THE LUNAR SAMPLE COLLECTION

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PREFACE

The Moon has been directly sampled in nine separate places. Additional samples have been received indirectly from other sites via exotic rocks found at the Apollo sites and via three meteorites from the Moon. Lunar samples consist of basalts from the mare regions, breccias from the ejecta from the giant impact craters and anorthositic materials from the highlands. Numerous soil samples and core tubes provide the opportunity to study the lunar regolith. Lunar samples consist mostly of calcic plagioclase, olivine, orthopyroxene, ilmenite and glass. Only three new minerals were found. The collection of samples from the Apollo missions are carefully curated at the Johnson Space Center in Houston, Texas. They are stored in nitrogen cabinets and cut with a band saw as needed. Much of the collection is a working collection studied by scientists worldwide. Some of the samples are used for educational purposes.

INTRODUCTION

Twelve men walked on the surface of the moon and carefully collected 2,196 documented samples of soils and rocks from the lunar regolith during 80 hours of exploration (ref 4). After careful preliminary examination, a large number of splits of these samples were distributed to scientists for detailed mineralogical, chemical and physical analysis and a few samples have also been loaned for public displays in museums and presidential libraries. Most of the lunar sample collection is still stored in an ultra- clean laboratory at the Johnson Space Center (JSC) in Houston, Texas.

The lunar samples were collected by the astronauts at great personal risk. All together, twenty-four astronauts traveled to the moon during nine trips (three astronauts went twice). The explosion of the fuel cell during the Apollo 13 mission emphasized the dangers involved in space travel. There was also the added danger of radiation from potentially fatal solar flares. Today, watching the films of the astronauts working on the lunar surface, it is hard to realize that they were working in a complete vacuum. Some of the tasks, such as the withdrawal of the Apollo 15 core, were quite difficult. On the last three missions there was the potential need for a long walk back to the Lunar Module in case the rover broke down. For geoscientists, the legacy of Apollo is to try to solve the puzzles presented by the samples that these men diligently collected during this extraordinary adventure.

Samples from the early missions were returned in sealed rock boxes. However, other samples were exposed to the atmosphere of the Lunar Module, Command Module and even (briefly) to the atmosphere of the equatorial Pacific Ocean. Once they reached Houston they were stored in pure nitrogen. Originally the lunar samples were quarantined in the Lunar Receiving Laboratory to make sure that no extraterrestrial life forms were present in the samples. During quarantine the samples were given a preliminary examination, cataloged and the rocks were orientated using artificial lighting to match the shadow patterns on the photographs taken by the astronauts. The results of the preliminary examinations were used to plan the distribution of samples to scientists for more detailed studies that could best be done in their highly specialized laboratories. Neither life forms, nor organic molecules nor water were found in the lunar samples so the quarantine was discontinued.

After quarantine the lunar samples received the most extensive and comprehensive analysis of any geological collection, ever, by an international team of principal investigators chosen by peer review. The results of these studies are reported annually at the Lunar and Planetary Science Conferences and the Proceedings (ref 1) of these conferences are the best way to access the literature. Analysis of the samples showed that there was a very diverse set of lunar rocks indicating early differentiation of the outer portion of the Moon. Plagioclase flotation from a global magma ocean apparently formed the early lunar crust. Trace-element-enriched residual liquids initially crystalized 4400 million years ago. Later, about 3900 million years ago, the giant basins formed during extensive bombardment of the lunar crust. Remelting of the lunar interior produced iron-enriched basalts from about 4000 to 3200 million years ago. Continued meteorite bombardment of the lunar surface formed a thick layer of debris called regolith.

LUNAR MINERALOGY

The mineralogy of lunar samples is rather simple with only a few major minerals (plagioclase, pyroxene, olivine and ilmenite) (ref 7, 17). The rocks formed in a completely dry and very reducing environment. Most of the iron is in a plus two oxidation state with a minor amount as metallic iron. The grain boundaries between minerals are remarkably distinct and there are no alteration products. Glass is present in the mesostasis of the igneous rocks. Minerals that might have been added by meteorites have all been melted or vaporized by impact.

There are a few unique features in lunar rocks (Table 1). Lunar plagioclase is almost pure anorthite (calcium feldspar). Maskelynite (shocked plagioclase) is common. Feldspars with ternary (Ca, Na, K) composition were found in rare lunar felsite particles. Lunar pyroxenes have a wide range of composition and those from the plutonic fragments have interesting exsolution features. New minerals that were found included armalcolite, tranquillityite and pyroxferroite. Quenched, iron-rich and silica-rich immiscible liquids were found in the mesostasis of mare basalts. Rocks that were exposed to the micrometeorite environment have a patina of glass-lined microcraters and glass splashes, large and small. Surface coatings of ZnS were found on some volcanic glasses. Akaganeite (FeOOH) was found on the surface of one Apollo 16 breccia.

Lunar minerals do not react appreciably with the Earth's atmosphere. The fine-grain soil gains weight only slowly on an analytical balance and the polished thin sections do not tarnish. The only identifiable problem is slow oxidation of iron grains. However, the classic problem of oxidation of iron with the catalytic help of lawrencite does not seem to be the problem that it is with some meteorites.

BASIC DESCRIPTION

The Apollo Collection is made up of 382 kilograms of rocks and soils (Table 2). In addition to the Apollo Collection, three cores were returned by the USSR with unmanned spacecraft and three meteorites have been identified to have a lunar origin. Each mission returned samples unique to that site as well as exotic samples from other places on the Moon. There are about twice as many breccias as basalts and there are numerous samples of regolith material. Almost all the samples have been described briefly and many have been studied in great detail.

BASALTS

An extensive collection of very fresh, yet very old, basalt samples were returned from the mare surfaces of the moon. There are 134 samples of basalt greater than 40 grams, 42 greater than 500 grams, 24 greater than 1 kilogram, 11 greater than 2 kilograms and the largest (15555) is 9.6 kilograms. They have a wide variety of textures ranging from variolitic to subophitic to equilgranular. Most are fine grained with an average grain size about 0.5 mm. but some have phenocrysts over 1 cm. Many have a high percentage of opaque minerals and some have metallic iron grains. Some are very vesicular with interconnecting vugs and vesicles, but the composition of the gas phase has never been determined. All lunar samples are very reduced with iron in the plus two oxidation state. These mare basalt samples represent pristine (uncontaminated by meteorite) volcanic liquids, presumably from deep in the lunar interior (ref 2). Mare basalts are very iron-rich ($FeO = 20\%$) compared with most terrestrial basalts. Some are also very titanium-rich ($TiO_2 = 13\%$), and some are very magnesium-rich ($MgO = 20\%$). Mare basalts range in age from 3200 to 4000 million years. Clasts of mare basalt are also found in some lunar breccias.

Another variety of lunar basalt (termed KREEP basalt because of its high trace-element composition) was found in abundance at the Apollo 14 and 15 sites (ref 10). The major element composition of KREEP basalt indicates that plagioclase was in the source region. However, only small fragments of this variety of lunar basalt have been found to be free of meteoritic contamination. Measured ages of pristine KREEP basalts (15382, 15366) were about 3900 million years.

BRECCIAS

Most lunar breccias are the lithified aggregates of clastic debris and melt generated by meteoritic bombardment in the ancient lunar highlands about 3900 - 4000 million years ago. There are 59 lunar breccias larger than 500 grams, 39 greater than 1 kilogram and 19 greater than 2 kilograms. Many of the breccia samples are ejecta from the giant basin-forming events (ref 18). Others are interpreted as melt sheets from the fallback of ejecta into large lunar craters. Some have a fragmental matrix made up of individual mineral fragments while others have a crystalline matrix from slow cooling of initially molten matrix. A few are soil breccias containing glass beads and a component of the solar wind. Most breccias are polymict and contain a wide variety of clasts. However, most clasts are themselves breccias. The determination of trace amounts of gold and iridium is a crucial measurement in the study of lunar breccias and their clasts because these elements indicate the amount of admixed meteorite component. Breccia clasts without gold or iridium are termed "pristine" and represent pieces of the original lunar crust before meteorite bombardment. Using trace siderophile and volatile elements some scientists have even assigned breccias to specific lunar craters (ref 8).

PLUTONIC FRAGMENTS

An early discovery of lunar sample analysis was that the lunar highlands contained an abundance of anorthositic material without a significant quantity of an equivalent mafic complement. This has been interpreted to mean that the anorthositic crust of the moon is relatively thick, otherwise the large basins would have dug up more mafic samples. Table 3 is a list of the "pristine plutonic fragments of the original lunar crust". Only one fragment of dunite (72415) was found. Both this sample and a troctolite (76535) were found to be very old approximately 4400 million years. Lunar anorthosites should also be quite old but they have proven to be difficult to date. The anorthosites have relatively high Fe/Mg ratios and are termed ferroan anorthosites (ref 15). Only anorthositic norite 15455 has been measured to be as old as 4400 million years. A second suite of plutonic rocks (termed Mg-norites) has been recognized (ref 9), but it is not thought to be directly related to the ferroan anorthosite suite because of the different trend in Fe/Mg ratios. Samples of this suite (78235 and 77215) have been measured to be roughly 4350 millions years old.

Late-stage differentiates of layered igneous intrusions or of a large scale magma ocean include: granite 14303, sodic ferrogabbro 14306 and quartz monzodiorite 15405. Ages of zircons in these granitic fragments are as old as 4350 millions years. Other clasts of lunar felsite have ages as young as 3900 million years.

GLASS

Glass is an important component in lunar samples and has been studied by many researchers. Glass occurs as mesostasis in basalts, as beads and agglutinates in soil and as splash on rocks. Agglutinates are fragment-laden, vesicular glasses that are made from solar-wind-enriched lunar soil by micrometeorite bombardment. In addition, there are also volcanic glasses presumably made by ancient fire fountains on the moon. Suspected volcanic glasses include the orange glass soil (74220), clods of green glass (15426) and numerous individual beads in other soils (ref 5). Some investigators have measured the compositions of hundreds of individual glass fragments and interpreted groupings in glass compositions to represent "rock types" but these compositions may be mixtures (ref 11).

SOILS

The lunar regolith is the interface of the lunar surface with the harsh space environment (ref 13). It is a mixture of a variety of rocks and soils derived by meteorite bombardment of the lunar surface. The lunar soils that have been exposed for a long time have a large amount of fine material (half is less than 50 microns) and a high percentage of agglutinate glass. These soils are termed mature (ref 12). However, the 80 kilograms of soil samples also contain over 1 million "coarse-fine" particles (1-10 mm) which are large enough for scientific studies.

Since some visible rays from large lunar impact craters extend half-way around the moon, everything on the moon should be represented in the soil samples. However, to a first approximation, the compositions of lunar soils can be successfully matched with mixtures of known lunar rock types (ref 16). Only one or two percent of exotic component can be accommodated. There is also about a two percent meteorite component in mature lunar soils. Immature soils were collected from trenches, from under rocks, where they were shielded, and from fillets around boulders. Altogether, a total of 167 carefully documented soil samples were collected. There are 35 soils over 500 grams, 15 over 1 kilogram, 4 over 2 kilograms and the largest (14163) weighs 7.8 kilograms. A third of each soil remains unsieved to avoid contamination and to preserve delicate features.

CORES

Twenty-one drive tubes were hammered into the regolith and three long drill strings were taken from the later missions (ref 6, 13). Some of the drive tubes were 4 centimeters in diameter but the drill strings were only 2 centimeters diameter. Eighty percent of these cores have now been carefully dissected and studied. Twenty percent are stored for future dissection. Aliquots from different depths are stored in individual containers and contiguous thin sections have been prepared for the entire length. The total length of the cores is 15 meters and the total weight is 20 kilograms. There are 53 separate segments of which only 11 remain unopened. The longest drill (from Apollo 17) was 2.86 meters long. The cores contain an interesting record of stratification that is controlled primarily by small craters in the local regolith.

CURATORIAL LABORATORY

Three different buildings have housed the lunar samples since they were returned to Houston. They were initially quarantined and cataloged in the Lunar Receiving Laboratory. From 1972-1979, they were processed in clean labs in Building 31. Finally, in 1979, a permanent Lunar Curatorial Facility was built to store and prepare lunar samples for allocation to scientists.

The present Curatorial Laboratory includes a high- efficiency, air filtration system to remove dust and other particles from the laboratory air so that it contains less than 1000 particles (less than 0.5 micron) per cubic foot. A positive air pressure is maintained with respect to the outside and the floor plan restricts access to vaults so that the areas which have the most traffic are downstream from the area where the samples are kept. All the building materials that are interior to the building are made of substances that do not shed particles. Overall cleanliness in the laboratory is important because the cabinets occasionally have to be opened for cleaning and regloving and any laboratory dust might carry contaminants into the cabinets.

Samples are stored and examined only in nitrogen filled glove boxes. The nitrogen gas (produced by boiloff of liquid nitrogen) is ultra-pure with less than 20 ppm Ar, 10 ppm oxygen and 10 ppm water. This relatively inert atmosphere prevents chemical changes in the rocks such as rust forming on iron grains. Only three materials are allowed to touch the samples: aluminum, teflon and stainless-steel. Teflon overgloves or stainless-steel tongs are used when handling the rocks so that the rubber gloves do not come in contact with them. Lead from automobile fumes and gold from jewelry are considered two of the worst potential contaminants. The tools used in the cabinets are cleaned with acid and rinsed in freon to avoid any lead contamination. A black-light (ultra violet) inspection is used to search for any organic residue on cabinets or tools.

For purposes of avoiding contamination, lunar samples are probably their own best containers so they are not cut up unnecessarily. For storage, the samples are sealed in multiple teflon bags. Although these bags protect the samples from particulate contamination, they are slowly permeable to gases. Consequently, for long-term storage, the bagged samples are also put into stainless-steel sample containers with bolt-on tops. An aluminum gasket between knife edges on the top and the bottom makes a gas-tight seal. These sample containers are stored in nitrogen cabinets in a bank-like vault with extra-thick, steel-reinforced concrete walls.

When a sample is needed for study, the sample container is taken from the vault and placed in an airlock entrance to the processing cabinet. The airlock is flushed with nitrogen until the sample can be loaded into the processing cabinet. A protocol for removing various layers of teflon bags is used to avoid carrying any particles into the processing cabinet. Cabinets are cleaned with liquid freon to remove all the sample dust between processing. Strict cleaning procedures are also used for all tools and equipment that are used in processing and this equipment is also given the multiple bag protocol treatment. Only one parent sample is worked on at a time in a cabinet to avoid cross contamination of one sample with another. Separate processing cabinets are used for each Apollo mission.

As each rock is processed, it is carefully weighed, photographed, and described. All photographs contain an orientation cube which relates the orientation of the subsample to that of the parent and to the original lunar surface orientation. This is important to the solar flare, cosmic ray and micrometeorite exposure studies. The extensive photographic documentation also tracks the splits of the various lithologies and clasts. Samples are dusted using a stream of nitrogen gas. Maps are made of the saw cuts and surfaces of the complex breccias using binocular microscopes. However, proper petrographic description under these conditions is difficult and the samples need to be redescribed by later studies outside the cabinet when the subsamples reach the individual scientists.

Large lunar samples are cut apart with a diamond-edge bandsaw inside a nitrogen cabinet. No cooling liquid is used so the sawed surfaces of some rocks probably get quite hot during this process. A metal smear can be seen on the saw surfaces of some of the hardest rocks. Slabs of the large rocks and the smaller samples are broken up with a stainless steel chisel. Both sawing and chipping undoubtedly add metallic contamination. Upon completion of a subdividing operation, the subsamples are reassembled into their original positions and photographed as a group for documentation. These photographs are used to construct three-dimensional diagrams and models to help scientists understand the exact position of their subsample with respect to that of the subsamples studied by the other investigators. Sawing done in orthogonal directions is superior to breaking the sample because clasts can be matched up across saw cuts and exact depths of subsamples can be determined.

Lunar core tubes were first x-rayed and then either cut out of their metal liner with a milling machine or they were extruded using a special device. They were then dissected layer by layer. Large particles were documented by photography and each distinct layer was separated and stored in an individual container. About one-third of the soil was left in the side of the tube and several peels with sticky methacrylate film were taken of this residual core to remove the layer damaged by dissection and to preserve some particles in their original orientation. Finally, epoxy (diluted with ether) was used to impregnate the remainder and polished, petrographic thin-sections were made along the entire length.

About 9,000 doubly-polished thin-sections have been prepared of lunar samples. After vacuum impregnation with epoxy, the sample is cut with a diamond blade using alcohol as a coolent, ground flat with SiC and polished with diamond. The polished sample is epoxyed to a glass slide, cut off, ground to 100 microns thickness and polished by hand to 30 microns using 1 micron diamond paste on bond paper. Contamination with terrestrial lead is carefully avoided. Ethyl alcohol is used instead of water to avoid formation of hydrous phases.

The samples that have been returned from scientists or displays are kept separate from the samples that have never been out of curatorial custody. In case of a fire or hurricane the lunar sample vaults are automatically isolated in "dead mode" so that no air, smoke or water can penetrate. A portion of the collection (about 50 kilograms) is kept in dead storage at Brooks AFB in San Antonio for extra safety.

SCIENTIFIC SUPPORT

The lunar sample collection is a working collection with about forty active investigative groups. The original set of 2,196 samples has now been subdivided into more than 84,000 subsamples. Each sample has a "data pack" containing complete documentation of the subdivision done in the Lunar Curatorial Facility. All of the samples and their weights are tracked by computer and an annual inventory is conducted of every subsample. After scientific study, any remaining sample must be returned to JSC along with a complete sample history so that returned samples can be used by other experimenters whenever possible. Samples that are dissolved or destroyed must be carefully documented.

An advisory team of scientists helps the Lunar Sample Curator make recommendations on requests from scientists who wish to study lunar samples. These recommendations are forwarded to NASA Headquarters for final approval. There has been a free and open world-wide distribution of samples to qualified scientists. Since the samples are most often studied by specialists, a preferred mode of operation for the study of complex samples is by groups of scientists, called consortia, who work together under a leader who coordinates their studies. This coordination helps avoid mixing chemical data, age data and petographic analysis from the multiple lithologies that are found in most lunar breccias.

Much of the collection has been cataloged more than once in mission and topical catalogs. In 1985 there is still an active effort to update the catalogs because new lithologies are still being discovered as the breccias are sawed to create new surfaces. Much of the coarse-fine collection, including the particles from the cores, remains to be cataloged.

Some of the important discoveries that have been made during the study of the lunar samples included evidence for an ancient anorthositic crust and an early moon-wide "magma ocean", a major interval of bombardment at about 4000 million years ago, and a second melting at depth to produce basaltic magma from 4000 to 3200 million years ago. It was found that the determination of trace amounts of volatile elements in lunar rocks was an excellent indicator of meteoritic contamination. This technique has since been applied to the study of terrestrial craters and ash layers. On the other hand, the expectation that the history of the particulate radiation from the sun would be recorded at various depths in the lunar core tubes was found to be too difficult in practice because the regolith is produced by stocastic cratering events instead of by uniform deposition. Scientific studies that are still going on in 1985 include: trace-element partioning between mineral phases and melt, regolith formation processes, analysis of volcanic glasses, dating zircons and granite clasts, and modeling Zr/Hf fractionation. The discovery of lunar meteorites has provided the promise of continued new discoveries.

New laboratory techniques that were developed during the study of lunar samples include: Nd/Sm and Lu/Hf age dating, U/Pb ion microprobe age dating, Is/FeO magnetic analysis, Ar 39/40 plateau age dating and precise, low-level, neutron activation analysis for Au and Ir and rare earth elements. These new techniques have had a significant impact on geochemical studies of many geological collections.

EDUCATIONAL PROGRAMS

NASA sponsors three public programs involving the loan of lunar samples for educational purposes. A traveling display program features samples that range in size from 70-160 grams encapsulated in clear acrylic pyramids. In addition, there are 44 permanent displays set up in museums (Table 4) with lunar samples either mounted in nitrogen filled cases or encapsulated in clear acrylic mounts. Another program, designed to be used as a science teaching aid in secondary schools, features six small samples in a clear plastic disk accompanied by written descriptions of each sample, a film, a sound and film-strip presentation, a teacher workbook and other printed material. A third program, limited to university level petrology classes, involves an educational package of petrographic thin sections of lunar rocks and soils. These thin sections are uncovered and require the use of reflecting light microscopes. A detailed, descriptive booklet accompanies that provides an excellent introduction to planetary science accompanies these educational thin section sets.

Table 1
Lunar Minerals

MAJOR MINERALS	ROUGH FORMULA
plagioclase - mainly anorthite	$CaAl_2Si_2O_8$
pyroxene	
ortho	$(Mg,Fe)SiO_3$
clino - pigeonite	$(Ca,Mg,Fe)SiO_3$
olivine	$(Mg,Fe)_2SiO_4$
ilmenite	$FeTiO_3$
MINOR PHASES	
armalcolite	$(Mg,Fe)(Ti,Zr)_2O_5$
tranquillityite	$Fe_8(Zr,Y)_2Ti_3Si_3O_{24}$
zirkelite - zirconolite	$(Ca,Fe)(Zr,Y,Ti)_2O_7$
chromite - ulvospinel	$FeCr_2O_4$
iron	Fe,Ni,Co
troilite	FeS
spinel - pleonaste	$MgAl_2O_4$
zircon	$(Zr,Hf)SiO_4$
baddeleyite	ZrO_2
rutile	TiO_2
apatite	$Ca_5(PO_4)(F,Cl)$
whitlockite	$Ca_3(PO_4)_2$
silica	SiO_2
ternary feldspar	$(Ca,Na,K)AlSi_3O_8$
Ba-sanadine	$(K,Ba)AlSi_3O_8$
pyroxferroite	$(Fe,Ca)SiO_3$
symplectite: in 76535, 72415	Cr spinel, 2 pyrox
akaganeite - on surface of 66095	FeOOH
cordierite, clasts in 15445,73263,72435	$Mg_2Al_4Si_5O_{18}$

Table 2

LUNAR SAMPLE INVENTORY

20 educational thin section packages (12 each)
201 lunar educational disks (6 each)
44 permanent and 15 traveling displays
9,000 petrographic thin sections

	WEIGHT	NUMBER	EVA	
BY LOCATION				
Brooks AFB	50 kg.	343		
Pristine Vault	280	20519		
Returned Vault	24	33548		
Principle Investigators	8	12082		
PAO	8	1648		
Gifts	.2	482		
Consumed during anal.	12	15344		
Total	382 kg.	84000		
BY MISSION				
Apollo 11	21.5 kg.	58	2.2hrs.	0.5km.
Apollo 12	34.4	69	7.6	2.0
Apollo 14	42.3	227	9.2	3.4
Apollo 15	77.3	370	18.3	23
Apollo 16	95.7	731	20.1	20.7
Apollo 17	110.5	741	22.0	31.6
Total	382 kg.	2196	80 hrs.	81 km.
BY TYPE				
Soils	80 kg.	167		
Breccias	133	79 over 300 grams		
Basalts	80	134 over 40 grams		
Cores	20	24 holes		
Other	69	(mostly small breccias)		
Total length of cores is 15 meters (52 segments).				
U.S.S.R.				
Luna 16	.101 kg.	35 cm.		
Luna 20	.050	27		
Luna 24	.170	160		
Meteorites				
ALHA81005	.031 kg.			
Y791197	.052			
Y82192	.036			

Table 3
Pristine Plutonic Fragments of the Original Lunar Crust

Sample	Type	Weight	Age (my)	Technique	Special feature
72415	Dunite	55 gr.	4450±100	Rb/Sr	Symplectite
76535	Troctolite	155	4260± 60	Nd/Sm	Symplectite
78235	Norite	400	4340± 40	Nd/Sm,Rb/Sr	
77215	Norite	846	4370± 70	Nd/Sm,Rb/Sr	
73255,27	Norite	clast	4230± 50	Nd/Sm	
72255	Norite	10	4080± 50	Rb/Sr	"civet cat"
76255	Norite	300			
15455	An. Norite	200	4480±120	Rb/Sr	
72435	mafic clast				Cordierite
15445	mafic clast				Cordierite
67435	spinel troct.	2			Mg spinel
15405,57	monzodiorite	3	4350	U/Pb	Zircon
14321,1062	granite clast	2	4100	Rb/Sr	
14303,209	granite clast		4350	U/Pb	Zircon
14306,60	sodic ferrogabbro		4350	U/Pb	Zircon
15362	anorthosite	4			
15415	anorthosite	269			
60025	anorthosite	1836	3850± 20		
67075	anorthosite	219			
67667	feldspathic Lhersolite		4180± 70	Nd/Sm	
78155	Granulite	401	4200	U/Pb	

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