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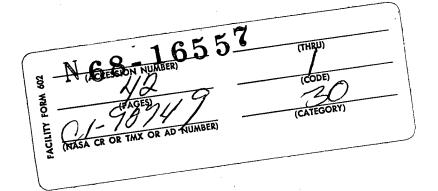
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Sampling of Planetary Surface Solids for Unmanned In Situ Geological and Biological Analysis: Strategy, Principles, and Instrument Requirements

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CALIFORNIA INSTITUTE OF TECHNOLOGY PASADENA, CALIFORNIA

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Abstract

The scientific principles, objectives, and requirements for unmanned sampling of planetary surface rocks for in situ analyses are reviewed. Sampling is a priority objective of early surface reconnaissance planetary missions, and the development of suitable sample-acquisition devices is critical to the progress of planetary exploration. Guidelines and priorities for sampler device development should be based on the requirements of high-priority analytical measurements that have been recommended in several NASA, National Academy of Sciences, and JPL planning documents. The sampling requirements for analytical instruments to be used for measurements of high priority are reviewed and shown to reduce to one set of standard parameters suitable for any combination of instruments in a payload. The sampling requirements for both geological and biological in situ analyses of soils and rocks are discussed and shown to be essentially identical; therefore, engineering development studies of sampling devices for both geoanalysis and bioanalysis experiments should be conducted not separately, but as an integrated study for which the term geosampling is an adequate description.

Sampling of Planetary Surface Solids for Unmanned In Situ Geological and Biological Analysis: Strategy, Principles, and Instrument Requirements

I. Introduction

Acquisition of samples by unmanned spacecraft systems for scientific analysis of solid rock and soil materials on planetary surfaces (the moon, Mars, Venus, and Mercury) is discussed in this report. The purpose herein is to (1) review the scientific principles and practices of unmanned sampling as they have been developed during. and in support of, the engineering study at JPL known as geosampling; (2) review and summarize the sample acquisition requirements of various scientific experiments and the sample requirements of specific analytical instruments; and (3) recommend a general direction and scope that future sampling development should take in light of current thinking regarding the present and anticipated needs of unmanned sampling devices for both biological and geological experiments in lunar and planetary exploration.

Although the desirability of sampling devices is clear, a full evaluation of the sampling requirement has yet to be made. Whether adequate sample acquisition and preparation devices can be developed for, and carried on, lander spacecraft will strongly influence the types and configuration of scientific instruments chosen as payload and may govern the extent to which unmanned scientific analysis of planetary surfaces is done. Consequently, it is important to determine the optimum and minimum sampling requirements for various kinds of analyses and various types of analytical instruments in consideration of the probable geological and biological properties of planetary surface materials, and from this, to establish principles and guidelines for the efficient design and development of sampling devices. From these considerations, coupled with the results of continuing engineering studies of specific sampling devices (which are not considered in this report), a rationale can be set forth by which sampling devices can be chosen for any given mission objective and instrument payload, without intuitive assumptions that sampling devices are, or are not, suitable.

In setting up these requirements and guidelines, it will be necessary to make them flexible enough to meet changing demands as the lunar and planetary program progresses, as knowledge of the sampling environments increases, and as analytical problems receive sharper focus. As the assignment of experiments for spacecraft payloads becomes more clearly defined, the sampling requirements will become clearer. However, many experiments cannot be assigned or even seriously considered unless it can be foreseen that a satisfactory sampling system can be developed to meet their specific sampling requirements. Sampling is essential to meeting the most important and critical early objectives of unmanned lunar and planetary exploration missions. To a large extent, the rapid development and future success of unmanned lunar and planetary surface exploration will depend upon the successful implementation of sampling techniques.

II. Objectives and Principles of Unmanned Planetary Sampling

A. Samples, Sampling, and Samplers

In this report, the term sampling means the collection and preparation of solid rock and soil materials by mechanical devices for *in situ* analysis by analytical instruments where the analysis is conducted as the specific objective of a scientific experiment.

Sampling of planetary solid material is necessary for geological and biological scientific investigations. The two basic objectives of these investigations are:

- (1) To determine the physical, chemical, and mineralogical properties of the surface and subsurface materials.
- (2) To determine whether life of any kind exists or ever existed in, or upon, the surface or subsurface materials.

To meet the geological and biological objectives, analytical measurements must be made on the solid materials of the planet. Measurements can be carried out:

- (1) By remote sensing (in special cases) from the earth or from manned or unmanned orbiting spacecraft.
- (2) By collection and return of samples to earth by manned or unmanned missions for standard laboratory analyses.
- (3) By in situ analysis by manned missions.
- (4) By in situ analysis by unmanned spacecraft.

This report is concerned primarily with the fourth category involving unmanned *in situ* analyses, although some of the discussion applies to other categories as well.

Geosampling is a term that has been used in recent years by space scientists and engineers to describe sampling of rocks and soils for geological analyses. Biosampling is the term similarly applied to sampling of soils for biological analyses. Samples are taken as fractional parts of a unit of material (of either biologic or geologic origin) which is of particular scientific interest. Since an individual analysis of a sample will represent an average for that sample, the sample should therefore truly represent the average properties of that portion of the unit being sampled. The sample may be taken as (1) an example of a homogeneous unit, or (2) representative of some specific portion, or feature, of a heterogeneous unit. Which of these two cases the sample actually represents may be unknown at the outset of sampling unless prior knowledge about the unit sampled is available; multiple sampling (and conjugate analysis) will determine partly the degree of heterogeneity of the unit.

To achieve the experimental objectives of analyzing planetary materials, a selectively obtained and properly prepared sample must (for most experiments) be delivered to an analytical instrument. Most analytical instruments considered for planetary exploration are dependent on a separate accessory device to acquire, prepare, and transport a sample to the instrument. This accessory device, and its various components, is referred to as the sampling system, sampling device, or sampler. The sampler is essential for providing a proper working interface between certain scientific instruments carried by the spacecraft and the indigenous planetary solid material.¹

Sampling requirements, and, therefore, sampler requirements, are based upon the following interdependent parameters:

- (1) Objectives of the spacecraft mission.
- (2) Objectives of the particular experiment for which sampling is required.
- (3) Nature of the analytical measurement that is to be made.
- (4) Operational mode and mechanical character of the analytical instrument that is to make the measurement.
- (5) Probable character of the material to be sampled.

¹In addition to providing rock and soil samples for various analytical instruments, sampling devices can be used to measure certain mechanical properties of the planetary surface. For example, since the sampler must make physical contact with the surface, the retarding forces imposed upon the sampler during contact are indicators of the bearing strength, cohesiveness, porosity, and particle size of the surface material. With suitable sensors attached to the sampler, these retarding forces can be measured and (with proper calibration) can yield a crude but quantitative measure of soil physical properties.

(6) Spacecraft payload weight, power, and mobility.

The interrelationship of these parameters will be covered in the following discussions. At present, it is sufficient to emphasize that, until each of these parameters is specified for a given planetary mission, no satisfactory choice of available sampling systems can be made.

B. Sampling/Analysis Modes: Deployment vs Acquisition

There are two principal modes of analysis that can be used with analytical instruments on unmanned spacecraft, depending on the nature and objectives of the measurement and on the weight capability and power available. These analysis modes define the interface configuration between sample material and analytical instrument.

1. Deployment. In this mode, the analytical instrument is brought to the sample instead of the sample being brought to the instrument. This normally involves positioning of the instrument in the proximity of the sample, or placing a sensor directly in contact with the undisturbed sample material. In this mode, the sample material is the uppermost surface of the planet, directly below, or immediately adjacent to, the instrument.

2. Acquired sample. In this mode, sample material is collected from the planet surface or subsurface by a separate sample-acquisition device and then transported and delivered, either directly or after an intermediate processing step, to the analytical instrument, which is mounted permanently to the spacecraft frame. This mode involves some deployment because the sampling device, or part of it, has to reach down or be positioned onto the planetary surface for sample acquisition.

Both modes of analysis have particular advantages and disadvantages depending on the analytical instrument, the experimental objectives, and the mission and spacecraft constraints. Table 1 presents a summary of some of the more important considerations necessary in selecting the mode of analysis.

The chief variables of any surface analysis experiment are (1) the geometry of the planetary surface in the immediate area examined, (2) the extent to which the examined material is statistically representative of the material unit with respect to the objectives of the experiment, (3) the degree to which local variations in the material can be examined, and (4) the relative difficulty in the operations of emplacing a deployable analytical instrument, or a sample acquisition device, on the planetary surface.

There is a wide range in the effect of these variables on the data obtained by each of the two modes of analysis. In theory, many analytical instruments (especially geological) can be deployed without acquisition or preparation of sample material. However, in practice, such a procedure would be inadequate for some analyses. For example, the alpha scatterer, when deployed, can yield suitable results regardless of whether the planetary surface at the analysis site is loose powder or hard bedrock. However, other instruments (e.g., X-ray diffractometer) can be designed for deployment operation only if the expected planetary surface is made up of loose, compressible powders. Others (e.g., the petrographic microscope and most biological experiments) can only be designed for acquired sample analysis because the rock or soil particles must be dispersed in a fluid medium or otherwise processed before the analysis.

From a reliability standpoint, the deployment mode appears better than the acquired sample mode, since in the latter, failure at any stage in the acquisition and processing of the sample can result in failure of the experiment; this could generally be mitigated, however, by a backup sampler.

However, attaining higher reliability by using the deployment mode may seriously compromise the scientific significance of the experiment, because, for most deployable instruments, only surface samples can be analyzed, and little or no processing of the sample can be performed. If processing is required prior to deployment analysis, then it may be as complex and weight costly as a sample acquisition device.

The final decision as to whether the analytical instruments on a given mission will be configured for deployment or acquired sample analysis modes will be based on tradeoffs between the following parameters:

- (1) Configuration constraints of the particular instrument.
- (2) Availability of flightworthy instruments of a given analysis configuration.
- (3) Availability of a suitable sample acquisition device or devices.
- (4) Mission and experiment objectives.
- (5) Weight and power limitations.

Parameter	Deployment	Sample acquisition
Definition	Complete analysis system or sensor unit placed against planetary surface	Material is collected from the planet surface or subsurface, processed, and presented to the analytical device
Relation to analytical instruments and spacecraft		
Requirements	Deployment mechanism required	Separate sample-acquisition device required
	Flexible and extendible wiring harness required	Sampler system must be able to acquire material from a variety of surface types
		Sample-acceptance system required (i.e., hopper, cups)
		Sample must be placed into cup or other contain
Limitations	Some analytical instruments inherently cannot be adapted to deployment type analysis	If sampler-system capability limited to either bed rock or soil surface, then two separate systems
	Analysis limited to uppermost surface material with most deployable instruments	must be furnished if surface type is not known in advance
	"Sample" generally cannot be processed for opti- mum analysis procedures	Deployment of sample-acquisition device for sub- surface sampling may be hindered by surface obstructions
	Deployment may be prevented by surface obstructions	
	Sensor susceptible to environmental effects (radiations, etc.)	· · ·
Disadvantages	Deployment mechanism and wiring harness may be as heavy and bulky as a separate sampler device Sample (i.e., planet surface) may not be suitably	If only one sample or one sampling site is to be analyzed, extra power and weight required fo sampler may exceed that for deployment
	configured for proper interface with instrument	mechanism The sampler mechanism may have to be deploye
	or sensing unit (if contact is required)	to reach certain sampling sites, thus requiring own deployment mechanism
		Several possible failure modes — acquisition, transport, sample presentation
Advantages	Simplicity, reliability, low power	Instrument design is not constrained by sample shape
	No visual (i.e., TV) control needed Can analyze any surface, regardless of the sur- face's suitability for sample acquisition	Sample can be properly prepared and presente to sensor (i.e., homogenization, grain size selection)
		Instrument firmly mounted to spacecraft frame
		Extra power and weight for sampler may be les than that required for deployment mechanism especially if multiple sites are to be analyzed
		Surface obstructions easier to avoid (with or wit out TV control) than in deployment mode

.

Table 1. Comparison of deployment and acquired sample analysis modes

Table 1 (contd)

Parameter	Deployment	Sample acquisition
Relation to experimental objectives		
Requirements	Proper positioning of instrument or sensor	Sample must not be deleteriously altered by sam- pling operation. (The allowed alterations depend on the nature of the experiment for which sampling is conducted)
Limitations	Some experiments could not be performed (i.e., most biological and several geological such as the petrographic microscope)	Samples are disturbed from natural condition
	Usually only one sample can be analyzed	
	Would require separate scraper for subsurface analyses; scraper may be more complex than a sampler	
Advantages	Some experiments are best performed this way	Subsurface samples can be analyzed
	Instruments can be positioned away from spacecraft	More samples can be analyzed
	Samples are undisturbed prior to analysis	Good standard samples can be supplied for instrument calibration
Disadvantages	Can only analyze uppermost surface material	If sampler device fails, experiment also fails
	Sample may not be properly configured (e.g., too coarse grain) for good analysis	
	With some deployable instruments, no standard or reference sample can be supplied for calibration	

In general, the deployment mode is simpler and possibly more reliable, but is less versatile and will produce less significant analytical data for most experiments than the acquired sample mode. The deployment mode can, however, be employed as a backup mode for some instruments (e.g., alpha scatterer) in case of failure of the primary sample acquisition system.

3. Sample types. The rock materials on planetary surfaces (moon, Mars) are expected to have physical, chemical, and mineralogical properties similar to those known on earth and for meteoritic rocks². Therefore, these materials will consist chemically of silicates, oxides, sulphides, carbonates, and free nickel-iron. The materials may occur as crystalline compounds (minerals) and as amorphous compounds (glass). Texturally, these materials may be solid or particulate, and fine or coarse grained. When sampled, these materials may be in either of two general forms:

- Bedrock. An indigenous part of the solid body of the planet, which originated by processes originating within the planet, has not lost its structure and character by decomposition, and usually underlies a layer of loose soil. Uncovered surface exposures are called outcrops.
- (2) Soil. The mantle of loose or cohesive particulate material that overlies bedrock³ and that formed by chemical and/or mechanical degradation of underlying bedrock, by impact (meteoritic) fragmentation, deposition of volcanic ejecta, and by admixture of atmospheric condensates and precipitates, organic matter, and infalling meteoritic materials.

Living biological material, if any, will be in the soil and not in the bedrock, although fossil biological material may exist in some types of bedrock.

²No detailed arguments will be offered for this assumption, except that (1) a prominent theory of the origin of the solar system states that all solar system objects are condensed from the same basic material, and (2) volcanic processes, known to form each of the above rock compounds on earth, can be mobilized on other planetary bodies, even though the origin of the mobilizing effects may be external (meteoritic) instead of from within the body.

³In some cases, a volcanic bedrock may overlie an older soil layer.

4. Sample source. An analysis, no matter how thoroughly carried out, is no better than the scientific significance and analytical quality of the sample that is selected. Which of the two probable types of planetary rock materials (soil or bedrock) is the most significant to sample and analyze depends primarily on the objectives of the experiment. If the objective of the experiment is to measure chemical or physical parameters which relate to the question of the origin of the planet (Ref. 1), then bedrock is the more significant sample. If, for example, the objective is to determine why certain areas of the planet's surface undergo seasonal color change (Ref. 2), then soil is more significant. Secondarily, and realistically, the choice depends on the type of rock present at the spacecraft landing site. If only one rock type is present, then that type will be significant. If soil and bedrock are present and accessible, then both should be sampled and analyzed separately.

For geological experiments in general, both bedrock and soil samples are important. The bedrock samples would supply data on internal (crustal) planetary properties and processes which may have a bearing on the fundamental problem (see National Academy of Sciences' recommendations, Ref. 3) of the history and origin of the planet and its relationship to other bodies in the solar system. Therefore, bedrock samples have fundamental significance. Soil samples, on the other hand, would supply data on active surface processes which may have little direct bearing on the history and origin of the planet, but which are of exceedingly important practical value for explaining telescopically observed surface phenomena (e.g., seasonal darkening) and for defining the detailed nature of the surface environment for future mission planning.

In general, experimental data obtained on bedrock samples will be easier to interpret than data from soils, since soil may have originated through a diverse series of events and processes. As to the probability of encountering bedrock or soil at a given landing site, uncovered bedrock is probably a rare condition on most planet surfaces. On earth, for example, large amounts of bare bedrock are only exposed in rugged mountainous regions, where, as on other planets, it would be difficult to land a spacecraft. Bedrock specimens in the form of boulders and pebbles are also found out of place in stream beds, alluvial fans, glacial deposits, and surrounding the rims of large meteoritic craters. Samples taken from outof-place boulders and pebbles are valuable, especially if the source of the boulder is known or can be deduced. Even so, bare bedrock and boulder deposits represent less than 1% of the earth's surface and the percentage would be much less if it were not for internal mountainbuilding processes and the surface scouring effects of winds, waters, and glaciers.

It is expected that, on other planets, rock surfaces likewise will be mantled with a layer of particulate soil. Therefore, the type of sample may not be as important as the types of experiments that may be conducted with the materials available on the landing site. Early missions should be prepared to sample material that is most likely to be found. Since there are many arguments for an ubiquitous soil mantle, it would be best to plan primarily, if not exclusively, for this type of surface rock.

As pointed out in Ref. 4, the ability to analyze meaningfully a loose surface aggregate of particles is especially important during the early unmanned phases of planetary exploration when complex bedrock sampleacquisition devices are a major engineering obstacle. For example, the surfaces of Mars and the moon may be covered in many places with particulate debris which is too deep (more than 0.5 m) to be reached by a remotely operated spacecraft drill of feasible proportions. In that event, the nature of the bedrock at more distant locations, and whether the surface particles were moved to the location dominantly by wind, volcanism, meteorite impacts, or by other mechanisms will have to be deduced from analyses of accessible surface soil at that sampling location.

For biological experiments, the most important material to sample and analyze is uncontaminated, unaltered soil at the surface-atmosphere interface and from the immediate subsurface. It can be expected that, as on earth, living organisms and related organic compounds will be associated primarily with loose or cohesive soil particles at, or very near, the topmost surface of the soil layer. Solid bedrock, such as igneous and metamorphic rocks, is not known to contain living organisms, although some forms of bedrock contain remnants (fossils, decomposed organic compounds) of living organisms. Some living organisms may reside on bedrock surfaces, such as lichen (a symbiotic composite of fungus and algae) on earth; however, it is unlikely that such life forms would exist without a corresponding biota in adjacent soil. Natural cracks and crevices in bedrock surfaces may contain accumulated particulate soil in which biological material may reside; thus, in a bedrock terrain, sampling of fracture fillings would be important.

The engineering implications of a bedrock sample requirement are twofold. Firstly, the physical properties of bedrock are, as previously mentioned, considerably different from physical properties of soil. Because of this, more energy is required to obtain a bedrock sample than a soil sample. Early engineering studies (Ref. 5) have shown that the same low-power, low-weight device cannot do both chores equally well; therefore, plans should be made for one rock type or the other, or two samplers should be used: one for bedrock, and one for soil. Secondly, and far more important, the accessibility to bedrock may be poor as compared to accessibility to soil, because it would require either a deep-drilling capability to penetrate a soil overburden, or a roving-vehicle sampler capable of traveling from the landing site and being directed to the nearest suitable bedrock outcrop (located, for example, in nearby mountainous terrain). In either case, bedrock sampling presents a much more formidable engineering task than soil sampling.

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5. Depth to bedrock. The depth to which a sample acquisition device must penetrate a soil layer to reach bedrock must be known or estimated to fix the design and operational mode of a bedrock-sampling device. At present, the depth of soil layers on the moon and planets is unknown, and little data are available for making accurate estimates. However, it is known that lunar soil in the equatorial maria (Surveyors I and III and Luna 9 and 13 landing sites) is greater than 15 cm in depth and probably at least several meters in depth. Large boulders of bedrock-like rock surrounding craters deeper than 5 m suggests that the minimum depth to large amounts of this bedrock-like material in the maria is at least 2 or 3 m. In the lunar highlands and on the steep slopes of large craters, the depth may be less. On the moon, then, the average bedrock-like surface is at least several meters below the uppermost soil surface, and bedrock material in the form of boulders may be found at the uppermost surface and presumably distributed downward throughout the soil layer to some unknown depth.

On Mars and Venus, even less is known about soil depth than on the moon. However, because of possible transportation and deposition of soil material by winds, the soil depth can be expected to range from very shallow or entirely absent to very deep. Therefore, bedrock may be exposed at the uppermost surface at some localities and deeply buried at others.

The depth capability of the sampling device must represent a compromise between the depth desired and feasibility within the normal spacecraft constraints of weight, power, and complexity. In past design studies of lunar bedrock sampling for the X-ray diffraction experiment (Ref. 6), a design depth of 25 cm was chosen. Considerable engineering work on sampling devices (Ref. 7) has shown that a 25-cm depth capability with low-power, low-weight devices is attainable.

Until direct soil depth measurements are made on the moon and the planets, the best estimate of required depth capability for reaching the bedrock-soil interface is of the order of 2 m. However, if surface boulders represent bedrock, and if wind-scoured bedrock occurs at the uppermost planetary surface, then a satisfactory sampling-depth capability on early missions is more on the order of several centimeters.

6. Sample preparation. In rock sample analysis, whether with bedrock or soil samples or for geological or biological experiments, many variables can affect the resulting data. One of these variables is sample preparation. Most analytical methods demand painstaking attention to sample preparation for best or even satisfactory precision, and much of the discrepancy in analytical precision can be traced to the way samples are handled and prepared. Although the actual experimental technique may be fairly straightforward, the sample in many analyses is the most difficult experimental variable to control. (This implies, as previously stated as a basic premise in planetary sampling, that the analysis of rock materials is not simply a matter of obtaining data on random samples.) Therefore, preparation of rock samples for analysis is one of the most critical steps in the analytical procedure of many experiments.

The objective of sample preparation is to provide a proper working interface between the rock material to be analyzed and the instrument sensors which make the analytical measurements. The term preparation here means any and all mechanical or chemical manipulations that the sample is subjected to prior to the actual analysis. There are five basic manipulations or steps in the sample preparation operation: (1) acquisition, (2) transport, (3) processing, (4) configuring, and (5) positioning. All of these manipulations are required when the acquired sample mode of analysis is used, whereas only processing and/or configuring may (but not necessarily) be required when the deployed instrument mode is used. These manipulations may, in practice, be carried out as separate independent operations and with a separate device for each, or, in the other extreme, all five manipulations can be conducted simultaneously and/or sequentially by a single device. In most sampling schemes proposed to date, collection, transport, and a limited amount of processing (e.g., maximum particle-size limitations) are simultaneously or sequentially carried out by the sampling system, and any further processing, configuring, and positioning are done prior to analysis by internal mechanisms within the individual analytical instrument. The five basic manipulations and their definitions are as follows:

- (1) Acquisition. The first step in acquired sample preparation is to dislodge and collect soil or bedrock material from the surface or subsurface of the planet. If the material to be sampled is loose surface soil, acquisition involves only collecting a volume unit of the particulate soil from a given location; if there is a wide grain-size distribution in the soil, some upper particle-size cutoff (usually approximately 1 mm) may be imposed during collection. In some cases, it may be desirable to collect only a single pebble or boulder, which in turn would require further processing. If the material to be sampled is cemented soil or hard bedrock, acquisition involves fragmentation and dislodgement as well as collection of the rock fragments. If subsurface material is to be sampled, the acquisition device must discriminate against overlying material during the acquisition process.
- (2) Transport. The next step in acquired sample preparation is the transport of the acquired material from the point of acquisition to the analytical instrument. This may be a direct transfer with no intermediate steps, or it may involve an intermediate processing step (item (3) below). The mode of transportation and distance will vary with the type of acquisition device employed and with the relative position of the sampling point and analytical instrument; for sampling at the spacecraft landing site, the direction of transportation will be primarily vertically upward, whereas for sampling outside the landing site (as defined by the area disturbed by the spacecraft-landing effects), a relatively large horizontal component of movement may be required.
- (3) Processing. Acquired samples for many analytical experiments must be subjected to a certain amount of processing to ensure further handling and proper interfacing of sample with sensor. For most

experiments, acquired samples must be of a certain total volume and be particulate, with either a maximum particle size or a specific range of particle sizes. If the sampled material is particulate soil, the processing step may consist only of particle sizing and dispensing of the required quantity of sample. If it is bedrock, cemented soil, or a single pebble, fragmentation will also be required. Once the acquired sample is in particulate form, it may require various processing steps such as further comminution (particle-size reduction), homogenization (mixing), splitting into separate and representative aliquots, size and/or density and magnetic fractionation, liquid treatment (incubation, extraction) for bioanalysis, and, in some instances, mixing in of a standard diluent or isotope labels for calibration or other purposes.

Although sample processing normally applies only to the acquired sample mode of analysis, processing may also be applied to planetary surface samples which are to be analyzed by the deployed instrument analysis mode. For some deployed instrument experiments and some planet surface types, it may be desirable or necessary to smooth or compress loose soil, remove large interfering pebbles, sweep clean a debris-littered rock surface, or remove a contaminated surface layer prior to positioning or emplacement of the analytical device.

- (4) Configuring. For some analytical instruments, acquired and processed samples must be properly shaped in bulk for positioning relative to the analytical sensor. The configuration requirement varies somewhat with the analytical instrument to be used (Section III-D). In general, individual samples must be of a specified amount (specified in terms of sample volume), bulk shape, and porosity. The configuration requirement is usually taken care of by the inherent porosity of the sample material and the shape and size of the sample container or receptacle used by each instrument. In some cases, the sample powder must be compressed to provide a smooth packed surface, or dispersed in a foreign matrix (solid or liquid) to provide separation of individual particles.
- (5) Positioning. The proper positioning of the sample in the analytical instrument is the last preparation step prior to analysis of the sample. With those instruments which can analyze only one sample,

this step may occur simultaneously with the configuring step. With instruments capable of multiple sample analyses, each sample is first placed in a container and then the container is moved to the analysis position (and/or the sensor is moved to the sample position).

In addition to the five basic sample preparation functions summarized above, three additional operations may be required in the analyses scheme. These operations may or may not affect the design and operation of the sampling system as a whole. Firstly, provision may be required for handling a standard earth sample which is carried within the analysis system for calibration and comparison purposes. Secondly, if multiple analyses are to be performed, provision may be required for either ejection of a sample from the analysis position, and/or removal and storage of samples for later re-analysis; therefore, a sample removal (and possibly ejection), storage, and retrieval capability must be supplied. Thirdly, if several instruments are to sequentially analyze the same prepared sample, a sample transfer mechanism will be required.

7. Sample preparation constraints. During sample preparation, each step must be conducted in a manner that is not detrimental to the quality of the sample. Factors which can affect the quality of the sample during preparation are discussed in the following subsections.

a. Contamination. Sample contamination consists of (1) cross contamination, resulting from mixing of parts of two or more consecutive samples; and (2) bias contamination, resulting from the addition of material to the sample from the sampler mechanism.

Cross contamination results when the sampling mechanism is not adequately cleaned between preparation of successive samples. Therefore, only the second or following samples will be affected, each containing a portion of all preceding samples. Cross contamination can also occur during sample selection, for example, when the sampling device must be capable of acquiring a bedrock sample from below an overlayer of soil. In this case, contamination of the drilled bedrock sample by material from the overlayer is possible and must be avoided by suitable mechanism design. Cross contamination can result if the same sample cup is used for consecutive samples, or if a continuous-flow sample over a single sensor port is used without adequate means for cleaning the port between samples; this may be particularly important if the sensor sees only that portion of the sample closest to the port, as in the X-ray diffractometer.

Bias contamination results when material from the sampling mechanism becomes mixed with the sample being processed. Examples include (1) earth organisms transported to the planet by the spacecraft system, (2) rocket exhaust materials, (3) metal chips from a drill bit, and (4) metallic grit from the abrasive action of sample particles during sample handling. Bias contamination by organisms, organic compounds, and iron is particularly undesirable for planetary surface experiments because the uncontaminated surface material may contain similar or identical constituents; in such a case, the analytical instrument may be unable to differentiate between sample constituent and contaminant.

b. Fractionation. Fractionation is a change in the relative abundance of chemical, mineral, organic, or particlesize constituents in the sample due to selective removal of part or all of one constituent during sample preparation. The result may be that the sample is no longer representative of the original soil or bedrock material.

The effect of fractionation will vary, depending on the number and type of mineral phases, organisms, and organic compounds that are present in the original rock and, also, upon the kind of fractionation. For example, severe size fractionation of a monophase sample would not change the relative mineral abundance, but might yield excessively fine or coarse particles, either of which might result in degraded analytical data. Density or shape fractionation of a polyphase sample would yield a sample that either is deficient in, or contains an excess of, one or more phases, to yield analytical data that do not quantitatively represent the original rock.

Fractionation will most likely occur during two stages of sample preparation: acquisition and transportation. The effects during each stage may or may not be cumulative.

During acquisition, the sampling device may selectively acquire only a limited size range of particles from the natural particulate soil or from previously drilled or pulverized material. In some cases, such as some biological experiments, selective sizing and retention of only the finer size fraction may be desirable. Because some

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mineral grains are naturally smaller than others and because some, during drilling or crushing, will be reduced in size more than others (due to different hardness, cleavability, etc.), each size fraction of the resulting aggregate may have mineral phase abundances different from the others. Consequently, selective sizing may leave behind a portion, or even all, of one or more constituent mineral phases, severely compromising geological experiments. The mode of acquisition, therefore, must be tailored to the analytical application. In general, however, it is best that all of the drilled or otherwise pulverized material, or a total volume unit of previously comminuted material (e.g., natural soil), is acquired. If the volume unit acquired is too large for the subsequent preparation scheme or for the final sample, then it should be split into parts containing equal phase abundances.

During transportation, motion of a polyphase particle aggregate relative to some confining structure, fluid medium, vibration, or gravity, magnetic or electric fields can quickly fractionate its particles if they have different properties such as size, shape, density, magnetic susceptibility, and dielectric constant. Rock-forming minerals have a wide range of these properties, and polyphase particle aggregates can be severely fractionated prior to analysis by one or all of the above mechanisms if improperly applied. For geological experiments, movement of polyphase particle aggregates, therefore, must be accomplished by transport mechanisms whose operating parameters minimize sample fractionation.

c. Overgrinding. Serious degradation of experimental data may result from overgrinding or overcomminuting of rock or soil fragments during sample preparation. Firstly, for some experiments, it is particularly important not to produce too many fine particles in the sample powder. In standard laboratory X-ray diffractometry, for example, an excess of particles below 1 μ in size produces diffraction-line deterioration and difficulties in interpreting diffraction data; with the miniaturized lunar diffractometer (Ref. 8), it was found that drill-produced specimens of hard bedrock (basalt) give poor diffraction data when samples contain an excess of particles $<20 \ \mu$ (Ref. 9) the optimum drill-produced size range is 20 to 50 μ . For the petrographic microscope experiment (Ref. 4), abundant particles below 50 μ produce clouding of the optical image; the optimum particle-size range is 50 to 300 μ . Secondly, during drilling or pulverization, frictional heating may cause the temperature of the particles to (1) exceed the stability temperature of the mineral phases composing the particle, (2) drive off condensed or absorbed volatile components of the sample, and (3) kill microorganisms attached to the particles or contained within particle aggregates. Therefore, the original nature of the sample material is changed prior to analysis. Thirdly, excessive grinding is likely to increase the contamination introduced into the sample from the grinder.

d. Compaction. High bulk density is desirable in samples for certain geological experiments (e.g., X-ray diffractometer, alpha scatterer). Aggregates of particles of sizes suitable for reliable analysis do not naturally compact under gravity to the greatest possible bulk density. Therefore, some degree of artificial compaction of the sample may be desired during the configuring step in sample preparation. The amount of compaction and, particularly, the method by which it is achieved must be properly chosen. Excessive, or improper compaction, although resulting in favorable increased particle density, can result in unfavorable preferred orientation of nonequidimensional particles and in excessive bending of flexible crystals. Simple compression of the sample powder is the best mode of compaction. A shear motion would tend to smear the particles and to orient nonequant dimensional particles parallel to one another and to the sample surface. Vibratory compaction is considered undesirable because it relies on gravity and tends to fractionate particles by size, shape, and density, yielding inhomogeneous sample aggregates.

III. Geosampling

A. Geoanalysis Principles

A fundamental objective of planetary exploration is the chemical, mineralogical, and textural analysis of geological materials. These materials may be divided into:

- (1) Rocks and individual minerals (solid bedrock, unconsolidated sediments, and soils).
- (2) Natural liquids (free standing, frozen, and pore).
- (3) Gases (atmospheric, absorbed, chemically bound, radiogenic, and occluded).

The most abundant and accessible planetary materials are surface rocks. Surface rocks are somewhat loosely defined here as rocks exposed at the immediate surface and readily accessible to a limited depth (several tens of meters) below the uppermost planetary surface. For purposes of this discussion it will be assumed that early unmanned planetary missions will be of a general reconnaissance nature, conducted for the purpose of characterizing surface rocks and delineating broad geologic problems for subsequent, more thorough study by manned or unmanned missions (as outlined for the best role of unmanned landers in Ref. 10, and as already performed by *Surveyor*).

Generally, rocks form distinct units of material which have characteristic chemical and physical properties and which are related directly or indirectly to adjacent rock units having similar or distinctly different properties. The most typical example is the unit of soil which overlies a bedrock unit. Since the soil may or may not be derived from the underlying bedrock, it need not share its chemical properties. The properties of each rock unit are indicators of the geological origin and history of the rock unit.

Comparatively little can be learned of the true nature and origin of soil or bedrock units unless small samples of the unit are collected for direct detailed analysis. The basic premise assumed in collecting small samples is that, if properly obtained, they can characterize the properties of the whole unit from which they came. If enough rock units are sampled, the entire planetary surface can be characterized. From this characterization much concerning the history and, therefore, the origin of the planet can be deduced. Therefore, effective sampling of surface and subsurface rocks is of prime importance in planetary exploration.

B. Important Geological Sampling Factors

No rigorous sampling rules applicable to all rocks and all geologic situations can be envisaged. However, a few general guidelines are important for the design of a sampling mission and, therefore, a sampling system:

(1) Planetary surface rocks, both bedrock and soil, can be considered a population of sampling units, each of which may vary in properties, both horizontally and vertically, and with time (the latter, for example, by erosive or volcanic action). The choice of sampling units depends upon the objectives of the mission and experiment. Therefore, the collection of rock samples should, if possible, be undertaken within the context of a specific set of scientific objectives, and with full knowledge of these objectives. Unless collected with the geological or biological objectives (analyses) in `mind, the samples may, for example, not be representative of the sampled unit or may not be of sufficient quantity to provide a representative sample for analysis. The significance of rock analyses is dependent to a greater extent upon the soundness of the sampling plan (i.e., why, where, at what time, and in what sequence to acquire samples) than on the actual sampling process and subsequent analysis.

- (2) Sampling and analysis of geological materials should, in general, be performed after fairly detailed visual reconnaissance which is designed to determine the approximate distribution and relationships of rock units and the possible degree of variation within each unit. For the moon, for example, enough visual reconnaissance has been conducted and enough is known at present about the distribution of major rock units such as the mare, highlands, crater floors, and crater rims to allow a fairly effective sampling plan. It can be assumed that, in the case of Mars, similar knowledge will be at hand from flyby and orbiter reconnaissance photographs prior to an actual lander mission when surface sampling will be desirable. In the case of Venus, however, prior knowledge of surface rock distribution may not precede an actual lander mission, because of the relatively opaque atmosphere which may preclude visible imagery of the surface. Hence, sampling on Venus may have to be conducted on a purely statistical basis using a grid system which divides the planet's surface into arbitrary sampling units.
- (3) Geosampling is performed for the purpose of characterizing each rock unit in terms of its textural, mineralogical, elemental, isotopic, and volatile composition. Surface samples should be taken at the spacecraft landing point first, followed by subsurface samples at the same location.
- (4) For geoanalysis, bedrock samples are generally more valuable scientifically than surface dust, soil, or rubble; however, the relative importance depends on the objectives of the particular mission and experiment. The basic premise, however, is that the chemical and mineralogical properties of rocks that reflect the principal origin of the rocks cannot always be determined from the products of the rock's alteration or dilution by external effects of atmospheres, radiations, and meteoritic infall.

Therefore, it is of the greatest scientific importance to obtain fresh, unaltered specimens of planetary bedrock. Next in importance, geologically, may be the surface rubble, dust, or soil which overlies the bedrock. For practical reasons, however, sampling may have to be done in reverse order to scientific importance, with surface rubble first, because it is generally more accessible and is more significant for further mission planning objectives.

- (5) When multiple analyses are possible, attention must be paid to chemical and physical variations within rock units and between rock units. Therefore, multiple sampling is required. Lateral variations should be determined by sampling along straightline traverses across rock units and across boundaries between adjacent rock units.
- (6) Variabilities within collecting localities (i.e., each spacecraft landing site or rover sampling site) must be measured to distinguish one rock unit from another, or to show that variations within a single unit are significant. Variations can be on any scale ranging from microvariations within a single sample to larger field-scale variations. Local variations can be high, and several samples must, if possible, be collected at every locality.
- (7) Several separate samples from each locality should be analyzed individually and the results averaged to obtain the average properties of a rock unit. The reason for this is that the magnitude range in value of a property within a unit is as useful as the average values for the whole unit; or, in statistical terms, the standard deviation is as valuable as the mean. An assessment of these magnitude variations is necessary to establish confidence that differences within individual rock units will not be confounded with differences between units. A composite sample made up of many specimens has little merit for scientific investigations as opposed to engineering or economic investigations, because the analysis fails to provide an assessment of internal variability, although it may provide satisfactory mean values for the unit.
- (8) Sample localities must be correlated precisely with the position of known features on the planetary surface and with the positions of other sampling localities. For the moon and Mars, it is not enough to analyze just any region or any rock on the surface; the experimental data become most valuable only if the exact location of the sample is known

and can be predetermined. If the nature of the surface is unknown, as for Venus, preliminary random sampling may delineate probable areas of interest for subsequent systematic sampling.

C. Priority Geoanalysis Measurements Requiring Sampling

Geological measurements of high or moderate priority for unmanned stationary landers have been outlined in Ref. 10. These measurements, although originally established for the moon, are equally applicable to the surface exploration of Mars and Venus. It should be noted that the measurements recommended in Ref. 10 are in agreement with recommendations made to date by both the Space Science Board of the National Academy of Sciences (Ref. 3) and by the NASA 1965 Summer Conference on Lunar Exploration and Science (Ref. 11). These priority measurements are listed in Table 2 along with a summary of the objectives, the probable analytical techniques to be used, and the general sampling requirements for each.

Of the 11 measurements recommended, only 4 require or prefer the acquisition of rock samples:

- (1) Mineral phase abundance.
- (2) Rock texture.
- (3) Major element abundance.
- (4) Volatile compounds.

Therefore, it can be concluded that the design emphasis in any program of development of sampling devices for unmanned geoanalysis experiments should be established on the basis of the sample requirements of these four measurements. Special measurements and special missions to planets other than Mars and the moon may require special sampling requirements that will have to be established as the need arises, although many special measurements may only require routine sampling techniques. It will be shown in the following subsections that meeting the needs of the four measurements listed above will also satisfy the needs of most other conceivable, but at present nonpriority, measurements.

D. Geoanalysis Instruments and Sample Requirements

Experimental methods of potential use in carrying out each of the priority geological measurements (Table 2) have been discussed in Ref. 10. Many of these measurements can be made by different techniques and, therefore, by various instruments. Each instrument has unique

Table 2. Priority measurements for unmanned lunar and planetary surface $explorations^a$

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Table 3. Geoanalysis instruments

				4	Analysis mode ^c	
Instrument	High priority ^a	Primary measurement ^b	Principle of analysis	Deployment preferred	Deployment or acquired samples	Particulate sample preferred or required
Petrographic microscope	×	Rock textures	Transmission optics; refractive index, birefringence	I	1	×
X-ray diffractometer	×	Mineral phases	Bragg diffraction pattern; crystal structure	1	×	×
X-ray spectrometer	1	Elements (Z 11-92; sensitivity increases with Z)	Characteristic X-ray energy emission	i	×	×
Alpha scatterer	×	Elements (Z 4.92; sensitivity decreases with Z)	Characteristic backward-scattered $lpha$ -particles and protons	I	×	×
Neutron activation	l	Elements (Z 1-92)	Characteristic induced gamma radiation	×	1	I
Neutron inelastic scatter	1	Elements (Mg, Al, Fe)	Characteristic induced gamma radiation	×	I	1
Gamma ray spectrometer	×	Radioactive isotopes (K ⁴⁰ , U, Th; Al ²³ , Na ²⁴ , Fe ⁶⁶)	Characteristic natural gamma radiation	×	1	1
Mass spectrometer	×	Volatiles (inorganic; to mass 66)	Magnetic or electrostatic mass separation	I	I	×
Gas chromatograph	×	Volatiles (organic; to molecular weight 300)	Flow separation by differential diffusion	1	1	×
Specific gas analyzer	×	Volatiles (H2O, O2)	Specific reaction affecting sensor or detector	1	I	×
"Many instrument types have been utilization on unmanned spacecraf ments have been considered (Ref. 1	proposed fr ft; those ins 10) to have	er or rock analysis, but only a few have been develo or ments now in or beyond the prototype stage fa high scientific priority on the basis of their specific	"Many instrument types have been proposed for rock analysis, but only a few have been developed to such a degree of operational status that they can be seriously considered for early utilization on unmanned spoceraft; those instruments now in or beyond the prototype stage fall into this category and are here classified as priority instruments. In addition, these instru- ments have been considered (Ref. 10) to have high scientific priority on the basis of their specific utility for geological analysis aboard unmanned spacecraft.	ney can be seri rity instruments. cecraft.	iously considered . In addition, th	l for early iese instru-
^b In some cases, the primary measur the petrographic microscope will y ducting measurements on a planeh	rement madı vield minera tary surface.	e by each instrument will also yield data of anothe llogical as well as textural data. Each instrument, l . On this basis, instruments are selected for makin	bln some cases, the primary measurement made by each instrument will also yield data of another type; e.g., the X-ray diffractometer will yield elemental as well as mineralogical data, and the petrographic microscope will yield mineralogical as well as textural data. Each instrument, however, has inherent virtues and limitations that determine its particular suitability for con- ducting measurements on a planetary surface. On this basis, instruments are selected for making measurements of specific geological parameters of a fundamental nature.	nental as well c determine its pa fundamental n	as mineralogical dat irticular suitability f iature.	data, and ty for con-

cAlthough some instruments can, in principle, be configured for either deployed or prepared sample analysis modes, the choice of deployment or sample acquisition is based principally on a tradeoff between instrument reliability, measuring accuracy, and measuring suitability; e.g., the alpha scatterer is most reliable (due to simplicity) when deployed; however, it would yield tradeoff between instrument reliability, measuring accuracy, and measuring suitability; e.g., the alpha scatterer is most reliable (due to simplicity) when deployed; however, it would yield there data with an acquired and properly prepared sample. If deployed, it would be incapable of making subsurface measurements. In cases where a given instrument has reached prototype tatus, it has been configured for one of the two modes, not both. For some instruments, reconfiguration to the other mode involves only minor mechanical changes (e.g., alpha scatterer), while status, it has been configured for one of the two modes, not both. For some instruments, reconfiguration to the other mode involves only minor mechanical changes (e.g., alpha scatterer), while in others, the change involves drastic redesign of the instrument (e.g., Bragg-focusing X-ray diffractometer).

Table 3 (contd)

					Analysis mode ^c	
Instrument	High priority ^a	Primary measurement ^b	Principle of analysis	Deployment preferred	Deployment or acquired samples	Particulate sample preferred or required
Differential-thermal analyzer	1	Devolatilization temperatures	Temperature profile at constant heating rate	1	1	×
Thermogravimeter	1	Devolatilization temperatures	Weight change at constant heating rate	I	1	×
Thermoluminescence photometer	I	Radiation exposure ages	Visible light emission at constant heating rate	1	1	×
Mid-infrared (8–15 μ) spectrometer	1	Surface thermal properties	Natural thermal emission	×	. <mark>1</mark>	1
Near-infrared (1–2 μ) spectrometer	1	Mineral phases	Characteristic reflection spectrum	×	×	
Gamma-gamma backscatterer	1	Bulk rock density	Backscattered gamma radiation	×	1	I
Particle-size analyzer	1	Particle-size distribution	Sieve separation of particle-size fractions	I	1	×
Magnetic-material pickup	1	Free-iron, magnetite particles	Magnetic susceptibility	×	×	1

capabilities and inherent limitations that determine its suitability for conducting the measurement on a planetary surface. The important factor here is that the choice of instrument for a given measurement will be based largely on the complexity of its sample requirements.

A summary of geoanalysis instruments suitable for the acquired sample measurements of Table 2, with an indication of applicable sampling mode (deployment or acquired sample) is given in Table 3. For those instruments requiring a particulate sample, a summary of sample requirements is given in Table 4. The specific requirements and limitations in sampling for each instrument are discussed in the following subsections.

1. Petrographic microscope. This is a vidicon-optical device for observing the microscopic texture of rock and soils through the light interference properties of the mineral or glass grains making up the sample. In its present configuration (Ref. 4) it utilizes a particulate sample and encapsulates the particles between two glass slides, in a transparent thermoplastic medium, for insertion and viewing between two lens systems. The maximum amount of information can be obtained from particles in the 60- to 300- μ size range. Particles smaller than 60 μ tend to cling to and obscure larger ones, and form agglomerates which appear as large indistinct clumps. The microscope has its own sample handling mechanism which accepts a crushed rock powder in a hopper open at the top. The hopper has a volume of $\sim 1 \text{ cm}^3$, and an entrance screen to prevent passage of particles larger than 300 μ . The hopper divides the <300- μ particle fraction into two size ranges, 0 to 60 $\mu,$ and 60 to 300 $\mu.$ Specimens from each size range are then prepared and viewed. The hopper can be purged to clear it for a second batch of sample material.

The petrographic microscope cannot be designed for deployment sampling. It instead requires that a separate sampling device acquire and transport a particulate sample to the microscope's sample hopper. As noted above, the optimum particle size is 50 to 300 μ , although all particles <300 μ are acceptable. If the coarsest available particles are less than, say, 20 μ in size, the sample would be accepted and processed by the microscope, but the experiment would yield relatively little useful information.

2. X-ray diffractometer. This is a goniometric device that irradiates powdered rock samples with a collimated beam of monochromatic X-rays and simultaneously measures intensity vs diffraction angle of sample-diffracted X-rays; the resultant spectrum of diffraction peaks is characteristic of the composition, atomic structure, and relative abundance of mineral and glass phases comprising the rock (Ref. 8). In addition, the diffractometer can be designed to perform as an X-ray spectrometer (discussed below), enabling it to make direct elemental analyses of rocks by measuring the emitted fluorescent X-rays stimulated in the sample by the collimated incident X-ray beam (Refs. 12, 13, and 14).

The sample requirements for the diffractometer are as follows (Ref. 6): (1) the sample must be in powder form with a preferred particle-size range of 1 to 20 μ , (2) particles as large as 1000 μ may be included as long as the sample has been mechanically comminuted and has not been size fractionated, and (3) particles less than 1 μ are detrimental to the diffraction data. Therefore, if the sample is artificially comminuted, excessive pulverization must be avoided.

The best sampling mode for the diffractometer, from the standpoint of analytical precision, is to acquire, prepare, and mount a powder sample for insertion into the goniometer. In this mode, the powder, following its acquisition, must be size limited to less than 1000 μ , then poured into a tablet-shaped cup whose dimensions are approximately 2 cm wide by 3 cm long by 0.3 cm deep, and which has a thin (0.002 in.) beryllium-foil bottom slightly curved cylindrically upward.

The powder then should be compacted slightly against the beryllium-foil bottom to increase the particle density at the foil-sample interface. The compacted sample depth should be between 1 and 2 mm. This requires an uncompacted volume of approximately 2 to 4 cm³ (or approximately 3 g) of powder. Vibratory compaction is not desired because it may cause deleterious size-and-density fractionation as well as artificial preferred orientation of particles within the powder sample. Simple compressive compaction is recommended, although means must be included to prevent rupture of the thin foil bottom of the sample cup.

The present prototype diffractometer (Refs. 10 and 15) is designed to accept multiple sample cups in succession. The cups are loaded with powder outside the goniometer and then inserted horizontally into the analysis position where they are then free to rotate as required by the goniometer. Following analysis, the cup is rotated back to horizontal by the goniometer and then can be pushed

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Table 4.

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Can sharø	sample	•	4	↓ ↓	-	•	¥ ¥	↓ ↓	¥ ¥	*	*	•
Sample condition after processing and analysis	(other than possible pulver- ization)	Encapsulated	Undamaged	Undamaged	Undamaged	Melted	Heated to 600°C	Heated to 900°C	Heated to 900°C	Heated to 900°C	Heated to 500°C	Size fractionated
Analysis time	required, h/sample	ī	1.5	ī	3-10	0.5	1.3	~0.1 and/or continuous	~2	1-10	8	0.1
Max allowable bulk tem- perature	rise, % above ambient °K	25	25	25	25	10	10	01	10	01	ŝ	100
Special	sensitivities	None	Fe, <1% by wt	Metals, <1% by wt	Metals, <1% by wt	Sampler out- gassing	Sampler out- gassing	Sampler out- gassing	None	None	None	All fragments <1% by wt
Size fractionation limis or	requirements	Prefer removal or separate sample of <60-μ fraction	Particles $<1~\mu$ undesirable	No restrictions	No restrictions	No restrictions	No restrictions	No restrictions	No restrictions	No restrictions	Extensive grinding unacceptable	Natural size fractions must not be altered by >1%
	Accept- able, μ	330	1000	Solid	Solid	<1000	<1000	1000	300	300	Solid	No re- quire- ment
Particle-size range	Best, μ	60-300	1–20	<20	<20	No re- quirement	No re- quirement	No re- quirement	<20	<20	<100	No re- quirement
. Min	Cume,	1.5	7	2	7	2–3	2–3	1	2	7	1	5-10
Porosity,	2 2	Dispersed particles	<60 (compacted)	< 60 (compacted)	<60 (compacted)	No requirement	No requirement	No requirement	<40 (compacted)	No requirement	< 60 (compacted)	No requirement
Shape	ot sample	Dispersed particles in transparent matrix	Flat cup 2 × 3 × 0.2 cm	Flat cup 2 × 3 × 0.2 cm	Flat cup, 5 cm diam, 0.1 cm deep	Nonspecific (in furnace)	Nonspecific (in furnace)	Nonspecific (in furnace)	Pelletized	Nonspecific (on tray)	Flat cup, 3 cm diam, 0.1 cm deep	Dispersed
	Instrument	Petrographic microscope	X-ray diffractometer	X-ray spectrometer	Alpha scatterer	Mass spectrometer	Gas chromatograph	Specific gas analyzer	Differential-thermal analyzer	Thermogravimeter	Thermoluminescence photometer	Particle-size analyzer

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out of the analysis position by insertion of the next sample cup. A standard earth sample is carried in the goniometer from launch and is analyzed first for instrument calibration following spacecraft landing. The standard is ejected by insertion of the first planetary sample.

In addition, the samples that are prepared in cup form for the X-ray diffractometer can also be used for analysis by the X-ray spectrometer and by the alpha particle scatterer.

Alternately, the X-ray diffractometer can, in principle, be configured for deployment sampling. This requires a different goniometer design than the present prototype instrument; some developmental work has been done on such an instrument (Ref. 13). In this configuration, sampling would be accomplished by deploying the entire goniometer to the planetary surface in such a manner as to bring a beryllium port firmly against the surface material. For success, this mode requires that the planetary surface material be loose, uncompacted powder with an average particle size of less than 100 μ and contain no particles larger than from 1 to 2 mm.

3. X-ray spectrometer. This instrument is designed to measure the elemental composition of rocks and soils by analyzing the fluorescent X-ray spectra of a rock sample excited by artificial beams of energetic particles (electrons, alpha particles) or electromagnetic radiation (hard X-rays, gamma rays). Space adaptable versions of this instrument have been proposed and some developmental work done (Refs. 16 and 17). The X-ray spectrometer can be configured for either deployment or acquired-sample sampling mode, although the acquired sample mode will give highest analytical precision. Samples should be in powder form, preferably with particle size less than 10 μ , although a mixture of particle sizes up to 1000 μ is acceptable. For acquired samples, the powder must be spread out in a tablet-shaped cup similar to that of the X-ray diffractometer; analysis, however, is performed on the top, uncovered surface of the compressed powder. Approximately 2 cm³, or 2 g, of loose powder are required for each analyzed sample.

4. Alpha scatterer. This device measures the elemental composition of rock or soil by irradiating the sample with alpha particles from a radioactive source (e.g., Cm^{242}) and measuring the energy spectra of backscattered alpha particles and yield of protons from alpha-proton reactions; these spectra are characteristic of the composition and relative abundance of elements present in the scattering material (Ref. 18). The instrument can be configured for either acquired sample or deployment sampling. A flight-qualified instrument has been developed for deployment sampling on *Surveyors V*, *VI*, and *VII*.

Sample requirements for the alpha scatterer are extremely simple. Basically all that is required is that the source-detector head be positioned approximately 3 cm from a sample surface. The sample can be either powdered or solid rock, so long as the upper surface is planar and relatively smooth on a millimeter scale. For an acquired sample mode, where high precision is desired, the sample should be powder with particle sizes $<20 \ \mu$, and formed into a smooth flat surface approximately 5 cm in diameter and 0.1 cm deep. Maximum acceptable particle size is 1000 μ , assuming the presence of abundant smaller particles.

5. Neutron activation and neutron inelastic scattering spectrometer. These two techniques are similar in that both measure the elemental composition of rock and soil by measuring the characteristic induced gamma radiation from a sample bombarded by neutrons from a radioactive source (Refs. 19 and 20.) This technique does not require sample preparation and deployment is the only feasible mode of analysis because a large sample area is required. The device is boom-deployed onto the planetary surface. The active sampling zone is on the order of 100 cm² by 60 cm thick (Ref. 21). The sampled area becomes intensely radioactive as a result of the neutron bombardment.

6. Gamma ray spectrometer. The gamma ray spectrometer measures the natural gamma spectrum emitted by planetary surface rocks and soils. The intensities and energies of the emitted gamma rays are characteristic of the composition and relative abundance of the radioactive isotopes (e.g., K^{40} , U, Th) contained in the sample. Sampling can be done only by the deployment mode and at the end of a long boom to position the spectrometer away from the spacecraft structure; spacecraft materials inherently contain traces of radioactive isotopes, which will interfere with the measurement if too close to the detector. In addition, interfering secondary radiations may be induced in the spacecraft components by cosmic ray bombardment.

7. Mass spectrometer and gas chromatograph. These instruments can be used to measure the inorganic and organic gaseous volatile constituents which are contained in rocks and soils and formed by thermal decomposition of rocks and soils. The instruments can be utilized independently or in tandem, the latter being more likely. Gases will be released from the sample by heating, and will be separated by differential diffusion in the gas chromatograph, and then introduced into the mass spectrometer for species identification. Only the acquired sample mode of sampling can be used. During analysis, the sample must be in particulate form to facilitate gas evolution at practical heating rates. Particle size is not critical but should be finer than 1000 μ . Approximately 2 to 3 g of sample powder is required per analysis and must be placed into a heating cavity of unspecified shape. The sample will be heated (to determine the temperature-vs-gas evolution profile) to at least 600°C and probably to melting (which will be between 1100 and 1700°C, depending on rock composition).

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8. Specific gas analyzers. These are devices that measure the composition and concentration of specific gases contained in rocks and soils, such as H₂O and O₂, and are insensitive to other gases (Ref. 22). The sampling mode is by acquired sample only. Samples must be powders with particle size less than 1000 μ and be placed in a heating cavity similar or identical to that used with the mass spectrometer/gas chromatograph system described above (and also similar to that of the DTA, DTG, and TLP instruments described below). Approximately 2 to 3 g of sample are required and must be heated slowly to 900°C.

9. Differential-thermal analyzer (DTA) and differential-thermal gravimeter (DTG). These devices measure the change in temperature (DTA) and the change in weight (DTG) of rock and soil samples heated at a constant heating rate; the Δ T-vs-time profiles indicate mineralogic transitions within the sample caused by enthalpic effects. These data can be used to characterize the mineralogical and volatile composition of the samples.

Both devices require acquired particulate samples and will accept particle sizes less than 300 μ , although optimum particle size is <20 μ . Approximately 1 g or 1 cm³ of loose powder is sufficient for each analysis. The powder is placed in a small crucible and heated slowly to approximately 900°C.

10. Thermoluminescence photometer (TLP). This device measures the visible luminescence of rock and soil samples as they are heated to approximately 500 °C. The pattern of luminescence intensity vs temperature (glow curve) is characteristic of the thermal and radiation history as well as composition of certain kinds of rocks,

particularly those containing carbonate, fluoride, and sulfide minerals. The usefulness of the technique, however, is severely limited by numerous complicating factors, many of which involve sample preparation. For example, variations in the particle size, the amount of grinding, and the temperature achieved during grinding can markedly and unpredictably influence the character of the glow curve. Samples must be acquired in particulate form with particle size <100 μ . Approximately 1 g of sample is needed for each analysis and must be spread out and packed smoothly in a flat cup approximately 1 to 3 cm in diameter.

11. Gamma-gamma backscatterer. This instrument measures the bulk density of surface rocks and soils by irradiating them with gamma rays and measuring the amount of backscattering, which varies with bulk density. Only the deployed mode of analysis can be used.

12. Particle-size analyzer. The purpose of this device is to measure the mean particle size⁴ and size distribution of natural particulate rock and soil material on, or below, a planetary surface. The proposed method (Ref. 23) consists of sieving the sample into a series of size fractions which are retained on individual sieves. The particle-size range to be measured is from 1 to 5000 μ . (Larger particles can be seen with television.) The sample must be acquired from the surface or subsurface (with depth depending on the objectives of the experiment and sampler limitations) in such a manner that its particle-size distribution is not altered during acquisition. This means that, for a given volume of material that is removed from the surface, no particles should be lost, and no particles should be pulverized or otherwise reduced in size. Because, with this device, the individual size fractions are retained intact, they can be used after the particle-size analysis for analysis by other instruments such as those for elemental, mineral, and biological analyses.

E. Summary of Geoanalysis Sample Requirements

The parameters previously discussed and summarized in Table 4 indicate that the sample requirements of all geoanalysis instruments are nearly identical. On the basis of these requirements, there are three distinct groups of geoanalysis instruments in which the instruments comprising each group (1) can share the same sample, either simultaneously or sequentially; (2) have the same particlesize requirements, same sample geometry, and same

^{&#}x27;Size is defined here as the mean particle diameter.

Table 5.	Instrument	groups	that can	share a	single san	nple
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Instrument	Mode of . sharing	Best particle- size range, μ	Sample volume, cm³	Sample geometry	Sample effects	Sample could be used further by other instru- ments
X-ray diffractometer Alpha particle scatterer X-ray spectrometer	Sequential	<2 <u>0</u>	2	Powder spread out and packed flat to produce large area surface and irradiated	Not altered significantly during analysis	Yes
Petrographic microscope	_	50–300	1.5	Powder grains dispersed in plastic matrix and viewed	Encapsulated in transparent matrix	No
Mass spectrometer Gas chromatograph Specific gas analyzer Differential-thermal analyzer Thermogravimeter Thermoluminescence photometer	Simultaneous	<100	1–3	Loose powder placed in furnace chamber and heated	Heated to at least 500°C, possibly to melting	No (generally)

Table 6. Acceptable sample requirements for all geoanalysis instruments

Acceptable requirement
Surface soil
1
Powder
<300 μ
3 cm ³
Metals <1% by wt
Minimum possible
No heating above 25% of ambient planetary surface temperature No excessive grinding

sample size; and (3) produce similar effects on the sample material. These groups are listed in Table 5.

In addition to the above groupings, the minimum sample requirements of all geoanalysis instruments can be met by one set of requirements shown in Table 6.

IV. Biosampling

A. Bioanalysis Principles

The strategy for biological exploration of planets considers sampling and life-detection analysis of surface soils as the principal objective (Ref. 24). Bioanalysis of planetary soils will have three specific objectives: (1) determine the composition and concentration of biochemical compounds in the soil; (2) establish the presence of biochemical processes indicative of living organisms; and (3) determine the biological environmental conditions (temperature, UV intensity) of the soil. The first two objectives relate to whether life is present, and the third objective relates to whether life could be present.

A few basic assertions regarding these objectives from the sampling standpoint are as follows:

(1) It is possible that planetary biochemistry does not include familiar biochemical compounds, that organic matter may be present in the soil but life forms absent, and that there may be no significant amount of organic matter in the soil (except that introduced by meteorites).

- (2) If any life exists on planets such as Mars, much, if not all of it, will be microbial in form and located at, or near, the planet's surface (Ref. 24), and probably will be associated with soil particles, rather than free in the soil (Ref. 25). There is some evidence that the highest concentration of microbial organisms may be associated with the smallest particles in the soil aggregate (Refs. 26, 27, and 25), although other studies show no such correlation (Ref. 28).
- (3) The planetary organisms and their byproducts may resemble known earth organisms; therefore, potential earth-derived contaminants must not be added to the planetary soil by the sampling system.
- (4) The total concentration of organic matter in planetary soil may be extremely low (0.01 to 10 mg/g soil), requiring collection and processing of relatively large amounts (grams to kilograms) of soil for positive analytical results. In any biochemical analysis to detect bioactivity in soil, the effects of inert (nonliving) organic matter must be overcome to detect a small fraction of active biomass; hence, there is the need for sample processing or highly sensitive analytical techniques.

B. Important Biosampling Factors

In biosampling, as in geosampling, it is important to precede actual sampling by a general reconnaissance of the surface to be sampled so as to select appropriate sampling sites, and to decide what kind and how much surface sampling may be required. If, for example on Mars, photo reconnaissance indicates that macroscopic forms of life exist (e.g., that observed seasonal surface darkening is biological in nature), then sampling procedures may have to be modified in accordance with the new objective of characterizing the life instead of detecting it. In particular, reconnaissance will give some indication of the localization of macroscopic biological forms and the concentration of total organics in the surface, knowledge which is necessary for determining the sample size requirements for surface analysis experiments. It will also indicate the extent to which roving sampler systems are required for sampling wide areas or boundaries between observed units of biological material.

It has been recommended (Ref. 24) that even the first and simplest planetary lander system be capable of surface mobility. In principle, this requirement means that the landed system must have the capability of sampling the surface at many diverse points away from the landing site. This sampling mobility is required for three reasons:

- (1) To escape the area altered, contaminated, or sterilized by the retrolanding maneuvers. (Although comparative samples should be obtained from within this area to determine whether the indigenous biochemical systems react to the retrolanding.)
- (2) To search for ecological sites where life would more likely be found, or if widespread, where it would be present in higher population density than elsewhere on the planet.
- (3) To reach sites of known, or suspected, biological activity (discovered on the basis of prior reconnaissance data) which are inaccessible to direct landing.

The minimum roving distance required is determined by the size of the area altered by the retrolanding. The size of the area altered depends upon the number and size of the retrorocket engines thrusting at the surface and upon the engine cutoff altitude. A preliminary analysis (Ref. 29) shows that, for a single Voyager rocket engine thrusting vertically downward, the following conditions would be imposed:

- A total of 400 lb of exhaust gases will be released, 25 lb below the 100-ft altitude.
- (2) The gases will consist of:

Gas	Weight fraction
. CO	0.05
CO_2	0.12
H_2	0,26
H_2O	0.26
N ₂	0.31

The gases are noncondensing at anticipated Mars daytime surface conditions. The carbon content will be oxidized. Eighty-eight percent of the products have molecular weights less than the (Mars) atmosphere and would dissipate upward by gravity buoyancy.

- (3) For engine cutoff altitudes of from 5 to 10 ft, the momentum flux of the exhaust gases is sufficient to transport loose dust as far as 100 ft from ground zero.
- (4) For cutoff altitudes below 50 ft, the 100°C exhaustgas-temperature isotherm is at a radial distance of approximately 26 ft, and the 0°C isotherm at 32 ft. For cutoff at zero altitude, the maximum gas temperature at ground zero can reach 2800°C.

Therefore, these preliminary data indicate that, for a single *Voyager* engine, ambient surface temperature could be altered out to a radial distance of approximately 30 ft and dust transportation could occur to approximately 100 ft.⁵

There is little scientific basis for fixing a maximum roving distance for the mobile sampling system, since the greater the ranging capability, the greater the chances of discovering areas of particular interest to biology (Ref. 24). Therefore, the maximum roving distance must be based on engineering constraints.

After a spacecraft system with mobile sampling capability has been landed, it is desirable to:

- (1) Investigate (sample and analyze) as many different surface soil types and localities as possible.
- (2) Follow concentration gradients across the surface, especially across boundaries between distinct biological populations. In some cases, it might be of interest to backtrack (Ref. 24).

Because of the mobility requirement, a conflict exists in the use of the same landed vehicle to perform extended roving, sampling, and complex analyses that require significant weight and power. In a rover, a large amount of the allotted weight and power would be required for guidance apparatus, communications system, and analytical instruments, whereas the remaining weight and power would go to locomotion and sampling which are the most important functions. The heavier and more complex the experiment payload, the shorter the roving distance from the landing site; hence, a compromise is necessary between mobility and experiment complexity (Ref. 24). A solution to this problem may be to limit the payload of the roving vehicle (or projectile) to a sample acquisition and storage system which collects single or multiple samples at various sites and returns them by locomotion or dragline to the main spacecraft for analysis and data transmission.

Regardless of whether both sampling and analysis is done by the same or separate spacecraft systems, the basic strategy outline for biological exploration of planets is based on the assumption that samples will be available that are not modified either by the initial landing operation or by the subsequent sample acquisition and processing operations. This implies that, if retrorockets are used for the final touchdown, a sampling system must be provided whose minimum capabilities are (1) to collect soil samples outside the area affected by the retroblast, and (2) to accomplish this aseptically. Therefore, even without a roving capability, the sampling system must have a reach capability.

It is generally assumed that surface soil will provide the best sample for biological analyses. Under some circumstances, however, especially for desert soils in the harshest environments, it may be better to obtain a subsurface soil sample, in which organisms would have been protected from desiccation, lethal solar radiation, and the contamination or sterilization effects of retrorocket exhaust. Therefore, the sample acquisition system would have to include not only a reach capability, but also a depth capability.

Sampling techniques can influence the type and number of organisms recovered from a soil aggregate. For examples: (1) certain types of aerobiological samplers are deleterious to moisture-sensitive strains of bacteria and viruses; (2) certain samplers have a built-in bias for, or against, particles of certain size (and consequently the type and number of organisms characteristically associated with that size of particle); (3) certain types of sample selection as well as processing may promote the growth of some types of organisms, while inhibiting the growth of others (Ref. 26). Therefore, in developing sampling systems, the various kinds of qualitative and quantitative bias that can be introduced into the biological experiments by the sampling devices and processes must be considered, and whether these effects are bad or advantageous must be determined by testing.

Sample acquisition and preparation should involve a minimum of sample handling and processing (Refs. 30 and 24). In spite of the low probable organism concentration in planetary soils and the possible larger number

⁵These numbers are only tentative and must be used with caution until the results of further tests become available.

of organisms associated with the smaller particles in a soil aggregate, any advantage that might be gained from enriching biological activity by processing a sample (e.g., by concentrating the finer particle-size fraction) can probably be surpassed at a lower power and weight requirement by collecting a larger bulk sample (Ref. 25). Most available evidence indicates that bulk sampling (i.e., the collection of all particles in a given volume of soil) is the most efficient and most practical sampling in terms of biological requirements and engineering constraints, and that it is most representative of the parent soil material and, therefore, its biological content.

C. Biological Measurements Requiring Sampling

Biological measurements for early unmanned lander missions to Mars have been recommended in a previous planning document (Ref. 24). The measurements recommended for these objectives are listed in Table 7. Also shown in Table 7 are the type of soil processing required and the applicable analytical instruments. In many respects, these measurements are also applicable to Venus, Mercury, and several other planetary bodies.

Soil acquisition is required for most of the recommended biochemical measurements except those for

			Applicable analytical instrument					
Objective	Measurement	Soil processing required	Gas chro- mato- graph— mass spec- trometer	Infrared spec- trometer	Fluorim- eter	Polarization and optical rotary dispersion	Radiation detector	Nephelometer
Determine biochemical	Organic compounds							
compounds	Total organics	Combustion to gas						
	Ratio C, H, O, N, S	Oxidation or reduc- tion to gas	x	x	-	-	_	-
	Molecular wt range	Wet chemical	-	x	—	-		
	Insoluble organics	Combustion, pyrolysis	x	x	-	-	-	-
	Functional groups	Wet	-	x	-	-	-	-
	Volatile compounds	Heating	x	-	—	-		· –
	Volatile organic compounds	Wet	x	x	x	-	.—	-
	Isotope ratios C ¹² –C ¹³	Combustion to gas	x	-	_	-	_	-
	Optical activity (net)	Wet	-	-	-	x	—	
Determine presence and character of biochemical processes	Effects of exchanges of matter and energy between biosystem and its environment	Incubation						
	Uptake and production of gases		x	-	x	-	x	-
	Uptake and excretion of soluble compounds		X	-	x	-	-	-
	Accumulation of anabolic compounds	Without media (in situ)	x		x	-	-	-
	Changes in total mass	In soil-extract media In preselected media	x	-	-	-	-	x
	Heat production		-		-	-	-	-
	Chemiluminescence		_	-	x	-	-	_

Table 7. Biochemical analyses of soil

atmospheric water content and surface UV intensity. It should be noted, however, that several of the soil measurements for biochemical processes such as gasexchange reactions can, in principle, be made without soil acquisition by deploying suitable sensors onto, or into, the soil layer.

Nearly all biological experiments now being considered for unmanned planetary exploration, whether for determining the composition of biochemical compounds or the presence of biochemical processes, involve rather elaborate sample processing steps to prepare the acquired soil sample for analysis. This processing is in addition to that required to get the sample initially into a proper physical form, such as suitable particle size and total volume. The processing may consist of one, or more, of the following operations:

- (1) Pyrolysis. Decomposition or degradation of the sample substances by dry combustion heating, primarily to convert it into a complex mixture of gaseous products, or release chemically combined gaseous constituents which can then be analyzed by a gas chromatograph-mass spectrometer system.
- (2) Volatilization. Conversion of condensed liquid or solid sample substances (by heating or reacting with added reagents) into a gas, or to liberate adsorbed gas for analysis.
- (3) Oxidation or reduction. Addition of oxygen or hydrogen, respectively, to the sample substances to convert the total carbon to gaseous CO_2 , or total oxygen to H₂O, respectively, for further analysis.
- (4) Addition of reagents or catalysts. Addition of solid or liquid reagents to produce volatile derivatives from nonvolatile sample compounds.
- (5) Hydrolysis. Decomposition of organic compounds by interaction with water, either cold or hot, and alone or in the presence of acids or alkalies.
- (6) Extraction. Dissolving certain sample substances with a specific liquid solvent. The extract can then be further processed or directly analyzed, for example, by IR-spectroscopy or fluorimetry.
- (7) Incubation. Isolation of sample material with, or without, an added nutritional medium (usually aqueous) in an apparatus in which conditions can be controlled and resulting cultures or metabolic processes (e.g., evolution or consumption of gases) observed for a period of time.

Because these processing steps are inherently part of the analytical scheme, they, and any attendant mechanisms, should be considered a part of the analytical system and not a part of the sampling system.

D. Bioanalysis Instruments and Sample Requirements

Apart from television observations (either through a camera or microscope lens), essentially all of the important biological analyses proposed for the exploration of planets may be accomplished by the following few analytical instruments:

- (1) Gas chromatograph.
- (2) Mass spectrometer.
- (3) Infrared spectrometer.
- (4) Fluorimeter.
- (5) Polarimeter.
- (6) Nephelometer.
- (7) Radiation detector.

These instruments are especially well-suited for analyses of biochemical compounds and detection of biochemical processes. A summary of their operating characteristics and sample requirements is listed in Table 8.

In addition to these instruments, several others are suitable for measuring the physical, chemical, and mineralogical (i.e., geological) properties of the biochemical soil environment. These are

- (1) Differential-thermal analyzer.
- (2) Specific gas analyzer.
- (3) Alpha scatterer.
- (4) X-ray spectrometer.
- (5) X-ray diffractometer.
- (6) Petrographic microscope.

These instruments were described in Section III-D and Tables 3 and 4.

In general, there are two basic modes for conducting biological experiments with unmanned lander spacecraft missions. The first mode is to use individual instruments with a simple, specific objective, and which are small, lightweight, and can operate independently. Examples are the Gulliver, Wolf Trap, and Minivator experiments.

Instrument	Priority	Can share same processed sample	Primary measurement	Principle of analysis	Sample requirement	Particular contamination sensitivities
Gas chromatograph	High	۲.	Gases, volatile compounds	Flow separation by differential diffusion		Any foreign organic compounds
Mass spectrometer	High	7	Gases, volatile compounds	Magnetic and electrostatic mass separation	Acavisition of 0.01–1 cm³ of	Equipment outgassing
Infrared spectrometer	l		Soluble molecular compounds	Characteristic absorption spectra	particulate soil material,	Foreign organic compounds
Fluorimeter ^a	I	ł	Specific compounds and biochemical processes	Characteristic light emission (UV—excited and bio- luminescence)	with particle size generally <2 mm, followed by one or more of several forms of processing (see text	Specific fluorescent or quenching organic compounds
Polarimeter (optical rotary dispersion)	I	¥	Specific biological compounds	Specific rotation of transmitted polarized light	discussion).	Inorganic opaque colloidal solids
Nephelometer ^b	I	7	Growth	Optical scattering		Inorganic opaque colloidal solids
Radiation detector ^c	1		Growth	Uptake of radioisotope-tagged media		Low energy eta , γ radiation
"For example, Multivator-Minivator. ^b For example, Wolf Trap.	tor-Minivator. 3p.					

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Table 8. Bioanalysis instruments and sample requirements

°For example, Gulliver.

The second mode is to use a complex integrated laboratory (such as the ABL concept) which is large, heavy, and which includes many diverse instrument components operating together or semi-independently. In either case, sample acquisition is required. Several of the individual instruments (Gulliver, Wolf Trap) have their own sample acquisition device (sticky string, vacuum cleaner), which is an integral part of the instrument. Other individual instruments, as well as the ABL integrated laboratory, require separate sample acquisition devices.

The primary sampling requirement for bioanalysis is the acquisition of uncontaminated surface particulate material, generally of particle size less than 2 mm and preferably of particle size less than 300 μ , with strong preference for the smallest particle sizes in the <300- μ fraction. For the individual life-detection instruments, only small amounts ~0.001 to 2 cm³ (1 to 2000 mg) of sample material are required, and the analysis is conducted essentially only once. Whereas, for the integrated ABL laboratory, large amounts of several liters (kilograms) are required and many analyses are conducted during the lifetime of the laboratory.

Sample handling should avoid heating samples above the maximum ambient temperature that the sample normally is subjected to in its pre-acquisition environment (e.g., noon surface temperature). For analysis of biochemical processes, sample preparation should not involve grinding or pulverization to reduce the particle size. For analysis of biochemical compounds, particle-size reduction may be required to increase the effectiveness of subsequent chemical processing.

E. Summary of Bioanalysis Sample Requirements

In summary, the sampling requirements for bioanalysis experiments are:

- (1) Surface particulate material (planetary soil). Particle size should be less than 300 μ , with further preference for the smallest size fraction within this size range.
- (2) Uncontaminated material (i.e., (1) acquired outside the area affected by retrorockets, and (2) not subsequently contaminated by spacecraft components or their derivatives).
- (3) Volume of sample required is (1) variable from 0.001 to 2 cm³ (1 to 2000 mg) for the individual bioanalysis instruments, and (2) 1 to 101(1 to 10 kg) for the integrated laboratory.

- (4) Sample material is not to be heated above maximum ambient planetary temperature.
- (5) Grinding or pulverization is allowed only for analysis of biochemical processes.

V. Comparison of Geosampling and Biosampling

It has been shown that, although bioanalysis of planetary materials may be distinctly different from geoanalysis of the same materials, biosampling (as presently inferred) is basically no different from geosampling. Both involve acquisition and some form of processing of surface soil material. However, the specific mode of soil sampling and the detailed sample requirements for the two kinds of analyses may differ somewhat, depending on the specific objectives of the missions and individual experiments. A comparison of the most important parameters for geological and biological samples is shown in Table 9.

Geological and biological experiments require:

- (1) Particulate rock samples.
- (2) Samples from surface and subsurface soil.
- (3) Sampling at multiple sites, if possible.
- (4) Multiple samples at each site, if possible.
- (5) Processing of acquired samples at least to the extent of excluding soil particles larger than approximately 2 mm in diameter.
- (6) Avoidance of contamination or other deleterious effects during the sampling operations.

The number of sites sampled, the depth of sampling, and the frequency of sampling all depend on the mission constraints and experiment objectives, and will influence, as well as depend to some extent on, the design and functional requirements of the sampling system to be used.

A fundamental difference between geological and biological analysis is the relative importance and extent to which sample processing must be conducted. In bioanalysis, all life-detection systems (experiments) proposed to date (except TV) require acquisition and rather complex processing of some amount of soil sample. Failure at any stage in the acquisition and processing of the sample can result in failure of the experiment. In geoanalysis, however, most experiments require only simple processing of acquired samples.

Table 9. Comparison of geological and biological sampling strategy and sample requirements for analyses of planetary surface materials

Parameter	Geological experiments	Biological experiments	Remarks
Materials to be analyzed	Rocks	Organisms and/or organic matter contained in, or attached to, rocks or rock particles	Both geological and biological experi- ments involve analysis of geological materials that will be present on all planet surfaces
Analyses to be performed	Inorganic chemistry of rocks Elements, isotopes Volatiles; condensed, chemically combined Minerals, glass phases Physical properties of rocks Particle size, intraparticle grain size Particle and grain shape Porosity, bulk density Cohesiveness, bearing strength Magnetic, electric constants Photometric constants (bulk material)	Organic chemistry Organic compounds Amino acids, purines, chloro- phyll, etc. Polypeptides, polynucleotides Volatile compounds Lower fatty acids, alcohols, aldehydes Hydrocarbons Biological catalytic activity, enzyme tests Optical activity in organic molecules Metabolic activity Isotope fractionation Growth (increase in mass) Photosynthesis	Geo-experiments are concerned with the properties of the rock matter, whereas bio-experiments are pri- marily concerned with any organic matter contained on or in the rock and are secondarily concerned with the rock matter
Suitable analysis mode	Deployed instruments (no sample ac- quisition) and/or acquired samples	Acquired samples only	Must have a sample acquisition device for biological experiments. Some geoanalysis instruments can be con- figured for either, or both modes, while others must have an acquired sample and still others can only be deployed
Surface or subsurface samples	Both desired. Surface samples satisfactory	Surface samples preferred. Subsur- face sample only would be unsatisfactory	Both geo- and bio-experiments require at least a surface sample. Subsurface samples would be used by both if available
Soil or bedrock samples	Bedrock preferred; soil satisfactory	Particulate soil preferred; bedrock secondary (for life vestiges)	Geoanalysis experiments prefer bed- rock because it is more indicative of primary planetary properties, pro- cesses, and history than soil. Bio- experiments prefer surface soil be- cause it is the most likely region of the surface where biological matter might be found
Sampling at multiple sites	Desired	Desired	Especially across boundaries between two distinct geological or biological units. Multiple sample may be re- quired to recognize and define such boundaries
Multiple sampling at each site	Desired, if each sample can be ana- lyzed separately. Otherwise unnecessary	Desired	Generally desirable for both geo- analyses and bioanalyses to increase statistical confidence in analytical data and to determine mean varia- tion in properties at a given sam- pling site

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Table 9 (contd)

Parameter	Geological experiments	Biological experiments	Remarks
Time frequency of sampling	Once at each site satisfactory unless other evidence suggests rapid geologic changes occurring be- cause of wind, seismicity, or volcanism	At least twice daily and seasonally	Frequent sampling at each or one sampling site may be extremely important biologically, but unim- portant geologically within the prac- tical lifetime of the spacecraft
Amount of sample required	Minimum of 1 cm ³ (~1 g) and maxi- mum of 10 cm ³ (~10 g) per instrument for acquired sample analysis. For nearly all instruments, 3 cm ³ (~3 g) is satisfactory. No sample required for deployed instrument analysis	Minimum of 1 cm ⁵ (~1 g) and maxi- mum of 20 cm ⁸ (~20 g) for indi- vidual bioanalysis instruments, and 1 to 10 l (~1 to 10 kg) for integrated laboratory	In general, small amounts of sample (grams) are satisfactory for most geoanalysis and individual bio- analysis instruments, whereas the integrated biological laboratory concept requires relatively large amounts of sample (kilograms). Usually, the amounts required are based on instrument requirements, but in bioanalysis, depend also on the expected concentration level of organic matter in the soil
Sample processing required or desired	Samples must be particulate with particle size $<1000 \mu$. Further size fractionation prior to delivery to instrument undesirable unless all size fractions can be examined. Pulverization of sample to $<20 \mu$ desirable for some instruments but undesirable for others	Natural particulate samples only, with particle size generally $<1000 \mu$. Size fractionation and concentration of finest fraction may be desirable for some experi- ments. Pulverization of sample undesirable, although some agita- tion to break up cohesive soils may be necessary	Processing of biological samples should be gentle and minimized to avoid damage to, or loss of, organisms and volatile compounds. Processing of geological samples can be rela- tively vigorous
Potential modifying effects to sample	Excessive grinding or pulverization and extreme temperature increases should be avoided. Contamination from spacecraft components and from mixing separate samples is relatively harmless, although sample mixing may yield uninter- pretable data	Temperature increase, lethal radia- tion exposure, and agitation of samples should be avoided prior to delivery to instrument. Con- tamination by organic and other compounds may negate the ana- lytical results. Mixing of samples is relatively harmless and may be required to increase the total amount of sample analyzed	Generally, the bioanalysis results will be affected more deleteriously than geoanalysis results by any external modifying effects such as heating, radiation exposure, and contamination

In addition, several important geo-experiments can be conducted without direct acquisition of a sample by use of the deployed instrument analysis mode; therefore, no sample processing is necessary and failure or success of the experiment does not depend on success or failure of a sampler system. In this regard, it has been strongly recommended that life-detection experiments be developed for deployment analysis to eliminate sampling errors which are a major factor in terrestrial soil analysis and interpretation (Ref. 30).

For bioanalyses, one instrument may be used for several types of analyses and with any one of several of the different types of sample processing. For example, the gas chromatograph may be utilized following either pyrolization or incubation of a sample for the analysis of volatile biological compounds or the detection of gasevolving biological processes, respectively. Whereas, in geoanalysis the instrument usually can perform only one type of analysis and function properly only after one specific form of sample processing. An example is the X-ray diffractometer which measures (directly) only the mineralogical composition of the surface particles of a powder pack.

Another distinct difference between geoanalytical and some bioanalytical experiments is the volume of sample material that may be required for definitive results. In

geoanalysis, the sample volume required for a satisfactory analysis is a function only of the instrument requirement; for example, if 2 cm³ of soil are supplied to the X-ray diffractometer, a successful analysis will result. In bioanalysis, on the other hand, certain bioexperiments designed to ascertain the mere presence of viable organisms (i.e., the individual instrument approach as opposed to the complex ABL-type laboratory) can succeed or fail simply on the basis of the sample volume acquired (the probability of detecting one organism in a soil that contains very few organisms varies with sample volume). The sample volume required for some bio-experiments, therefore, is dependent not on the instrument requirements, but on the concentration of organisms in the sample. For example, if the organic content is low, it may be necessary to collect and process 100 cm³ of soil before a definitive analytical result can be obtained, and if the organic content is high, a definitive result could be obtained with a sample as small as 1/100 cm³. Therefore, in regard to the burden imposed on the sample acquisition device, the amount of sample material that may have to be collected is more or less low and fixed for geoanalysis experiments, but can be high and variable for bioanalysis.

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The most important requirement of biological sampling which does not apply to geological sampling is the strict aseptic conditions which must prevail for successful biological analyses. Sample acquisition and processing devices must be sterile to prevent contamination of the analysis by minute amounts of earth-derived organic or biologic materials.

The terms geosampling and biosampling have been used extensively in past sampling studies. Although there are fundamental differences in geoanalysis and bioanalysis, there are basic similarities between the functions of geosampling and biosampling. Both functions consist of the same two basic operations: (1) acquisition of rock or soil materials from a planetary surface, and (2) presentation, in suitable form, of this material to analytical instrument systems. Differences in the objectives of sampling arise only after the samples are acquired and analyses are to be performed. Geoanalysis experiments are concerned with the inorganic properties of the rock matter, and bioanalysis experiments are concerned with the organic matter contained in, or attached to, the rock.

There is no compelling reason to retain the term biosampling in the technology of planetary sampling-system development. The operation defined by this term is identical to the operation defined by the term geosampling. The sampling of planetary materials for both geoanalysis and bioanalysis experiments is to be performed on geological materials, and therefore, by definition, the term geosampling is adequate to describe those functions and requirements necessary for the collection and preparation of solid planetary materials for the purposes of bioanalysis or geoanalysis.

VI. Summary and Conclusions

Sampling is of fundamental importance in planetary exploration because successful implementation of sampling techniques will strongly influence the course of planetary exploration by allowing conventional analytical methods and tools to be applied to fundamental scientific problems. More specifically, the availability of suitable sample acquisition devices limits the choice of scientific instruments that can be assigned to a spacecraft payload. The choice of instrument for a given measurement will be based, in large part, on the complexity of its sample requirements.

The principles and guidelines for the efficient design and development of sampling devices must be based on:

- (1) The probable biological and geological conditions to be encountered on the planet.
- (2) The priority of scientific measurements being considered for payload.

Guidelines must be flexible enough to meet changing demands as planetary exploration progresses, as scientific objectives receive clearer definition, and as knowledge of the sampling environments increases.

Sampling of planetary surface materials for analysis by instruments should be performed for answering specific scientific questions. Samples, to be scientifically significant, must be representative of the unit of planetary material that is sampled. This implies (1) intelligent selection of samples within the framework of specific scientific objectives, not random selection; and (2) proper acquisition and preservation of all pertinent qualities of the sample.

There are two modes of analysis available to some instruments that define the interface configuration between the analytical instrument and the material to be analyzed:

(1) Deployed instrument. Positioning of the instrument near undisturbed planetary surface material; this mode is applicable to only a few (mostly geological) experiments.

(2) Acquired sample. Surface or subsurface material is dislodged, acquired, processed, and transported to the analytical sensors; this mode is applicable to all biological and most geological experiments.

Each of these analysis modes may be performed at points on the surface directly below, immediately adjacent, or radially outward from the spacecraft body. Some scientific instruments can be designed for either, or both, analysis modes; others must inherently operate in only one of the two modes.

The deployed instrument mode is relatively simple, lightweight, low power, and more reliable, but has less scientific importance and versatility than the acquired sample mode. The acquired sample mode is relatively complex and heavy, requires more power, and is less reliable, but is scientifically more important because it allows more fundamental experiments to be conducted and is much more versatile than the deployed mode.

The choice of which analysis mode to employ for a given mission must be a compromise of the following considerations:

- (1) Inherent analysis requirements of the analytical instruments.
- (2) Availability of flightworthy instruments of a given analysis (mechanical) configuration.
- (3) Availability of suitable sample acquisition devices.
- (4) Mission and experimental objectives; i.e., whether surface, subsurface, or both types of materials are to be analyzed.
- (5) Weight and power limitations.

The choice of a suitable sampler for a given scientific payload can be made only after the following conditions are specified:

- (1) Mission objectives and, specifically, the objectives of the analytical experiment or experiments for which a sample is required.
- (2) Operational mode and sample requirements of the analytical instrument or instruments to be used.
- (3) Probable character of the planetary surface at the landing or sampling site.

(4) Spacecraft payload, power, and mobility limitations.

Planetary solids will be encountered as two types of geological materials:

- (1) Soil, which is a mantle of loose or cohesive particulate material, including fine dust and large blocks at the uppermost surface, and which may be of diverse origins.
- (2) Bedrock, which is solid rock indigenous to the solid body of the planet, and which, usually, is covered by a mantle of particulate soil of highly variable thickness.

Living biological material and its derivatives will be associated with soil particles at the uppermost planetary surface; its vestiges may be associated with bedrock.

For geological experiments, both soil and bedrock samples are important to the overall objective of determining the origin and history of planets and their relationship to the solar system. For biological experiments, soil is the more important sampling material.

Realistically, the choice of rock type to sample (soil or bedrock) depends on the rock type available at the spacecraft landing site; if only one rock type is present, then that type will be a significant sample to acquire. If both soil and bedrock are present and accessible, then both should be sampled and analyzed separately.

However, the capability to sample both soil and bedrock raises engineering problems because of the drastic difference in mechanical properties generally exhibited in bulk by the two rock types. Since engineering studies have shown that the same low-power, low-weight device cannot do both chores equally well, a compromise must be made: either plans must be made to sample for one rock type or the other, or two samplers must be included, one for bedrock, and one for soil.

The best approach in early missions is to have the capability of sampling what is most likely to be found. Since there are many arguments for a ubiquitous soil mantle, it would be best to plan primarily, if not exclusively, for this surface rock type.

Sampling of planetary surface materials in early planetary missions will be conducted for priority analytical measurements. These are inclusive of both geologic and biologic scientific objectives as follows:

- (1) Life detection.
 - (a) Biochemical compounds.
 - (b) Biochemical processes.
- (2) Volatile compounds.
- (3) Major element abundance.
- (4) Mineral phase abundance.
- (5) Soil and rock texture.

Therefore, the sampling requirements can be based exclusively on the requirements of these measurements and on the specific requirements of instruments that can perform these measurements.

The basic principles of biosampling and geosampling are identical. In addition, the detailed sample requirements for geoanalysis and bioanalysis experiments are practically identical. Because of this commonality, one set of standard parameters is sufficient for specifying the general sample requirements for all experiments, all instruments, and for both geological and biological objectives. These parameters are listed in Table 6. Sample volume is one exception. Biological experiments may require relatively large amounts of sample material for definitive scientific results, whereas geological experiments require relatively small, fixed amounts of sample. In addition, sampling devices for biological experiments must be aseptic, whereas, for geological experiments, the devices need not be.

Appendix

Surface Models and Performance Requirements for Laboratory Testing of Sampler Devices

A set of surface models and corresponding test materials are recommended as a tentative working standard for purposes of design, development, and testing of sample mechanisms which may be considered for planetary lander experiments. These models are representative of probable uppermost surface materials on Mars and on the moon. They are based on present knowledge and inferences as to the geological, biological, and meteorological processes that are active, or were once active, on these bodies.

The models are described in Table A-1. The order of listing of these models does not imply priority as to which is most likely to be encountered or which is most likely to contain biological material; each model may represent actual surface conditions existing locally at different points on the lunar and planetary surfaces, and each could conceivably contain biological materials. This is not an exhaustive list covering all possible surface types, but instead represents a range of most likely surface types to be encountered.

The models are intended for use only in studying and developing systems which are to be used for sampling the uppermost part of planetary surfaces. Layered or other complex models are not specifically designated. However, actual surface terrains may consist of any, or several, combinations of the representative materials; e.g., 2 in. of fine loose dust overlying rubble which, in turn, overlies solid bedrock. Another might be 10 m of volcanic ash overlying hard basalt, or vice versa.

Recommended test performance requirements and minimum sample requirements for samplers for both biological and geological experiments are listed below. These requirements are minimal and designed for comparing on a general basis the relative performance of

No.	Surface model	Approximate physical characteristics	Possible geological mode of formation	Examples of earth materials for test purposes
1	Cemented powder	Particles less than \sim 50 μ in size which are lightly to moderately cemented	Iron-oxide coated and cemented silt Sputter-cemented silt Frozen ground	Hardpan, adobe, dry lake bed Permafrost
2	Loose, slightly cohesive fine powder	Particles of size between 5 and 10 μ	Wind-blown and deposited particles	Silt or loess
3	Noncohesive, sorted sand	Particles between 100 and 500 μ	Wind-sorted particles	Sand; may include silicate; oxide, halide, carbonate, nitrate, and sulfate minerals
4	Rubble	Mixture of fragmented particles of all sizes less than \sim 10 cm	Impact-pulverized bedrock Volcanic ejecta	Crushed unsorted igneous rock, such as pulverized basalt (Little Lake, Pisgah crater)
5	Friable, porous rock	Lithified volcanic ash particles of all sizes less than ${\sim}4$ mm	Viscous volcanic magma comminuted by effervescence	Rhyolite tuff (Bishop tuff)
6	Solid bedrock	Massive crystalline rock, fine to medium grain size. May or may not be slightly to moderately vesicular	Surface lava flow Exposed subsurface intrusive rock	Basalt (Little Lake, Pisgah crater)

Table A-1. Surface models and equivalent test materials^a

various sampler devices or systems in the various probable planetary surface materials. More detailed sampling requirements (to be spelled out when the need arises) may have to be met for certain analytical experiments.

(1) Environmental test conditions.

Test	Pressure, torr	Gas	Temperature, °C
Lunar simulation	<10-6	vacuum	+120 ±10 and -100 ±10
Martian simulation	∼4 (5 mbar)	CO_2	+25 ±5 and -75 ±5

- (2) Performance requirements.
 - (a) Acquire particulate material from each of the specified surface models (Table A-1).
 - (b) Transport the material to a point of delivery for one or more analytical experiments.

- (c) Process the material before delivery only to the extent of excluding all particles larger than approximately 1000 μ .
- (d) Implace (pour, dump, etc.) the particulate sample into a 1-cm diam hopper, or into a 2- × 3-cm flat cup.
- (3) Individual sample requirements.
 - (a) Minimum sample amount (at delivery point):
 1 g (or 2 cm³ bulk powder).
 - (b) Particle size limits: $<1000 \mu$.
 - (c) Contamination: addition to the sample of less than 1% (by weight) of metals or other component materials of the sampler devices.
 - (d) Alteration: maximum temperature rise of bulk sample to be less than 20% of ambient test temperature (in °K).
 - (e) Particle-size and particle-density fractionation: no specific requirement, but such fractionation should be minimized during acquisition and transport stages of sampling.

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