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# Design and Specifications for the Highland Regolith Prototype Simulants NU-LHT-1M and -2M

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### LIST OF ACRONYMS AND SYMBOLS

 $AN_{XX}$  relative abundance of the anorthite mineral endmember in the plagioclase

solid solution series

CIPW Cross, Iddings, Pirsson, and Washington

KREEP potassium, rare-earth elements, and phosphorus

KSC Kennedy Space Center

LHT lunar highlands type

LOI lost on ignition

MSFC Marshall Space Flight Center

S sulfur

TM Technical Memorandum

U.S. United States

USGS U.S. Geological Survey

# TECHNICAL MEMORANDUM

# DESIGN AND SPECIFICATIONS FOR THE HIGHLAND REGOLITH PROTOTYPE SIMULANTS NU-LHT-1M AND -2M

#### 1. INTRODUCTION

In discussions with the Marshall Space Flight Center (MSFC), it was agreed that the first prototype regolith simulants produced by the United States (U.S.) Geological Survey (USGS) would simulate the lunar highlands regolith predicated on the NASA decision to put a base at one of the lunar poles that are highlands sites. It was also agreed that these simulants would be based on sample information for the Apollo 16 site because it is the only Apollo site that is close to being representative of the lunar highlands since it is more than 200 km away from the nearest mare. However, compositional modeling and petrographic studies indicate that Apollo 16 regolith samples contain a variable but significant amount of mare and potassium, rare-earth elements, and phosphorus (KREEP) components (up to 5% plus mare and up to 20% KREEP¹). The effect of this "contamination" is that, although the Apollo 16 regolith is not completely representative of "typical" highlands overall, it is still compositionally quite close as determined by orbital elemental and lunar highland meteorite studies.<sup>2</sup>

The goal of these prototype simulants is to develop reproducible basic methodologies and procedures to produce simulants and to match the modal mineral and glass content and grain-size distribution of the Apollo 16 regolith samples as closely as possible. The purpose of this Technical Memorandum (TM) is to detail the specifications that were used to develop the lunar highlands type (LHT)-1M pilot and LHT-2M prototype simulants.

#### 2. CHEMICAL COMPOSITION SPECIFICATION

After studying the literature on the Apollo 16 regolith and samples, it was decided to base the specification on an average chemical composition of the Apollo 16 regolith samples rather than any specific surface or core regolith sample. The basis of this decision was that an average composition would be more representative of the highlands regolith as a whole than would any specific sample. A second reason was that, after a literature survey, it was realized that the available data and information available for the Apollo 16 regolith samples was inadequate for the investigation requirements.

Specifically, no studies provided the mineralogical information required to prepare simulants. Highly detailed and excellent particle-type modal analyses exist for Apollo 16 regolith samples but no mineral modal analyses exist. That is, no mineral modal data exists that specifies the amount of minerals (or glass) present in a regolith sample. The published particle-type modes present the amount of lithic, breccia, monomineralic, agglutinate, and glass particles present in the regolith. Thus, if the question is asked of how much olivine is present in a specific sample, the only available data is for the amount of monomineralic olivine. However, olivine may also be present in the lithic, breccia, and glass clasts; therefore, the total amount of olivine present in the sample is undetermined. Also note that a particle-type mode does not provide the total amount of glass present in a sample since glass can also be present in the lithic and breccia particles, and agglutinate particles are only partially composed of glass.

The literature was reviewed for average chemical analyses and one analysis was selected to be the basis for the preparation of the LHT simulants. The one selected (see appendix A) was compiled from the database compilation of the Lunar Sample Curator, was representative when compared to other averages, and had the additional virtue of being the only compilation that included nearly complete averages for the minor and trace elements.

# 3. SPECIFICATIONS FOR LUNAR HIGHLANDS TYPE COMPONENTS

In order to create the LHT simulants, the two basic component materials required are crystalline and glass. The crystalline component is that portion of the simulant that is created by blending natural and synthetic crystalline source materials that contain one or more of the required minerals to simulate the lunar regolith (Stillwater norite, anorthosite, etc.). The glass component is composed of the two subcomponents pseudo-agglutinate and "good" glass. The specifications for each component are presented in sections 3.1 and 3.2, respectively. The feedstocks used were obtained with the cooperation and generous assistance of the Stillwater Mining Company of Nye, MT. The rock components were hand picked from the mine's waste dump. The mill sand used was provided by special arrangement with the company, which provided the wet material in 55-gallon drums.

#### 3.1 Crystalline Component

Table 1 identifies the average composition of Apollo 16 soils taken from NASA, 1982.<sup>3</sup> The data presented is a subset of the source data that presents data for 80 elements. The subset for this TM is the elemental data required to calculate major and minor minerals.

Table 1. Average Apollo 16 "soil" composition from table 7.

SiO <sub>2</sub>	45.09
TiO <sub>2</sub>	0.56
$Al_2O_3$	27.18
FeÖ	5.18
MnO	0.065
MgO	5.84
CaO	15.79
Na <sub>2</sub> O	0.47
$\overline{K_2}$ O	0.11
$P_2\bar{O}_5$	0.12
$Cr_2O_3$	0.107
Š	0.064
Total	100.58

Table 2 presents the calculated normative mineralogy for the Apollo 16 averages of table 1. Table 2 presents two different normative mineral amounts—weight percent (Wt%) and mole percent (cationic) (Mole%). These normative mineral amounts are the target for preparing LHT-1M and -2M. That is, the goal was not matching the chemical composition shown in table 1 but matching the calculated mineral amounts of table 2 (i.e., total plagioclase, orthopyroxene, clinopyroxene, olivine, ilmenite, apatite, and pyrite).

Table 2. Calculated normative mineral content based on the NASA (1982) average from table 1 (using IGPET 2005 and CIPW norm programs by Michael Carr, Terra Softa Inc®).

CIPW Normative Minerals								
	Wt. %	Mole %						
Q (Quartz)	0.00	0.00						
OR (Orthoclase)	0.65	0.64						
AB (Albite)	3.96	4.19						
AN (Anorthite)	71.33	71.20						
Total Plagioclase	75.94	76.03						
AN Content	_	94.44						
DI (Diopside Clinopyroxene)	4.70	4.62						
HY (Hypersthene Orthopyroxene)	10.14	10.23						
Total Pyroxene	14.84	14.85						
OL (Olivine)	7.60	7.92						
IL (Ilmenite)	1.05	0.77						
AP (Apatite)	0.28	0.25						
CM (Chromite)	0.16	0.12						
PR (Pyrite)	0.12	0.06						

The study procedure was to use the chemical composition of available source materials (table 3) to run a reiterative process of calculating bulk chemical compositions from source material blends utilizing a mixing program (an Excel routine) and calculating Cross, Iddings, Pirsson, and Washington (CIPW) normative mineral proportions until the best possible mineralogical match was achieved. This procedure involved simply matching the amounts of the normative minerals calculated for the source materials to the target normative mineral amounts without consideration of mineral composition (i.e., the amount of target plagioclase was matched without consideration of  $AN_{XX}$  content (molecular Ca/(Ca+Na)) since composition of these minerals in the source materials is fixed). The source materials have lower Ca/(Ca+Na) and higher Mg/(Mg+Fe) ratios than lunar regolith, which will be inherent to any mix.

The calculated best match mix then determines the amount of the source materials to be mixed to create the crystalline component of LHT-1M and -2M (i.e., that will be mixed with the glass components). The primary difference between 1M and 2M is that the study only specified and allowed for the minerals (plagioclase, pyroxenes, olivine, and ilmenite); whereas, for 2M, the other minor minerals were also included (i.e., synthetic whitlockite ( $\beta$ -tricalcium phosphate), fluorapatite, and pyrite (troilite substitute)). The proportion of whitlockite to fluorapatite was arbitrarily set at 2:1, since data do not exist for the amount of these minerals in Apollo 16 regolith samples; however, Frondel does indicate that "...in lunar rocks, whitlockite is much more abundant than apatite..."

Table 3. Crystalline source materials used for LHT-1M and -2M.

Lithology	Minerals	Used For
Stillwater Norite	Plagioclase, Orthopyroxene, Clinopyroxene	1M, 2M
Stillwater Anorthosite	Plagioclase, Clinopyroxene	1M, 2M
Stillwater Hartzburgite	Olivine, Orthopyroxene, Chromite	1M
Twin Sisters Dunite	Olivine, Chromite	2M
	Natural Ilmenite (Beach Sand)	1M, 2M
	Synthetic Whitlockite (β-tricalcium phosphate)	2M
	Natural Fluor-apatite	2M
	Natural Pyrite	2M

### 3.2 Glass Component

Section 3.1 specifies the mineral composition for the crystalline component of the LHT prototypes. The crystalline component is mixed with some amount of glassy materials (pseudoagglutinate and other glass) to create the final blend. The glasses in the Apollo 16 regolith are highly variable in composition, ranging from high alumina to mafic (Mg + Fe rich). No average composition for Apollo 16 regolith glasses has been published but it is assumed that their average will be approximately the same as the bulk compositional average. On this basis, it would be reasonable to use the crystalline mix defined by the crystalline specification as the basis for making glasses; however, because of convenience, the mill waste, termed "mill sand," from the mill processing the ore from the Stillwater Mine was used. This decision was predicated on the fact that the waste material has the composition of a feldspathic norite (table 4) and whose normative mineralogy is similar to that of the crystalline component (table 5).

Table 4. Chemical composition of Stillwater mill waste (average of two analyses, in Wt%). The only sulfur (S) value on hand is <500 ppm S, so S is not included in the analysis.

Oxide	Wt. %
SiO <sub>2</sub>	46.60
TiO <sub>2</sub>	0.115
$Al_2\bar{O}_3$	21.55
Fe <sub>2</sub> O <sub>3</sub> *	5.65
MnO	0.09
MgO	9.50
CaO	12.60
Na <sub>2</sub> O	0.965
$K_2\bar{O}$	0.12
$P_{2}^{-}O_{5}$	0.07
LŌI**	2.74
$Cr_2O_3$	0.12
Total	100.12

#### Notes:

 $<sup>^{*}</sup>$  All Fe as Fe<sub>2</sub>O<sub>3</sub>.

<sup>\*\*</sup> LOI, lost on ignition volatiles.

Table 5. Calculated normative mineral composition of the Stillwater mill sand using data of table 3. Oxides were normalized to 100% and an estimate of FeO:Fe<sub>2</sub>O<sub>3</sub> calculated following the method of Irvine and Baragar.

CIPW Normative Minerals								
	Wt %	Mole%						
Q (Quartz)	0.00	0.00						
OR (Orthoclase)	0.73	0.72						
AB (Albite)	8.43	8.81						
AN (Anorthite)	55.87	55.05						
Total Plagioclase	65.03	64.58						
Calculated AN Content	_	86.20						
DI (Diopside)	6.51	6.46						
HY (Hypersthene)	22.71	23.71						
Total Pyroxene	29.22	30.17245.53						
OL (Olivine)	2.93	3.22						
MT (Magnetite)	2.42	1.72						
IL (Ilmenite)	0.23	0.16						
AP (Apatite)	0.17	0.15						
CM (Chromite)	0.19	0.13						

The study simulant design specified two types of glassy material, pseudo-agglutinate (or agglutinate for short) and simple or "good" glass. Both materials were prepared using a plasma melter at the laboratories of Zybeck Advanced Products®, Boulder, CO.

The goal for producing pseudo-agglutinate was vesiculated cindery glass particles that contain a significant amount (>10%) of included crystalline material. This material was created through a custom partial melting process. The proportion of glass to crystalline material is highly variable due to the process used, and no attempt or consideration was given to trying to constrain the ratio other than it should be dominantly glass. An unplanned but useful byproduct of the agglutinate process was the generation of a small amount of glass beads (spherules) of variable composition that were also included in the simulants. In effect, these beads simulate the glass beads that occur in small amounts in most lunar soils (typically about  $1-2\%^{6,7}$ ).

Complete melting of Stillwater sand without vesicles or inclusions was the method used to produce "good" glass.

#### 4. MIXING SPECIFICATION

The amount of agglutinate in the lunar regolith is basically a function of maturity or length of time the surface material is exposed to micrometeorite impacting, which produces agglutinate.<sup>8</sup> The amount of agglutinate in the regolith varies from approximately 0–65% with an average of about 25–30% for regolith surface samples.<sup>2,9</sup> However, studies of Apollo 16 cores show that maturity in the cores generally decreases downwards.<sup>10,11</sup> It should be noted that maturity is primarily determined by the magnetic resonance value I<sub>S</sub>/FeO and that agglutinate content only approximately correlates.<sup>9</sup> The amount of agglutinate was arbitrarily set somewhat lower than the surface average value to reflect this decrease in maturity in the Apollo 16 core for LHT-1M (table 6). On further reflection that many user applications would likely be simulations involving surface materials, the agglutinate content of LHT-2M was adjusted to reflect a more typical Apollo surface regolith value of 30% (table 6).

Table 6. Specified proportions of crystalline to glassy components for LHT prototype simulants.

	Crystalline (%)	Agglutinate (%)	Good Glass (%)
LHT-1M	80	16	4
LHT-2M	65	30	5

A survey of the literature that contain detailed modal analyses indicates glass particle contents (other than agglutinate) of approximately 1-5%, not including the glass present in the breccia and lithic particles; thus, the good-glass target was set at the high end of the range (table 6).

Note that the total glass content of the LHT simulants will be somewhat less than the total of agglutinate plus glass since the agglutinate subcomponent is vesicular and contains crystalline inclusions and, thus, is not all glass.

#### 5. PARTICLE SIZE SPECIFICATION

It was determined by agreement that LHT-1M and -2M would not contain particles greater than 1 mm and thus would consist of grains from ≥1 mm to dust size. The primary guideline for particle size distribution is Butler et al., <sup>12</sup> who published detailed data on 19 Apollo 16 surface soil samples for 16 size fractions (fig. 1). The grain size distribution envelope established for the lunar regolith by Carrier for the <1-mm soil fraction (fig. 2) is also used. <sup>13</sup>

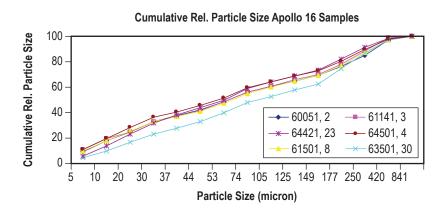


Figure 1. Cumulative grain size distribution for six Apollo 16 regolith samples.

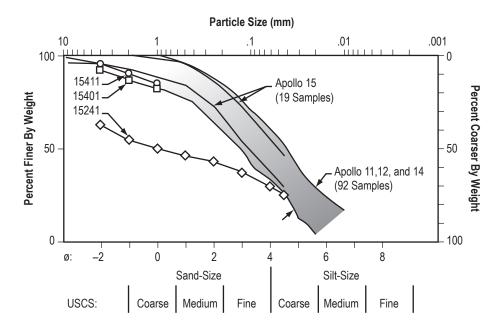


Figure 2. Figure 2 of Carrier (1973) showing regolith grain size distribution envelope based on Carrier's compilation of grain size information for Apollo 11–15 regolith samples.

# **APPENDIX**

Data from (NASA, 1982) is presented in table 7. Major elements (>0.1%) are reported first as both the usual oxide notation and elements. Data is compiled from the Data Base Compilation of the Lunar Sample Curator, NASA Johnson Space Center, Houston, TX.

Table 7. Compilation of average composition of lunar soils for 80 elements.

Mare											
	High Ti Low Ti			Low Ti		High	land	Basin Ejecta			
	A-11	A-17	A-12	A-15	L-16	A-16	L-20	A-14	A-15	A-17	
Al <sub>2</sub> O <sub>3</sub> , Wt. %	13.78	10.97	13.71	10.32	15.51	27.18	23.07	17.41	17.54	20.6	
CaO, Wt. %	12.12	10.62	10.55	9.74	12.07	15.79	14.07	10.79	11.57	12.86	
Cr <sub>2</sub> O <sub>3</sub> , Wt. %	0.3	0.46	0.35	0.53	0.29	0.107	0.15	0.22	0.28	0.26	
FeO, Wt. %	15.76	17.53	15.41	19.75	16.41	5.18	7.35	10.36	11.58	8.59	
K <sub>2</sub> O, Wt. %	0.15	0.076	0.27	0.1	0.1	0.11	0.08	0.58	0.17	0.16	
MgO, Wt. %	8.17	9.62	9.91	11.29	8.79	5.84	9.26	9.47	10.41	10.29	
MnO, Wt. %	0.21	0.24	0.22	0.25	0.21	0.065	0.11	0.14	0.16	0.11	
Na <sub>2</sub> O, Wt. %	0.44	0.35	0.48	0.31	0.36	0.47	0.35	0.7	0.42	0.41	
P <sub>2</sub> O <sub>5</sub> , Wt. %	0.12	0.07	0.31	0.11	0.14	0.12	0.11	0.5	0.16	0.14	
SiO <sub>2</sub> , Wt. %	42.17	39.87	46.17	46.2	43.96	45.09	44.95	48.08	46.59	45.08	
TiO <sub>2</sub> , Wt. %	7.67	9.42	3.07	2.16	3.53	0.56	0.49	1.7	1.32	1.62	
AI, Wt. %	7.29	5.8	7.25	5 .46	8.31	14.38	12.2	9.21	9.28	10.9	
Ca, Wt. %	8.66	7.59	7.54	6.96	8.63	11.29	10.06	7.71	6.27	9.19	
Cr, Wt. %	0.21	0.31	0.24	0.36	0.2	0.07	0.1	0.15	0.19	0.18	
Fe, Wt. %	12.25	13.63	11.98	15.35	12.76	4.03	5.71	10.36	9	6.68	
K, Wt. %	0.12	0.063	0.22	0.08	0.08	0.09	0.066	0.46	0.14	0.13	
Mg, Wt. %	4.93	5.8	5.98	6.81	5.3	3.52	5.59	5.71	6.28	6.21	
Mn, Wt. %	0.16	0.19	0.17	0.19	0.16	0.05	0.085	0.11	0.12	0.085	
Na, Wt. %	0.33	0.26	0.36	0.23	0.27	0.35	0.26	0.52	0.31	0.3	
O, Wt. %	41.6	39.7	42.3	41.3	41.6	44.6	44.6	43.8	43.8	42.2	
P, Wt. %	0.05	0.03	0.14	0.05	0.06	0.05	0.05	0.22	0.07	0.06	
S, Wt. %	0.12	0.13	0.1	0.063	0.21	0.064	0.08	0.088	0.08	0.06	
Si, Wt. %	19.84	18.63	21.57	21.28	20.54	21.07	21	22.46	21.77	21.06	
Ti, Wt. %	4.6	5.65	1.84	1.29	2.11	0.34	0.29	1.02	0.79	0.97	
Ag, ppb	9	9.8	62	50	95	9.6	16.2	17.5	56	6.5	
Ar, ppm	1	1.2	0.3	0.7	_	1.2	_	1	_	-	
As, ppm	0.32	_	0.082	0.01	0.41	0.14	0.28	0.066	_	-	
Au, ppb	3.7	2.5	2.2	2.11	2.5	8.47	4.93	6.7	3.3	4.9	
B, ppm	3.5	2	9.3	_	4.3	5.9	39	19	_	-	
Ba, ppm	140	85.7	413	122	215	127.3	89.6	767.5	279	190	
Be, ppm	2	_	5	1.31	2.2	1.2	_	5.5	2.8	-	
Bi, ppb	1.5	7.7	1.5	0.36	4.9	1.8	2.7	1.7	0.17	-	
Br, ppm	0.239	0.093	0.165	_	0.21	0.217	0.13	0.41	0.06	-	
C, ppm	135	82	104	95	_	106.5	_	130	125	155	
Cd, ppm	0.045	0.032	0.046	0.062	0.8	0.097	0.048	0.181	0.042	0.04	

Table 7. Compilation of average composition of lunar soils for 80 elements (Continued).

Mare								1		
	High Ti Low Ti			Hiah	land	F	Basin Eject	ta		
	A-11	A-17	A-12	A-15	L-16	A-16	L-20	A-14	A-15	A-17
Ce, ppm	50	25.3	104	31.4	33.4	30.3	20.5	185	54	46
CI, ppm	30.2	5.7	31	7.6	53.5	20.9	13	44	5.9	_
Co, ppm	32	35	43	54.4	37	25.3	40.5	35.8	42	33
Cs, ppm	0.18	0.3	0.3	0.23	0.95	0.11	0.11	0.63	0.19	0.18
Cu, ppm	11.5	11	10.3	8.2	31	8.26	19	11.1	7.9	6.4
Dy, ppm	20.2	12.2	24.6	8.6	10.9	6.8	5	39	13.6	11
Er, ppm	11.5	7.9	15.35	5.13	6.3	4.39	2.5	23.5	7.86	6.5
Eu, ppm	2	1.66	1.9	1.01	2.3	1.23	0.98	2.64	1.3	1.35
F, ppm	278	_	132	45	242	72	37	219	60	_
Ga, ppm	4.3	7.5	4.3	4.43	4.4	4.5	3.7	6.8	3.6	4.7
Gd, ppm	16.3	11.4	25.7	8.1	9.8	6.7	3.06	34.8	11.74	10.07
Ge, ppm	1	0.198	0.32	0.17	1.44	0.76	0.46	0.7	0.42	_
H, ppm	51	59.6	45	63.6	_	56	_	79.6	52	98
He, ppm	60	36	10	8	_	6	_	8	_	_
Hf, ppm	8.9	7.3	12.7	5.2	4.75	3.9	2.9	22.2	7.6	5.5
Hg, ppm	0.015	_	0.023	_	_	0.004	_		_	_
Ho, ppm	5.4	_	5.3	1.7	2.5	1.5	0.88	7.8	3.3	_
I, ppb	_	2	_	_	_	5.6	12	_	35	_
In, ppb	_	2.4	90	3.4	35.6	31	19	89	7.6	3.4
Ir, ppb	7.8	5.4	5.6	3.1	9.7	12.4	9.5	12.4	8.3	8.8
La, ppm	17.3	7.32	38.8	11.3	11.5	11.7	7.6	69.4	24	16.9
Li, ppm	16.5	9.77	19.5	9.09	9.7	7.4	5.7	29.8	10.8	11.7
Lu, ppm	1.6	1.03	1.93	0.72	0.84	0.59	0.4	3.1	0.98	0.88
Mo, ppm	0.7	_	0.34	_	_	0.34	_	_	_	_
N, ppm	119	60	84	80	134	89	107	92	190	81
Nb, ppm	15.8	19.1	34	13	15.9	12.8	12	56	16	18
Nd, ppm	42.6	23	75.6	23	26.9	19.3	10.8	105	35	27.6
Ne, ppm	5	2	2	2	_	1	_	2	_	_
Ni, ppm	206	131	189	146	174	345	208	321	282	286
Os, ppb	14	_	6	1.79	30	22	_	_	_	_
Pb, ppm	2.9	0.8	4.8	1.033	6	2.58	1.15	10.02	2.5	1.922
Pd, ppb	21	_	9.7	6.2	_	24	_	50	_	_
Pr, ppm	7.7	_	10.1	3.8	_	4.97	4	23	_	_
Rb, ppm	3	1.2	7.28	2.7	1.85	2.48	1.65	15.25	5	4.21
Re, ppb	5.26	0.47	0.34	0.39	0.36	0.82	3.19	1.15	_	_
Rh, ppm	0.1	_	0.4	_	0.077	_	_	_	_	_
Ru, ppm	0.6	_	0.047	_	0.046	0.01	_	_	_	_
Sb, ppb	4.1	25.4	47	30	3.8	9.7	5.7	3.4	_	26
Sc, ppm	62.8	65	39.2	37.1	39.9	8.9	17	21.9	22	18
Se, ppm	0.39	0.27	0.3	0.18	0.39	0.24	0.3	0.031	_	0.23
Sm, ppm	11.7	8	20.3	5.85	8.8	5.38	3.39	30.9	9.6	8.1
Sn, ppm	0.7	_	0.3	_	1.7	0.22	0.8	_	_	_
Sr, ppm	193	166	138.9	104.2	234	168	140.8	183.8	152	150
Ta, ppm	1.5	_	1.58	0.55	1.4	0.5	0.5	4.1	1.05	0.87
Tb, ppm	3.3	2.63	4.07	1.4	1.5	1.07	0.8	6.4	4.2	1.72
Te, ppm	0.07	0.01	0.05	_	0.088	0.023	0.051	0.031	_	_
Th, ppm	2.24	0.82	6.63	1.76	1.07	1.87	1.44	13.5	4.15	3.01
TI, ppb	2.1	1.4	2	0.94	1.6	7.7	6.2	22	_	2.4

Table 7. Compilation of average composition of lunar soils for 80 elements (Continued).

		Mare								
	High Ti Low Ti			High	land	E	Basin Eject	a		
	A-11	A-17	A-12 A-15 L-16			A-16	L-20	A-14	A-15	A-17
Tm, ppm	1.5	_	2.02	_	0.73	0.67	0.41	3.9	_	_
U, ppm	1.37	0.26	1.61	0.483	0.3	0.52	0.45	3.48	0.99	0.9
V, ppm	66	128	110	191	73.5	25.5	38	49	84	52
W, ppm	0.24	0.14	0.74	0.31	_	0.31	_	1.9	_	0.52
Y, ppm	107	74	145	47	48	39.3	49	242	73	64
Yb, ppm	10.6	7.48	13.7	4.53	5.59	3.86	2.4	22.7	7.3	6.15
Zn, ppm	23	49	6.3	12.8	25	24	34.1	28	14.5	20
Zr, ppm	331	236	503	175	308	163.8	192	842	278	262

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