over time.

4.2.1. Instrument-wide

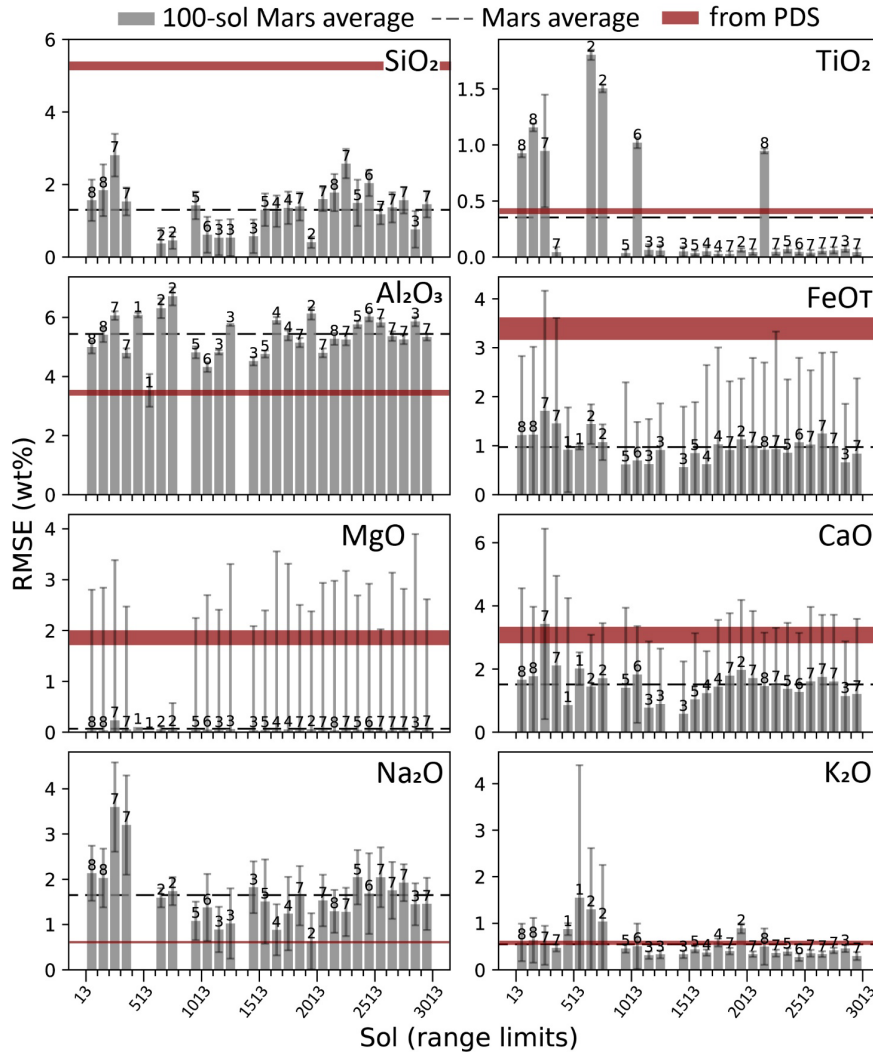


Figure 3. ChemCam-reported RMSE values based on pre-flight laboratory calibration (red box) versus RMSE values from calibration targets on Mars (dashed line and gray bars, error bar = standard deviation among CT RMSEs), sorted into 100-sol ranges by element expressed as wt% oxide. Numbers above gray bars represent the number of calibration targets analyzed during each time period. Red boxes represent the range of RMSE values reported on PDS (Table 1): y-value = average value, height = standard deviation.

149 Mars-based RMSE values for analyses grouped every 100 sols are shown and compared to
 150 laboratory values for ChemCam in **Figure 3** and SuperCam in **Figure 4**. Both figures show that
 151 model quality does not seem to systematically decay or change over time for any element, and
 152 that the overall, averaged RMSE (Table 1) is representative at any time period.

153 Figure 3 shows that geochemical predictions of CT composition are irregular but maintain a
 154 roughly constant average over the 3013 sols of the ChemCam mission. Improved average
 155 accuracies for some elements versus the lab-based values (e.g., SiO₂, MgO, FeOT) are likely
 156 because the limited CT compositions match well to the chemistries of the calibration set.

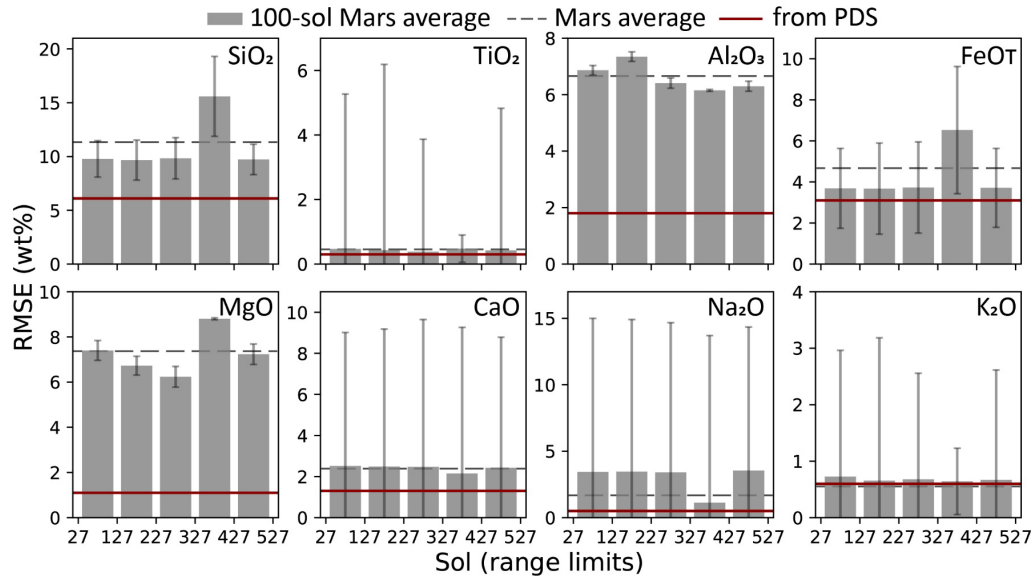


Figure 4. SuperCam-reported RMSE values based on pre-flight laboratory calibration (red horizontal line) versus RMSE values from calibration targets on Mars (gray bars, error bar = standard deviation among CT RMSEs), organized by 100-sol range and major element expressed as wt% oxide. Each time period contains measurements from 22 standards except sols 327-427, in which only nine standards were analyzed.

Variation in RMSE values among the CTs (error bars in Figure 3) is caused by inconsistent model performance among the suite of CTs; i.e., some are predicted more accurately than others, as seen in Figure 2.

SuperCam results show more consistent (i.e., more precise) RMSE values based on Mars CTs over time. Though SuperCam data are from a shorter time period and may change over time, SuperCam CTs have proved homogeneous (Madariaga et al., 2022), unlike some on ChemCam (Vaniman et al., 2012). Elemental differences are similar to ChemCam results; for example, less abundant oxides (TiO₂, CaO, K₂O, and Na₂O) exhibit much greater variability in performance among CTs and, as a result, less precise overall prediction accuracies.

4.2.2. Calibration target-specific

To further examine the longevity and reliability of the CTs over the lifetime of the missions, **Figure 5** shows predicted values of the ChemCam CTs **a)** igneous glass “shergottite” and **b)** S-doped ceramic nontronite “NAu2 Hi-S” over time versus their actual abundances.

If LIBS spectra of these samples are reproducible, then the predicted values should be nearly identical over repeat analyses, as observed for most elements within shergottite (excepting an unusual period from 213-313 sols). The shergottite was also used by Blaney et al. (2014) to

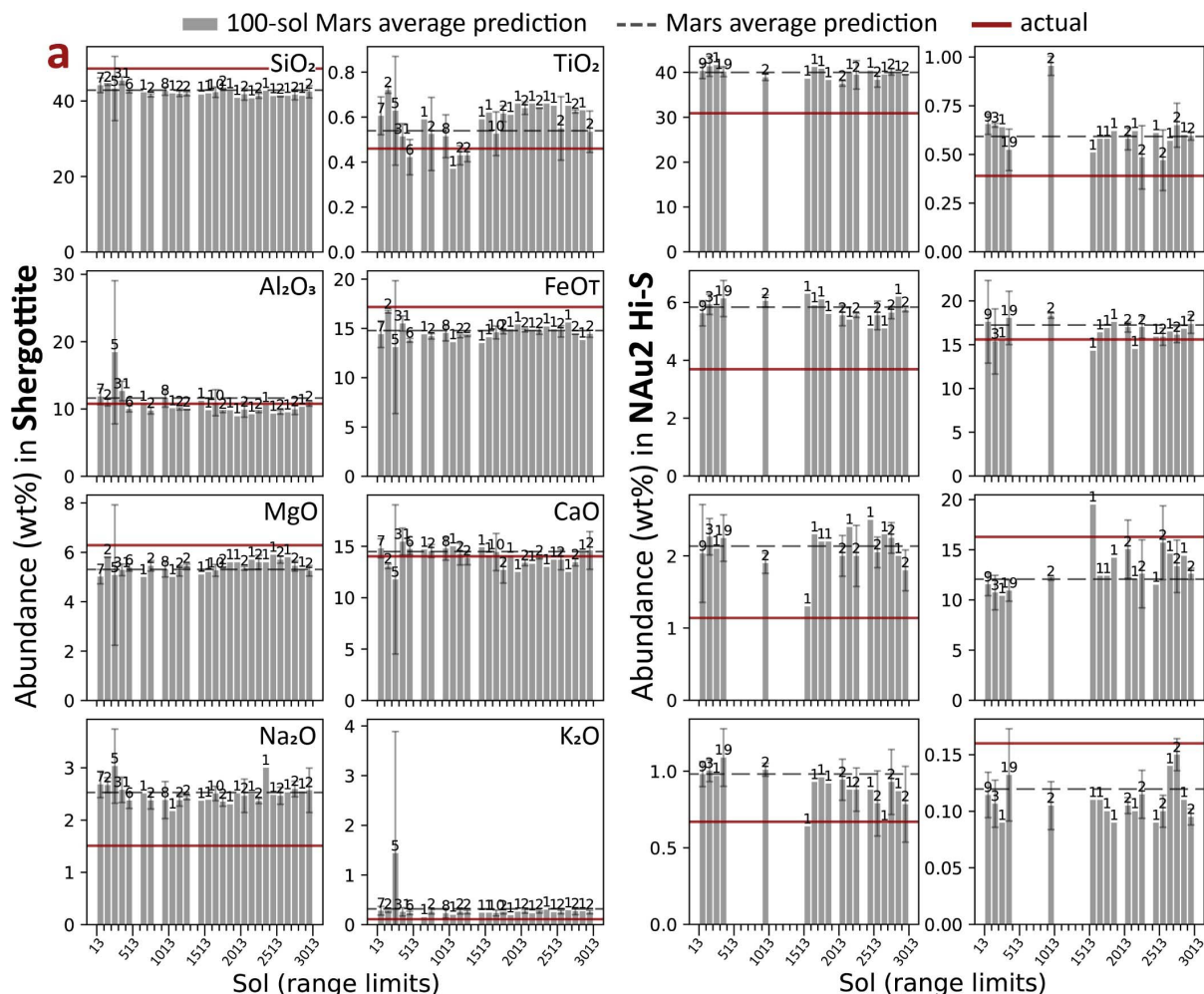


Figure 5. ChemCam predictions for calibration targets as given on the PDS site, for **a)** ‘Shergottite’, an igneous glass, and **b)** ‘NAu-2 Hi-S’, a nontronite ceramic containing high amounts of sulfate. Average predictions (gray bars) and their standard deviations (y-errors) are compared to actual values (red horizontal lines) per 100-sol range and major oxide. Numbers above bars represent the number of analyses during that time period. Layout of b) panels matches the layout of a) oxides.

examine precision from 25 analyses over sols 271, 352, and 357 (Supplement Table). Precision for Al_2O_3 , MgO , CaO , Na_2O , and K_2O is significantly better over the longer timeframe and the other elements are comparable. Evidence of homogeneity is seen in the results for NAu2 Hi-S, which shows large variety in the predicted abundances of most major elements, and is attributed to sample heterogeneity at scales larger than that of the laser beam (Vaniman et al., 2012).

5. Implications

It is often the case that remote instruments undergo changes during launch and cruise that may affect their performance upon implementation. Orbital instruments perform in-flight calibrations that then validate and improve instrument performance (e.g., Xaypraseuth, 2007; Mahanti et al.,

2016). For LIBS instruments, the best diagnostics come from utilizing the on-board CTs. Some differences between terrestrial and on-Mars results were acknowledged by the ChemCam team (Clegg et al., 2017), which implemented a channel-by-channel Earth-to-Mars correction. Calibration transfer methodology was improved by Thomas Boucher (2018) but not implemented for ChemCam; it is now part of the Python Hyperspectral Analysis Tool used to process SuperCam data (Laura et al., 2022).

How should the results presented here be utilized by the planetary community? For ChemCam, the message is mixed. In general, the analysis presented here support the conclusions of Blaney et al. (2014) that the restricted number and compositional range of CTs has limited the scope of Mars-based calibration accuracies, such that laboratory-based errors (Clegg et al., 2013) are more reliable.

For SuperCam, the accuracies given in Anderson et al. (2022) regularly overestimate the accuracy of on-Mars measurements of the 22 studied CTs, despite optimizing calibration models based on post-landing analyses of the CTs. This may result from the lack of appropriate calibration transfer methods; however, Laura et al. (2022) indicates that investigations of calibration transfer corrections are ongoing and have the potential to improve Mars-based predictions. For now, users of SuperCam data may consider using the Mars-based values reported here for more realistic estimates of current prediction accuracy.

Finally, our results provide reassurance that the CTs on both ChemCam and SuperCam remain viable and robust despite ongoing excavation by the impinging lasers. In particular, the performance life of the ChemCam CTs is impressive and lends confidence to their ongoing use during its extended mission.

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Open Research

Data Availability Statement

All predictions and LIBS analyses from NASA ChemCam and SuperCam instruments used by this study are publicly and freely available in the Planetary Data System archive via Mars Science Laboratory and Mars 2020 nodes in the Geosciences Node at pds-geosciences.wustl.edu. CHEMCAM: The appropriate repository (i.e., clean, calibrated spectra from Mars) location is https://pds-geosciences.wustl.edu/msl/msl-m-chemcam-lib-4_5-rdr-v1/mslccm_1xxx/data/ and the data release schedule is found at https://pds-geosciences.wustl.edu/missions/msl/index.htm#Release_Schedule. Relevant data are .csv files with 'ccs_' in the filename (see description at <https://pds-geosciences.wustl.edu/msl/msl-m->

[chemcam-libs-4_5-rdr-v1/mslccm_1xxx/catalog/ccam_libs_rdr_ds.cat](https://chemcam-libs-4.5-rdr-v1/mslccm_1xxx/catalog/ccam_libs_rdr_ds.cat)). Mean spectra, which are averages of 50 shots on the sample location, are used for quantification. Predicted value (MOC) tables are grouped by ranges of sols at https://pds-geosciences.wustl.edu/msl/msl-m-chemcam-libs-4_5-rdr-v1/mslccm_1xxx/data/moc/. SUPERCAM: The appropriate repository location is https://pds-geosciences.wustl.edu/m2020/urn-nasa-pds-mars2020_supercam/data_calibrated_spectra/ and the data release schedule is found at https://pds-geosciences.wustl.edu/missions/mars2020/index.htm#Release_Schedule. Relevant data are within .fits files that have a 'CL_' product name (see Tables 5-1 and 6-4 in https://pds-geosciences.wustl.edu/m2020/urn-nasa-pds-mars2020_supercam/document/M2020_SuperCam_EDR_RDR_SIS.pdf; see also https://pds-geosciences.wustl.edu/m2020/urn-nasa-pds-mars2020_supercam/document/SuperCam_Bundle_SIS.pdf). MOC are based of the mean spectra of 30 shots – found in the statistics table – and are updated in a single .csv file at https://pds-geosciences.wustl.edu/m2020/urn-nasa-pds-mars2020_supercam/data_derived_spectra/supercam_libs_moc.csv.

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Figure 1.

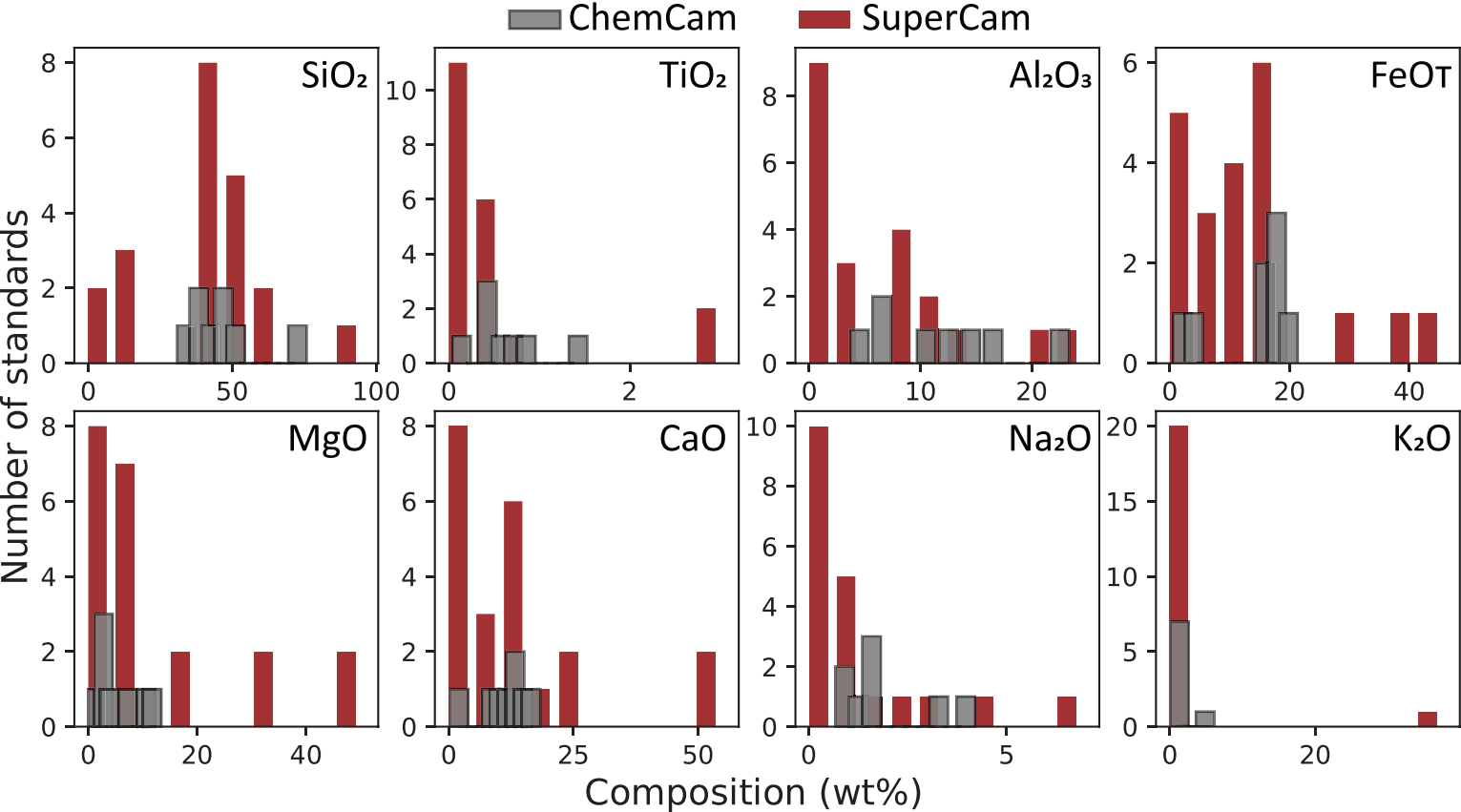


Figure 2.

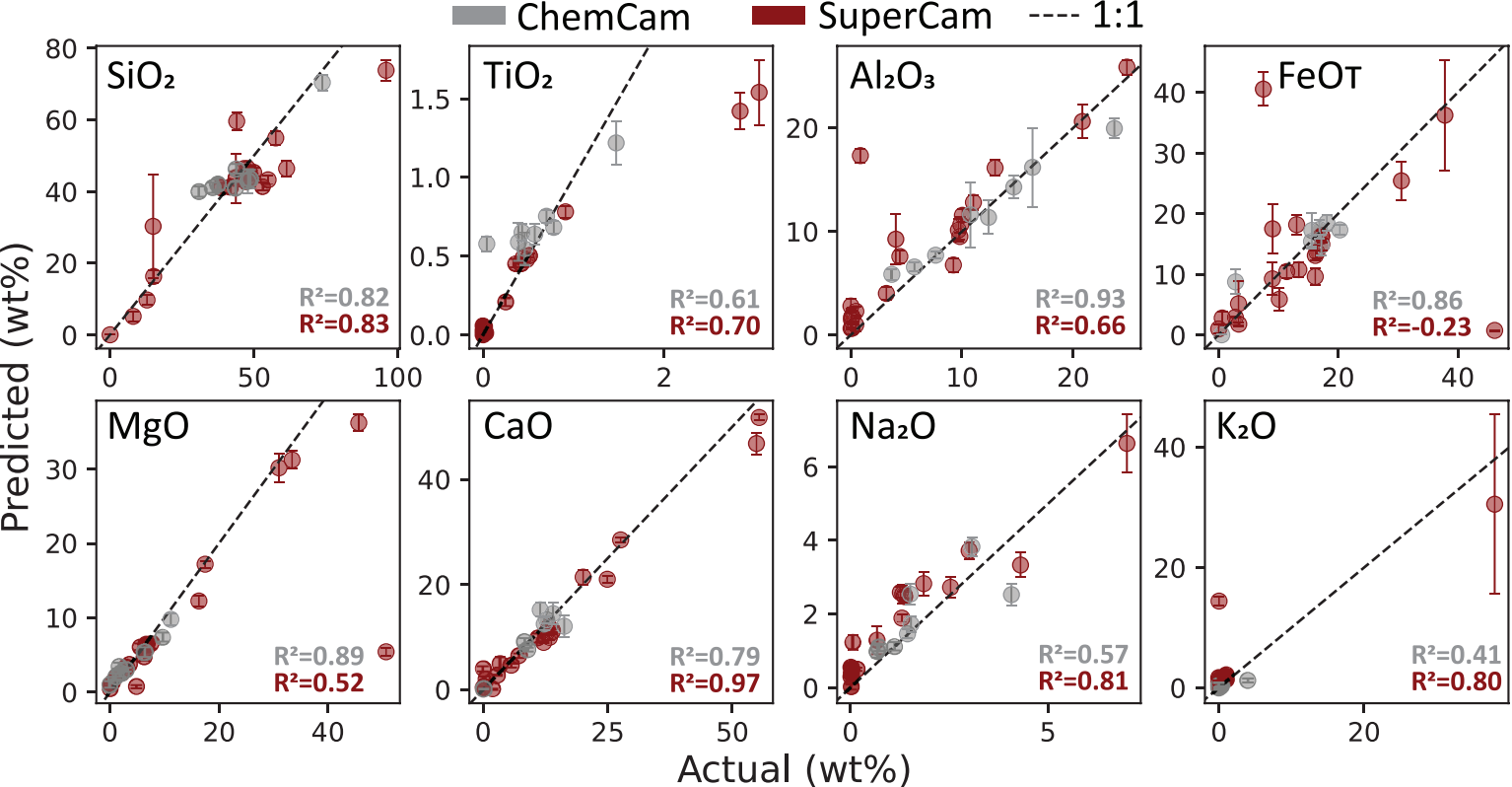


Figure 3.

■ 100-sol Mars average - - - Mars average ■ from PDS

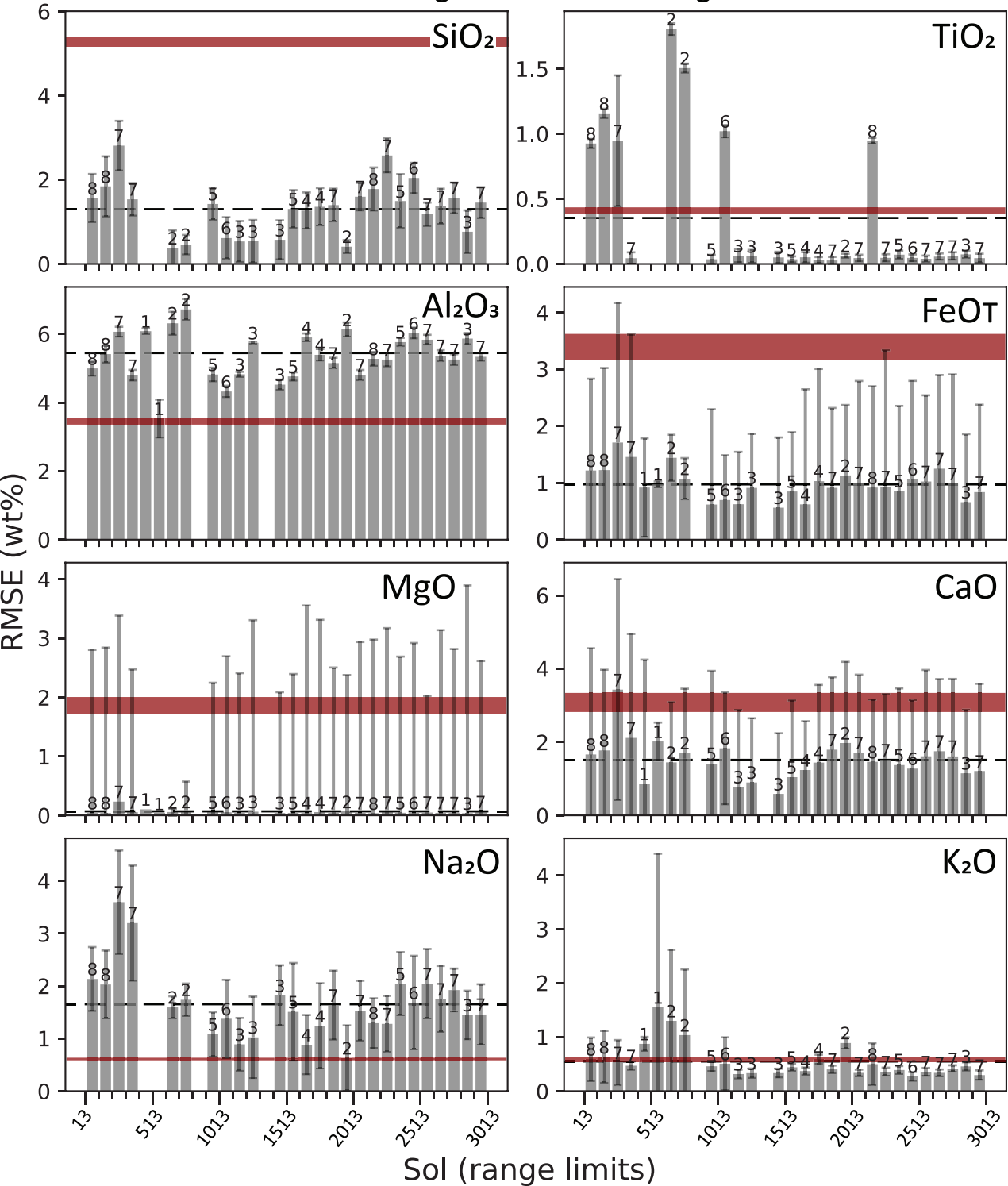
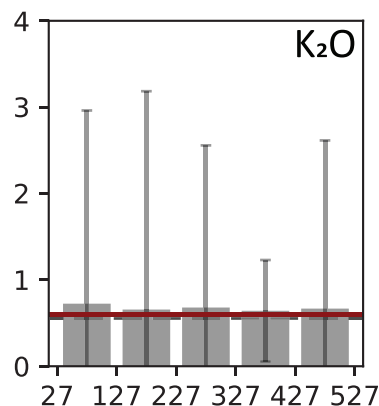
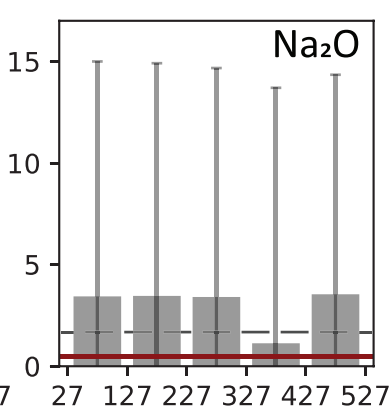
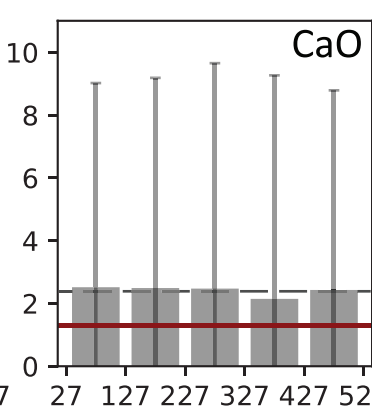
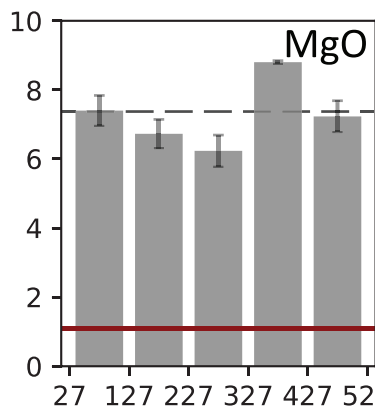
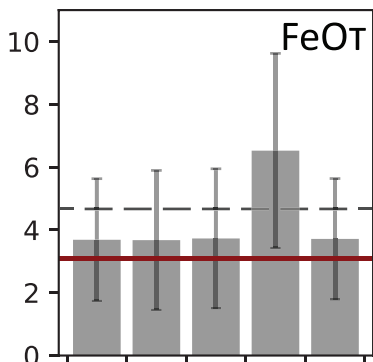
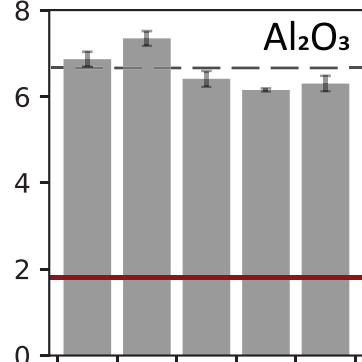
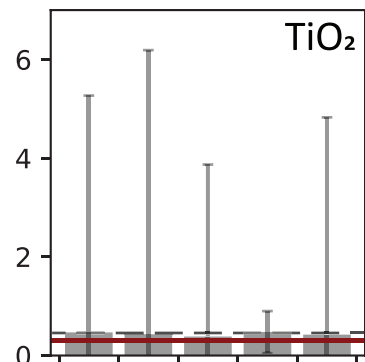
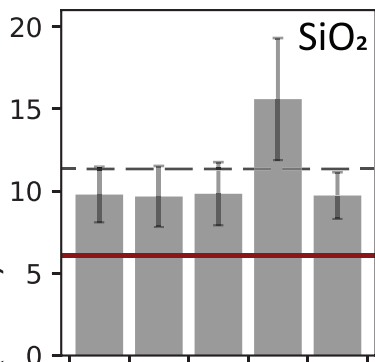


Figure 4.

■ 100-sol Mars average --- Mars average — from PDS

RMSE (wt%)



Sol (range limits)

Figure 5.

