NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Technical Report No. 32-853

JPL Spacecraft Sterilization Technology Program: A Status Report

Compiled by
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PREFACE

This document presents the technical status of JPL's spacecraft sterilization program, including hardware development, physical plant design and development, and sterilization technology research and development. Status reports of current tasks have been prepared by the cognizant engineers and scientists, and relevant material previously published by the Jet Propulsion Laboratory has been abstracted.

The document was compiled by D. Drummond and V. Magistrale, both from JPL's Environmental Requirements Section, Project Engineering Division. The compilers wish to thank the authors of the various papers appearing herein for their cooperation in providing the latest state-of-the-art information, the division representatives of the JPL Sterilization Committee for their valuable assistance in procuring the reports from their respective divisions, and the JPL Office of Research and Advanced Development for assistance in establishing complete coverage of the program.

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ABSTRACT

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This document presents the technical status of JPL's spacecraft sterilization program, including hardware development, physical plant design and development, and sterilization technology research and development.

I. INTRODUCTION

V. Magistrale

A previously published document (Ref. 1) presents NASA policy on spacecraft sterilization for planetary missions as follows: "NASA acknowledges the body of data and theory underlying the possibility of life and life-related molecules on the planets and the importance of scientific investigations of extraterrestrial life forms. It recognizes that the contamination of the planets with terrestrial microorganisms would pose a potential hazard to these scientific investigations, and it is also realized that more must be known concerning life forms of the planets before the potential effects of returning extraterrestrial matter to Earth can be evaluated. It is the policy of NASA to prevent the biological contamination of the planets until sufficient information concerning them has been obtained to ensure that biological studies will not be jeopardized and that no hazard to Earth exists."

Additionally, in the scientific community there is general agreement (Refs. 2–7) that the ecologies of the various planets should be preserved in order to permit biological observations of samples uncontaminated by organisms introduced from Earth.

According to a NASA requirement, the probability of landing a viable organism on Mars must be less than 1 in 10,000 (Ref. 7). The reasoning which led to this probability figure is reviewed in a JPL report by L. D. Jaffee (Ref. 5). An earlier, similar analysis was reported by R. W. Davies and M. G. Comuntzis (Ref. 2). The present interpretation of this requirement is that each landing spacecraft must be subjected to a sterilization treatment so that the probability of survival of any organism is less than 1 in 10,000. Mars flyby or orbiting spacecraft are also subject to sterilization unless their trajectories are so biased that the probability is less than 1 in 10,000 that either the spacecraft or its emissions (attitude control gas, waste propellant, etc.) could reach Mars.

The sterilization requirements apply to the total mission, including spacecraft, booster, and their emissions.

A. History of the Sterilization Program at JPL

The JPL sterilization program has been in existence for approximately 3½ years. The first spacecraft to be subjected to a sterilization requirement were those of the *Ranger* series. The *Ranger* sterilization experience provided the basis for current concepts of spacecraft sterilization technology.

Concurrent with the *Ranger* Project, and during the early phases of the *Surveyor* Project, the Hughes Aircraft Company carried out much spacecraft sterilization research and development.

As the *Ranger* project progressed, many waivers were granted for parts that could not withstand the sterilization environment; sterilization had not been a design requirement. Stringent *Ranger* and *Surveyor* sterilization requirements were voided in the last quarter of 1962.

Present NASA policy (Ref. 1) states that future unmanned lunar spacecraft will be decontaminated to "protect the moon from widespread or excessive contamination until sufficient information has been obtained concerning the moon to ensure that scientific studies will not be jeopardized."

The current effort to provide sterilizable hardware for a Mars lander spacecraft was initiated in FY 1963 with twelve tasks supported by the NASA Office of Lunar and Planetary Exploration and three tasks supported by the NASA Office of Bioscience. As stated by JPL to the Biosciences Subcommittee of the OSS–Space Sciences Steering Committee on September 12, 1963, the major objectives of the program were:

1. To begin the exploratory development of spacecraft components that will satisfy the sterilization and reliability demands of a planetary landing vehicle.

2. To begin exploratory research in the development of biological procedures and techniques that will determine the actual sterilization requirements which the hardware must fulfill.

Since its inception, the sterilization program has steadily expanded and now includes research and development efforts concerned with sterilizable component parts and spacecraft hardware, facilities for the handling of sterilizable spacecraft, and microbiological techniques necessary to monitor and certify sterility of the spacecraft before launch. The present status of these efforts is presented herein.

B. Engineering Sterilization Requirement

For a Mars mission requiring sterilization, the following general requirements have been specified by NASA (Ref. 1, p. 4):

1. The lander will be assembled in "clean rooms" at specified levels of assembly.

2. The landing assembly will be subjected to an approved sterilization procedure.

3. The landing assembly will be enclosed in a bacteriological barrier to maintain cleanliness and sterility. After decontamination, the enclosure will not be opened within any portion of the Earth's atmosphere which might recontaminate the landing assembly.

Neither the exact nature of the assembly clean room nor the level of assembly at which clean room operations are to start has yet been decided. The intent of clean assembly of a spacecraft is to lower the biological contamination load to such a level that terminal sterilization can be carried out with a high degree of sterilization reliability but without compromising the operational reliability of the spacecraft.

Implementation of the requirement is subject to information derived from JPL Project EASL (Experimental Assembly and Sterilization Laboratory), present research by JPL and NASA, and an analysis of the particular Martian mission to be performed.

In the development of sterilizable hardware, the JPL Research and Advanced Development Sterilization Program has used as guidelines the aforementioned general requirements and the following specifications:

> All hardware (including experiments) included in a planetary lander must be capable of withstanding both heat sterilization and ethylene oxide (ETO) exposure. These environments are defined in "Compatibility Test for Planetary Dry Heat Sterilization," JPL Environmental Test Specification XSO-30275-TST-A, April 1963, and "Ethylene Oxide Decontamination Requirements," JPL Environmental Test GMO-50198-ETS-A, December 1963. (See footnote 1, Section II.)

A recapitulation of these specifications is as follows:

1. Heat Sterilization:
   Type approval (TA) test (nonflight hardware): 145°C for 36 hr, 3 cycles.
   Flight acceptance (FA) test (to provide sterile equipment): 135°C for 24 hr, 1 cycle.

2. Ethylene Oxide (ETO) Decontamination[1]:
   Type approval (TA) test: 12% ethylene oxide and 88% Freon 12 (ETO-F12) environment for 24 hr at 24°C and 24 hr at 40°C for 2 cycles.

[1]In keeping with this requirement, ethylene oxide is considered a decontaminant and not a sterilant. The function of ETO is to reduce the microbial load.

These specifications were used for all of the procedure and component development described in this report.

To guarantee both mission success and certification of capsule sterility, the most probable sequence of sterilization will be:

1. To subject all subsystems to FA testing at 135°C for 24 hr as a first test in the FA sequence.
2. Prior to flight to provide for a completely assembled lander system terminal heat sterilization of 135°C for 24 hr.

This sequence requires that all flight equipment be assembled, tested, and transported under environmental conditions that keep the microbial loads to a minimum. Ethylene oxide decontamination may be used to reduce the load from surfaces.

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II. ELECTRONIC PARTS ENGINEERING AND RELIABILITY

W. H. Lockyear

The NASA sterilization policy requires that all Martian spacecraft activity be conducted in such a manner as to reduce the probability of contaminating Mars with viable Earth organisms to less than 1 in 10,000. Both the mission trajectory (that is, the possibility of impact) and the degree of spacecraft sterility must be considered. However, only the hardware element, particularly the relationship of the sterilization policy to electronic parts, is discussed here.

Although the necessary degree of spacecraft sterility can be achieved through thermal, chemical, or radiation techniques, the thermal method appears to be the most attractive possibility for sterilization of electronic parts. This is not to imply that radiation and chemical methods, particularly ethylene tetraoxide (ETO) gas, are not of interest. Indeed ETO/electronic-parts studies of significant proportions will soon be initiated by JPL. However, the major effort in the current electronic parts sterilization program at JPL has been concerned with heat sterilization.

A. Primary Objective

The primary objective of the electronic-parts sterilization program is to establish an approved list of heat-sterilizable electronic parts. It is the intent of this program to consider and study the effects of the thermal sterilization environment on electronic parts, specifically in relationship to the reliability of the devices. The sterilization environment has been accepted as a biologically established constraint. That is, a given temperature for a certain time duration is required to produce the required sterile conditions. Current requirements detail 135°C for 24 hr as a satisfactory sterilization environment for flight equipment. It should be understood that the equipment is in non-operational storage condition during this sterilization cycle. Application of the sterilization requirement to electronic parts during type approval testing results in the following increase in the demands: The parts shall be capable of meeting the operational reliability requirements after being subjected to three 36-hr periods of non-operational storage at 145°C. The necessity for proof and assurance that the electronic parts of interest will indeed meet type approval demands resulted in the Sterilization Parts Program, which has been designed to meet and fulfill a Laboratory need. It should be obvious, however, that the results of the effort will be applicable to all spacecraft operating within the given engineering constraints[1].

The sterilization program will produce a variety of documents describing techniques, processes, procedures, and test results. The primary objective will be contained in a summary document outlining electronic-parts sterilization candidates for spacecraft applications. This document will present, in tabular form, the part candidates, a brief description of each part, a listing of the specific part's supporting procurement document, and the status of the test program applicable to the part in question.

The entries in the sterilization parts document have a close direct relationship with the tabulation of parts in the JPL Preferred Parts List (PPL) in that candidates for the sterilization program must have a preferred rating. Therefore, a discussion of the major policies and philosophy supporting the PPL will clarify the intent of the sterilization parts list.

The PPL is a compilation of high-reliability electronic parts applicable to all JPL electronic spacecraft equipment. Listing of the items indicates that they are of interest to Laboratory design engineers. Rigid controls govern the addition and deletion of entries from the PPL. Parts are only removed when (1) actual test data indicates the failure of source process controls, (2) a qualified replacement part is available, (3) the detailed part specification is cancelled, or (4) the part design standard drawing is cancelled.

Parts are added to the PPL only after (1) a JPL need has been verified, (2) the part has been qualified to the

[1]For more than four years the laboratory has been studying sterilization effects resulting from 135°C heat applied for 24 hr. This procedure is in accordance with the acceptance, in 1963, of this temperature/time relationship as adequate for sterility of flight equipment by NASA. In July of 1965, NASA expanded the acceptable temperature/time relationship into a range beginning at 105°C for 336 hr, including 135°C for 24 hr (actually 22 hr) and extending to 160°C for 3 hr. As of this date, no official action has occurred to change the "sterilization requirement" from 135°C for 24 hr for flight gear. However, the Laboratory has given careful consideration to the results of a change to a temperature/time other than 135°C/24 hr.

Laboratory's rigorous qualification requirements, (3) appropriate support specifications have been prepared, and (4) the support documents have been cleared (accepted) by the manufacturer of the part.

It is the intent of the PPL to provide a list of appropriately qualified "high use" standardized items. It is not the purpose of the PPL to list all electronic parts employed on any given spacecraft. The number of part types listed in the preferred list are maintained at a minimum consistent with the nominal spacecraft demonstrated requirements and appropriate qualification information.

The PPL also includes parts reliability levels (i.e., Hi-Rel, Preferred, and OSE), definitions of reliability levels and their basic qualification and acceptance test requirements, and general de-rating consideration statements.

For several years JPL has stressed the importance of the PPL and has spent much time and effort in compiling it. Therefore, the sterilization parts list and the PPL are interrelated to the maximum degree possible to capitalize on previously gained experience. Indeed, a part is not generally considered a candidate for sterilization test studies unless it is listed in the PPL. Of course, the development of special "low usage" parts or new-state-of-the-art parts will necessarily modify this policy, but qualification will remain a major requirement in the parts sterilization effort.

The discussion of the sterilization parts list has related mainly to the thermal-sterilization environment. However, considerable attention is also being given to ETO thermal compatibility. The ETO studies, utilizing the available literature information on parts and materials as well as test effort as required, will seek to answer the basic question: Is ETO, when employed as a gaseous sterilant, less than fully compatible with all categories of electronic parts? Information produced by the ETO studies will be included in the sterilization parts list.

B. Secondary Objectives

The secondary objective of the parts sterilization program is to establish maximum information in the new era of higher operating temperatures and longevity of electronic parts. This includes the study of part wear-out phenomena, the study of de-rating in the matrix type tests, the study of the relationship of piece-part parametric ratings to the true capacity of the part, and the study of the overall reliability of parts over long periods of time. Special consideration is being given to the production of data in real time to serve as control or reference information for accelerated life studies. The secondary objective also includes the production of figures-of-merit information relating certain similar parts within a given parts category. The data is also expected to produce strong support for parts screening efforts. The failure-analysis portion of the program will result in product improvement through close cooperation with the parts manufacturer.

C. Parts Sterilization Program

At the start of the JPL parts sterilization program a basic philosophy was established. It included two major points: First, the program was designed to establish the previously discussed approved list of heat-sterilizable electronic parts together with appropriate assurance and/or proof that the itemized parts can indeed survive the sterilization environment. Second, the general reliability characteristics of the parts in question must be ascertained as they relate both to the sterilization environment under consideration and to the requirements relating to long operational life.

In cooperation with NASA sterilization policy and the established basic philosophy, the Laboratory is pursuing an extensive electronic parts program to fulfill these objectives. The current parts program, utilizing heat as the sterilant, was initiated in May 1963. However, a small capacitor sterilization investigation was conducted as early as March 1962. The earlier effort explored certain of the monitoring concepts employed in the current program.

Comparison of these early test programs with the present test effort forcefully illustrates the magnitude of the current undertaking. The early tests included about 500 capacitors made up of 15 part types. The present program involves 42,814 parts made up of 262 part types (see Table 1). The presently planned and implemented test phase will produce about 418,000,000 part-test hours of data. The testing will be completed in 1967 (excluding follow-on considerations). As of July 1965, approximately 70% of the part testing effort was implemented. In addition to the planned and implemented tests, certain follow-on tests are anticipated. These tests will involve new parts not available at the initiation of the given test. The follow-on tests will also include parts proposed as substitutes for unapproved devices (parts failing the test criteria). Certain tests will also be performed to provide

Table 1. Current electronic parts sterilization test categories

Part category	Number	Total number of parts
Capacitors	28	4,200
Resistors (fixed)	82	14,294
Resistors (variable)	24	2,640
Diodes (general)	36	3,856
Diodes (varactor)	4	384
Fuses	8	7,120
Thermistors	8	800
Transistors	32	3,200
Crystals	5	150
Relays	9	1,800
Microcircuits	7	700
Inductors	19	3,040
TOTAL	262	42,184

additional data and information of greater depth where special problems and difficulties exist or are discovered.

The major portion of the parts sterilization test program is being accomplished through contract with industry. Only the more complex state-of-the-art items remain at JPL as in-house work.

The scope of the parts sterilization program is further broadened by three other related studies: the previously discussed ethylene tetraoxide gas sterilant tests, the electronic-part packaging considerations, and the studies to produce appropriate sterilization screens. Since the ETO effort has been discussed, no further comment is needed here.

The packaging studies are being made to establish methods of handling sterile electronic parts from the end of the production line to component fabrication. As the result of manufacturing processes, many parts are sterile as they leave the production line. Adequate methods of handling, shipping, storing, and control must be established to maintain this sterility, or sterilization techniques must be needlessly reapplied. Since existing information indicates that the number of thermal sterilization cycles is limited, and that the effects of ETO are in question, every effort must be made to maintain sterility at source.

D. The Program Approach

In order to fully understand the parts sterilization program, some degree of comprehension of the test requirements and test approach is required. As will be recalled, the previously discussed program basic philosophy established two concepts: (1) the program is to produce electronic parts sterilization information, and (2) the program is to produce general parts reliability data.

To achieve success under the basic philosophy, the procedures must necessarily vary in certain minor points, depending on the specifics of the part in question. However, in general, the parts are subjected to the following procedure: The electronic parts to be tested are selected from the Preferred Parts List on the basis of anticipated usage. All manufacturers supplying parts for the sterilization test program are contacted prior to the procurement of test specimens. The intent of the program is carefully explained. The manufacturers are encouraged to modify, improve, screen or select the proposed test specimens in any manner seeming prudent to the manufacturer. The Laboratory requires only that similar parts be available for future procurement. Part procurement specifications are employed in the purchasing of the specimens to assist in uniformity and quality. Where feasible for a particular part type, products of different manufacturers are represented. In this way, a part type evidencing significant degradation can be compared among manufacturers. If the basic part type is susceptible to change, all parts of that type should evidence degradation regardless of manufacturer; otherwise changes may be due to a particular manufacturer's quality control or design. Each manufacturer's part type is normally divided into four groups. Group A is the control group. These parts are not temperature-cycled (simulating heat sterilization). Group B is temperature-cycled three times (nonoperational), first to 145°C for 36 hr in an inert atmosphere, and then to 25°C for 24 hr. Groups C and D are similarly temperature-cycled for six cycles. The method of simulating heat sterilization was selected so that it would be compatible with JPL Specification XSO-30275-TST-A (see Section I).

The four groups yield data in the following areas:

1. Group A is a control group (no sterilization environment is applied) and is compared with Groups B and C during the 10,000-hr life test.
2. Groups B and C are the heat-cycled groups. Three and six cycles are used in an effort to determine

the effects of a different number of heat cycles both initially and during the 10,000-hr life test.

3. Group D is stored at maximum rated temperature, nonoperational, for 10,000 hr; then the parts are life-tested for 250 hr at maximum rated temperature and voltage along with the Group C parts (which serve as a special control group). Group D is intended to simulate a mission in which specific equipment would be nonoperational during the flight and then turned on at or near flight destination.

Groups A, B, and C are subjected to a 10,000-hr dynamic life test, with parts operating at maximum rated electrical and temperature conditions, following the sterilization environment.

The accumulated data will be analyzed statistically at the 95% confidence level, using the statistics outlined in JPL Specification ZPP-2040-Gen A, "General Specification Computation and Submittal of Component-Test Statistics," and those specified in the test documents.

Basically, all of the measurements for a specific parameter within a group are combined, and the mean (average) and the variance (variability of the measurements) are computed after each test step measurement. The means and variances of the before and after temperature-cycling measurements for Groups B, C, and D are statistically compared in order to determine whether the temperature cycling had a significant effect on the measurements: that is, did the mean or average of the parameters shift or did the variability of the individual parameter measurements change? This same before-and-after comparison technique is used at the 100-, 250-, 1000-, 2000-, 4000-, 6000-, 8000-, and 10,000-hr life test steps to test for changes in parameter measurements. In addition, the Group A (control group) mean is compared with the applicable Group B and C mean at each of the life test steps in order to determine whether the temperature-cycled parts reacted during life tests in a way significantly different from the reaction of the parts that were not temperature-cycled. During life testing, catastrophic failures are recorded, and this information is used to determine failure data for the final analysis. Upon completion of life testing, the following additional statistics are computed:

1. The final analysis of the means and variances included within group comparisons and between group comparisons.

2. Graphs of the mean of the measurements for each parameter vs time during life test.

3. Exponential failure rate at 90% confidence for each group.

4. Failure rates. (A Weibull distribution will be used to indicate whether the failure rate is increasing, decreasing, or constant, or is a combination of the three failure modes.)

5. A comparison of the history of the previous parameter measurements of each failed part with the previous parameter measurements of the unfailed parts from the same group. From this information the feasibility of predicting failures from early nonconforming parameter measurements will be investigated.

6. A summary of the test program, including sterilization parts recommendations.

E. The Approach Philosophy

There are three major sections in every given test project: (1) a test design resulting from an hypothesis, (2) a test performed in accordance with the design, and (3) an appropriate analysis of both the specimen failures and the produced data. In addition to these general requirements, it is also prudent in most cases to provide a feedback loop from the data reduction/failure analysis portions of the test to the test design to improve and perfect the test method.

With these concepts in mind, the previously discussed elapsed-time test approach will be compared to the compacted-time or accelerated test approach. First a description of a nominal accelerated test approach is given. This plan, commonly referred to as the step stress test, consists of a method of subjecting a given sample of test specimens to a certain stress P_1 for a specific time interval t_1. At the end of the test interval, the stress is removed and the specimens are allowed to stabilize to standard test conditions. Parametric measurements are performed and recorded. The failed devices are removed from the test. The remaining sample specimens are then subjected to a higher stress P_2 for the selected time interval. Again the stress is removed and the specimens are allowed to stabilize to the standard test condition, the attendant parametric measurements again being performed and recorded. This sequence of events is continued until all specimens or some specified percentage thereof fail. Concurrently with the described test action utilizing the time interval t_1 other similar test efforts involving a number of selected test intervals, t_2, t_3, $\cdots$, t_n, are being carried out.

The data are assembled in graphical form. A possible presentation of this information is shown in Fig. 1, wherein a function of stress $f(S)$ is plotted against time. (The abscissa is normally a logarithmic expression.)

The elements of the distribution curves (examples $S_1, \cdots, S_6$) lying on any ordinate $t_1, t_2, \cdots, t_n$ are made up of failure data obtained at the various stresses resulting from $P_1, P_2, \cdots, P_n$. The slope intercept is drawn through any chosen point, say the median M, on each distribution curve. This intercept is extended by extrapolation to any chosen operating level, yielding a predicted failure rate time t_x at that operating level. Time t_x will usually be from 30 to 50 times above the longest test time interval involved in the actual test.

The step stress accelerated test is only one approach to achieve high stress loading in the interest of compacting test time. Another method employs high constant stress for defined time periods or until a given percentage of the test specimens fail. This method also allows a slope intercept to be drawn by extrapolation, relating measured failure distributions and a predicted failure rate at some grossly extended time. These methods should yield similar predicted values as end results.

The major advantage of the accelerated test is obviously one of time compaction during the specimen screening. The major disadvantages relate to (1) the questionable accuracy of the required extrapolation methods, (2) the unknown anomalies resulting from the assumption that the required temperature cycling associated with the parameter measurements is insignificant, (3) the assumption that the failure modes throughout the stress range are constant and similar to those modes encountered in practical application, and (4), in the case of step stress techniques, the assumption that prior steps do not affect subsequent test levels.

Comparison of the elapsed-time test vs the accelerated test time techniques centers around two major issues, time and acceptable proof. The accelerated approach has a tremendous time advantage. However, the accelerated approach does not provide a test-proven hypothesis. Therefore, an implemented program with the accelerated approach must in general depend upon other correlating elapsed-time information for substantiation of the produced accelerated data. The corollary of these statements indicates that the elapsed-time approach is slow, but the test will produce factual evidence in accordance with its test design. For these reasons, it is obvious that the elapsed-time approach can support the accelerated studies with solid basic information in order that the accelerated technique may be examined, compared, and improved. The reverse statement is not true, in that the accelerated approach does not necessarily produce baseline informa-

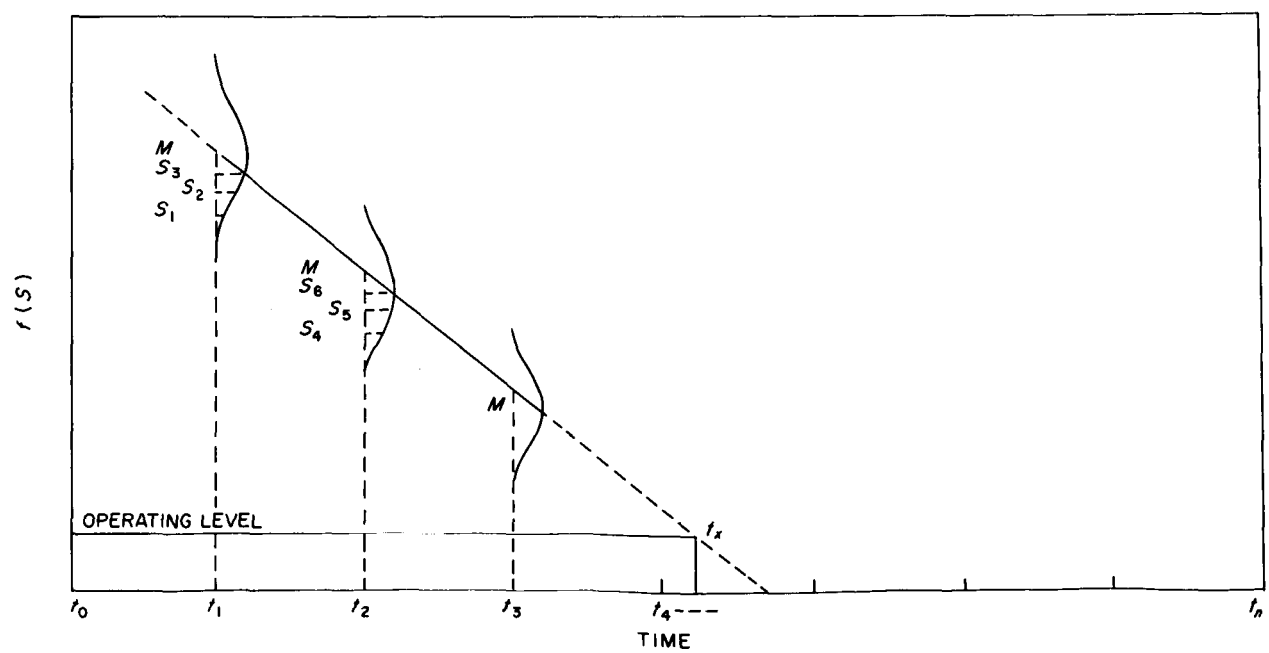


Fig. 1. Presentation of step stress information

tion. The elapsed-time approach has been employed to study the effects of sterilization. There are enough unknowns within the program without introducing problems through questionable measurement techniques.

These comments are not meant to imply that there is no interest in accelerated testing. Indeed, the advantages and disadvantages of accelerated testing techniques are being explored at the Laboratory. The analysis of failed test specimens is important information, as is the study of the produced test data; together they describe modes of failure. However, the most important aspect of the current work is the feedback of the failure mode information into the test design. Proof that the hypothesis is correct must be obtained prior to any extensive use of the technique.

F. Program Status (July 1965)

The parts sterilization test phase, using the Preferred Parts List as a selection criterion, is largely under way. Although this does not necessarily mean that testing has been started, action involving electrical tests is in process (Table 2). Testing of one type of part, the capacitor, is well advanced, 10,000 hr of testing having been completed. And it is appropriate here to examine briefly some of the general aspects of the capacitor test.

For this test program, 4200 capacitors, representing 28 distinct types of the following dielectrics were selected:

Ceramic (medium K style)

Porcelain

Glass

Mica (dipped style)

Solid tantalum

Foil tantalum

Paper/plastic

Mylar

In accordance with the general plan, prior to ordering the units for test, the purpose of the sterilization test program was explained to each capacitor manufacturer selected for evaluation. Therefore, each manufacturer had the same opportunity to submit "premium" capacitors with the best capabilities of meeting the objectives of the test program. In addition, the manufacturers were required to "burn in" each capacitor for a period of 250 hr at maximum rated dc voltage and temperature before shipment.

The 10,000-hr capacitor phase of the program has been completed. However, owing to certain wearout observations made in the later part of the 10,000-hr phase, a considerable portion of the earlier capacitor phase will continue on to 16,000 hr of test time. In addition, a follow-on test has been initiated to provide specific supplemental capacitor information.

As summarized below, the dielectrics most significantly affected by sterilization temperature cycling were the ceramic and solid tantalum types.

1. Ceramic Capacitors

The capacitance and dissipation factor of the ceramic dielectric increased as a result of sterilization. However, during the 10,000-hr life test at rated dc voltage and temperature, the capacitance dissipation factor decreased gradually and approximated the values observed before sterilization.

The temporary increase in capacitance and dissipation factor resulting from sterilization is probably acceptable, providing the circuit applications permit a maximum capacitance increase of 20% and a maximum dissipation factor value of 2.5% to 1 kc.

2. Solid Tantalum Capacitors

As a result of sterilization, the insulation resistance of certain test specimen groups decreased by a factor of 10. After 100 hr of life test at rated dc voltage and temperature, the insulation resistance of these specimens generally increased to the values observed prior to sterilization. However, at this same period of time, several catastrophic failures occurred in one of the test groups whose insulation resistance was affected by sterilization, whereas the first catastrophic failures of the test groups unaffected by sterilization occurred after 4000 hr. After 8000 hr of life test, the test groups affected by sterilization had only a slightly greater number of catastrophic failures.

Although this evidence is inconclusive, it would appear that solid tantalum capacitors whose insulation resistance is significantly decreased as a result of sterilization are more likely to become catastrophic failures in a shorter period of life when stressed at rated dc voltage and temperature. However, test groups whose insulation resistance was unaffected by sterilization produced catastrophic failures after 4000 hr and became progressively worse through 8000 hr, probably because of wearout caused by stressing at maximum rated dc

Table 2. Electronic parts sterilization program schedule (as of July 1965)

Test		1965									1966											1967												Completion
Type	Number	A	M	J	J	A	S	O	N	D	J	F	M	A	M	J	J	A	S	O	N	D	J	F	M	A	M	J	J	A	S	O	N	D	
Capacitors	ZPP-2101-Gen	5			3	← Follow-on test →				5																								December 1965
Fixed resistors	ZPP-2102-Gen				4	← 9,950 hr →		5																										October 1965
Trimming resistors	ZPP-2103-Gen				4	← 8,300 hr →				5																								January 1966
Varactor diodes	ZPP-2105-Gen				4	←				7,120 hr							5																	August 1966
General diodes	ZPP-2104-Gen		1		4	2				3																	5							May 1967
Transistors	ZPP-2113-Gen			2	4	3																5												December 1966
Microcircuits	ZPP-2121-Gen			3	4	←				1,000 hr											5													November 1966
Fuses	ZPP-2107-Gen					1			2		3																	5						May 1967
Crystals	ZPP-2116-Gen							1			2		3			5				1			2		3									August 1968
Relays	ZPP-2119-Gen					1			2		3																		5					July 1967
Magnetics	ZPP-2124-Gen					1			2		3																		5					July 1967
Thermistors	ZPP-2110-Gen					1		2		3																		5						June 1967
Capacitor follow-on	ZPP-2108-Gen					1		2		3																		5						June 1967
Temperature gradient	ZPP-2126-Gen						1				5																							December 1966

1 Start of test.

2 Sterilization and environment, start.

3 Life testing, start.

4 Present status (July 1965).

5 Complete.

voltage and temperature. In an attempt to reduce the number of catastrophic failures and to isolate the effect of sterilization from normal wearout, a retest of solid tantalum has been initiated using a voltage/temperature type of matrix.

G. Conclusions

The early information obtained from the first phase of the capacitor test coupled with the initial data returns from certain other operating test categories can be utilized to support several interesting observations as early conclusions. First, the technical data produced will certainly satisfy the prime objective of the effort, that is, to produce a list of approved sterilizable parts. In order to preclude obsolescence, the sterilization list and its supporting test endeavor must be a continuing effort, capable of accepting, within certain restrictions, new or improved parts. This is particularly significant as related to state-of-the-art devices, including semiconductors and microcircuits. Second, special attention must be given to maintaining the identity of test specimens. It is certainly desirable for product improvement by the manufacturer to up-grade a given part. However, changes in materials and processes may totally negate existing sterilization data. This is a particularly serious problem because of the long test time required to produce the longevity information. Proper and complete identification will help preclude this loss, and JPL will soon initiate a rigorous parts identification program to meet this need.

The third observation relates to the distortion of information due to the presence of multiple degradation factors. A prime example of this problem can be observed when the information produced by the sterilization environment is compared with the data resulting from the long-life portion of the studies at elevated (rated) temperatures. The degradation factors occurring as a result of sterilization tend to become obscure with time during the life test. This is an undesirable situation. However, the total value of the test is not materially distorted even with the loss in identity of the specific degradation. The rationale for this statement can be understood if it is remembered that the intent is to produce information about approved parts. One of the major requirements for approval is that the part be capable of demonstrating reliable, long-life characteristics.

A fourth general observation indicates that data relating to secondary objectives may well be so copious as to partially overshadow the primary objective results. Early indications point to the potential ability to produce wearout data in certain parts, the relationship of true part ability to its rating information, derating data, and figure of merit information as types of information envisioned from the tests.

ABSTRACT

"Component Parts Sterilization," *Space Programs Summary No. 37-17, Vol. II,* Part III-E-2a, Jet Propulsion Laboratory, Pasadena, California (Confidential).
(Classified material not abstracted)

III. FACILITIES

Abe Cohen

A. Project EASL

1. Functional Objectives

An Experimental Assembly and Sterilization Laboratory (EASL) was conceived in February 1965 and completed in June 1965 to meet urgent project requirements for a small facility in which the procedures and operations proposed for the assembly of sterilizable spacecraft could be practiced and developed.

The laboratory embodies all the elements of an actual assembly facility in order to permit conformance with anticipated procedures and to confront all concerned personnel with specific problems in realistic day-to-day operations leading to the ultimate development of sterile spacecraft.

Specific objectives of this facility include laboratory investigations and operations to determine:

1. The effects on spacecraft design which are introduced by the bioclean requirements.
2. Problems in the hardware/personnel/bioclean interfaces and those which may result from the introduction of microbiological technicians into the operating area.
3. Bioclean facility restraints on the performance of required hardware tests.
4. Requirements and techniques for microbiological sampling in the spacecraft assembly and test operations.
5. Effects on, and requirements for, special tools, fixtures, jigs, and test equipment.
6. General "time and motion" implications in all operations conducted under the bioclean restrictions.
7. Conditions, processes, and operations which should be strengthened, modified, relaxed, etc., including the existing bioclean environmental requirements themselves.

In substance, while the concept of spacecraft subsystem and system assembly and test for a nonsterile spacecraft is well understood and documented, the bioclean requirements for sterile spacecraft operations introduce a new element which must be fully evaluated from start to finish of spacecraft assembly and test.

2. Facility Guidelines

Primary guidance for both the design and operation of the proposed laboratory is set forth in a NASA document entitled "Interim Requirements for Bioclean Facilities." Various portions of the following directives, specifications, and standards also apply to the facility design:

1. *Clean Room and Work Station Requirements, Controlled Environment,* Federal Standard No. 209, Government Printing Office, Washington, D.C., December 16, 1963.
2. *Standards and Guidelines for the Design and Operation of Clean Rooms and Clean Work Stations,* Technical Order 00-25-203, U.S. Air Force, Washington, D.C., July 1, 1963.
3. "NASA Unmanned Spacecraft Decontamination Policy," *NASA Management Manual,* Chapter 4, Washington, D.C., September 9, 1963.
4. "Environmental Test Specification Compatibility Tests for Ethylene Oxide Decontamination Requirements," Specification GMO-50198-ETS, Jet Propulsion Laboratory, Pasadena, California, December 12, 1963.
5. "Environmental Test Specification Compatibility Test for Planetary Dry Heat Sterilization Requirement," Specification XSO-30275-TST-A, Jet Propulsion Laboratory, Pasadena, California, May 24, 1963.
6. *Manual of Design Criteria and Construction Standards,* NASA Facilities Publication NPC 325-1, NASA, Washington, D.C.

3. Project Scope

The scope of the proposed project is illustrated in Fig. 1. In actual operation of the laboratory, technicians are required to construct spacecraft parts/subassemblies under the rigorous constraints imposed by the bioclean working environment. Special personnel restrictions with respect to hygienic practices, clothing, and bioclean procedures and techniques also apply.

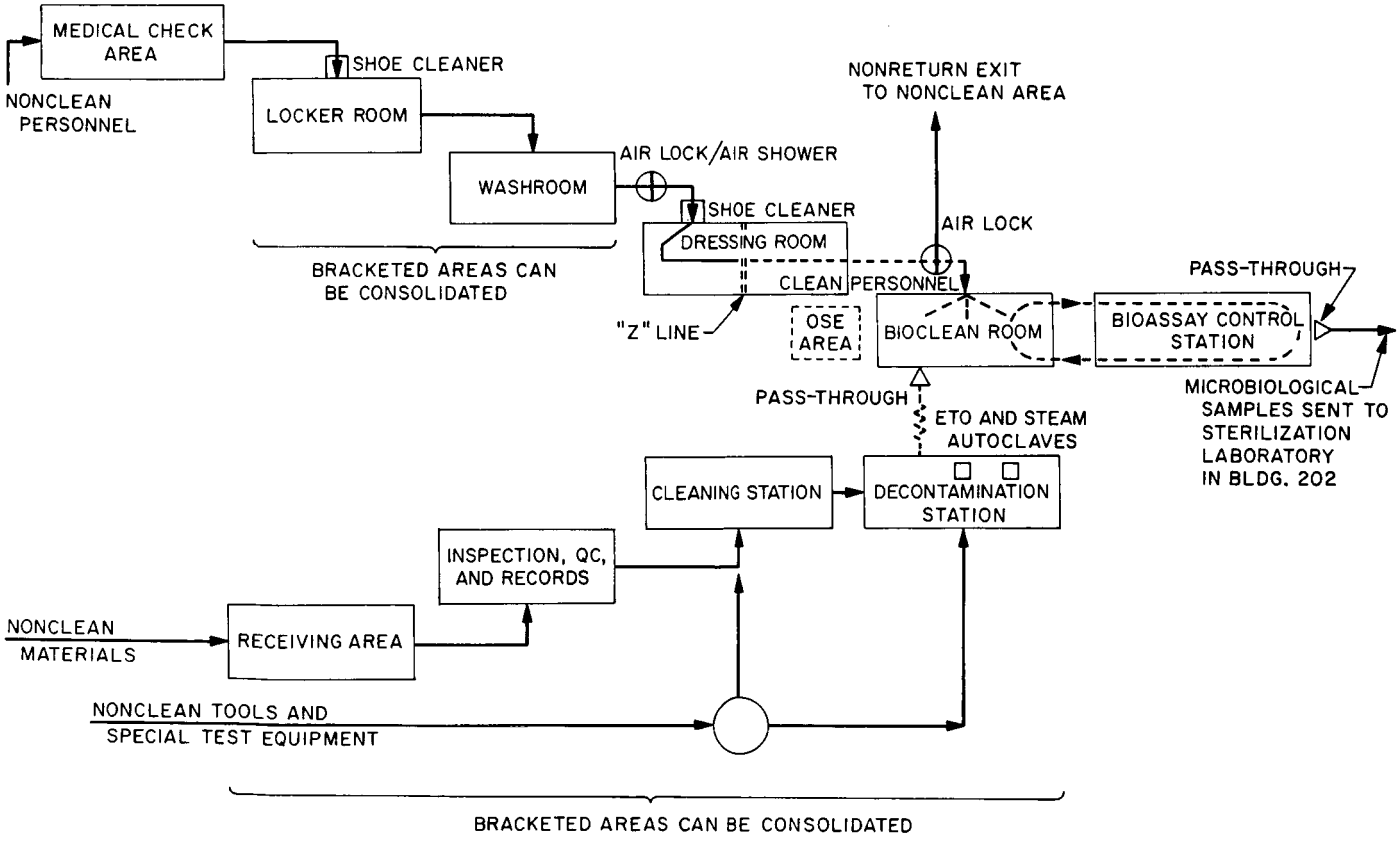


Fig. 1. Experimental assembly and sterilization laboratory (EASL) flow diagram

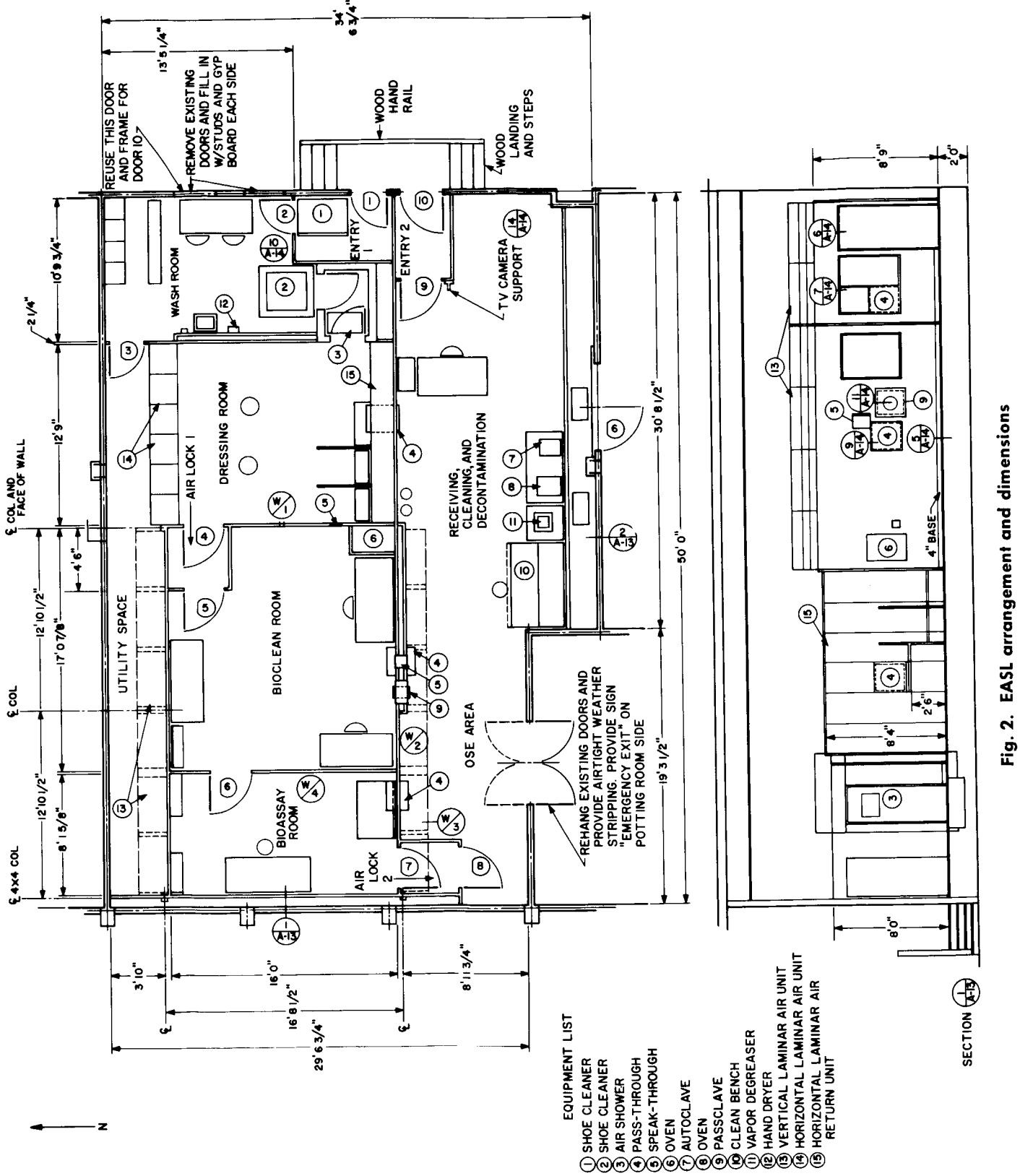


Fig. 2. EASL arrangement and dimensions

4. Description

The EASL floor plan, dimensions and general arrangement are shown in Fig. 2. Figures 3 through 9 illustrate actual features of the facility.

5. Project Results

Checkout and certification operations conducted in the facility during June 1965 demonstrated that the essential criteria of NASA's "Interim Requirements for Bioclean Facilities" can be met.

Particulate counts indicate that the clean rooms are at least one order of magnitude cleaner than class 100 as specified in Federal Standard No. 209. Similarly, microbiological assays of the dormant facility demonstrate negligible microbial contamination in the clean room environments. Ultimate performance of the facility will be a function of the degree of control exercised on (1) operating personnel, (2) materials, tools, and test equipment, and (3) the processes undertaken in the various rooms of the facility.

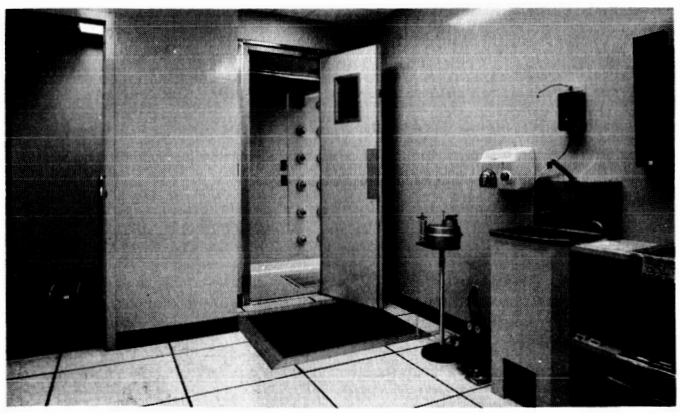


Fig. 3. Locker and washroom area

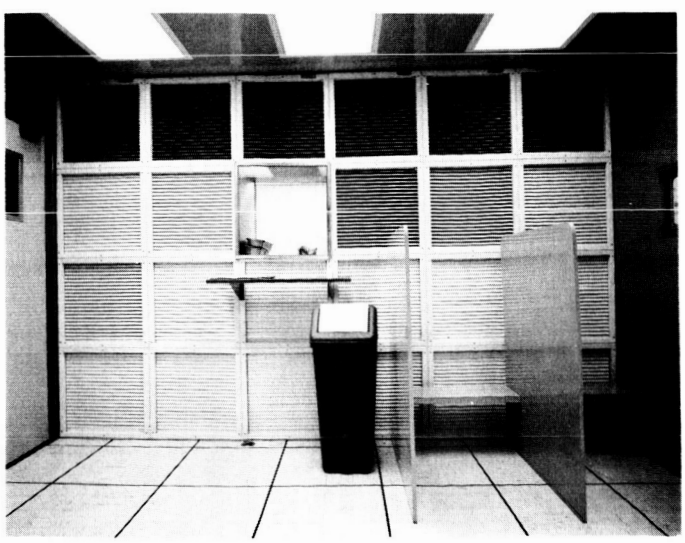


Fig. 4. Dressing room (semiclean side)

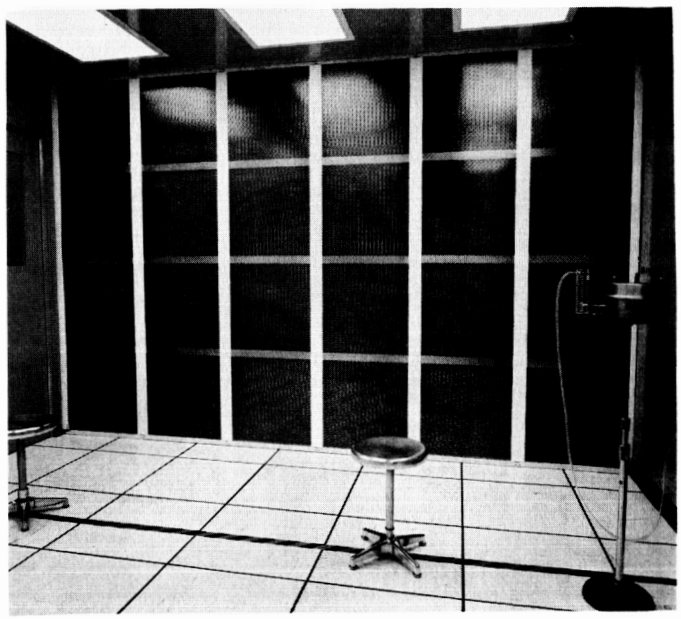


Fig. 5. Dressing room (clean side)

Fig. 6. View of bioclean room (from dressing room)

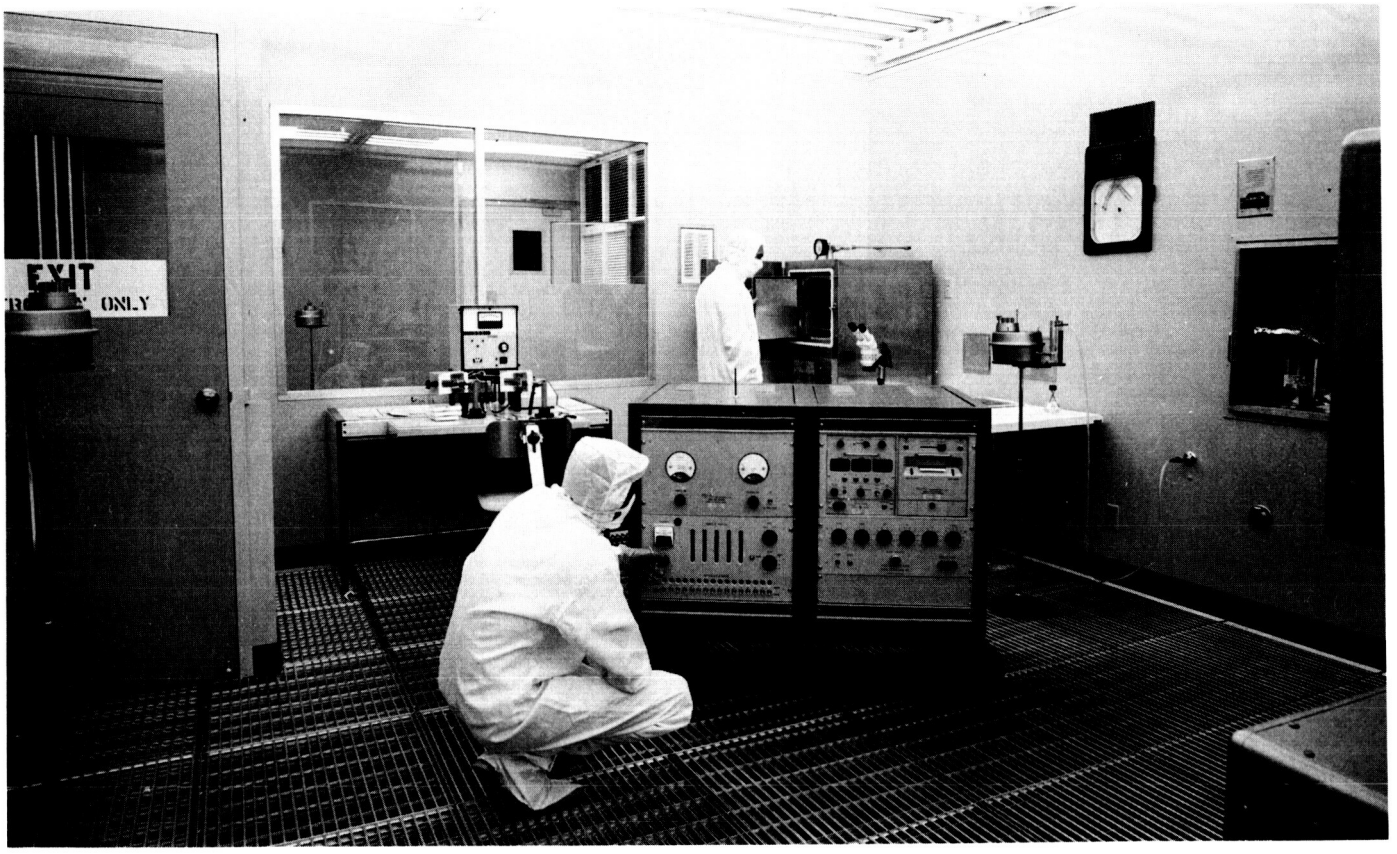


Fig. 7. Particulate monitoring operation in bioclean room

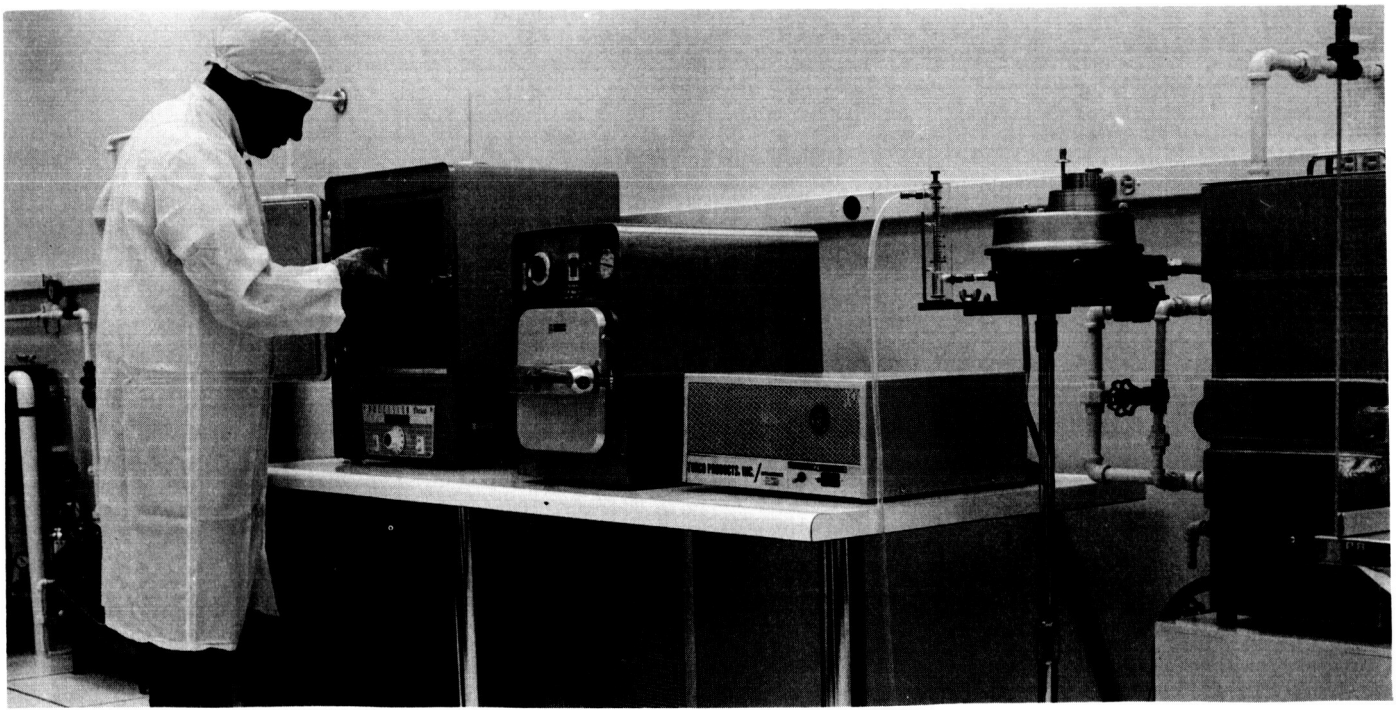


Fig. 8. Decontamination operation in receiving area

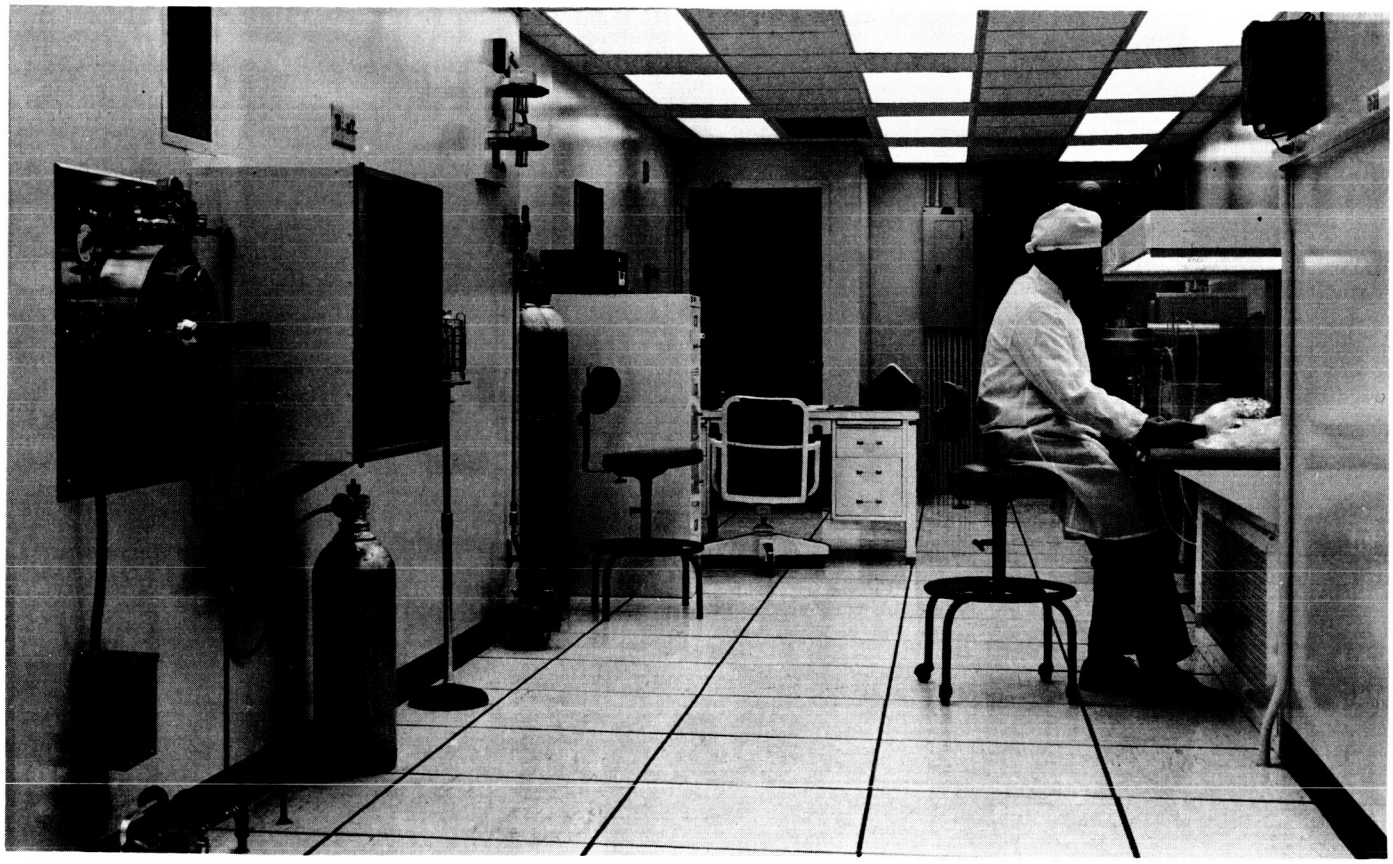


Fig. 9. General view of OSE and receiving, cleaning, and decontamination areas

B. Assembly Test and Sterilization Facility (AT&SF)

In the spring of 1963, JPL initiated studies to determine the basic feasibility of designing and constructing a facility for the assembly, test, and sterilization of spacecraft capsules which might have a significant probability of entering the atmosphere of a planet. As NASA policy for the decontamination and sterilization of unmanned planetary landers evolved and project planning for the *Mariner B* program developed, JPL contracted with the architectural and engineering (A&E) firm of Daniel, Mann, Johnson and Mendenhall to study and develop facility criteria. The architectural and engineering efforts were supplemented by the consulting services of Prof. George S. Michaelson, Director, Environmental Health and Safety, University Health Service, University of Minnesota, and Mr. Harold W. Wolf, Senior Sanitation Engineer, U.S. Public Health Service. These criteria development studies undertaken November 4, 1963, were paced to conform with *Mariner* Project development and published[1] after completion on April 2, 1964.

1. Design Concept

In facility design, the basic concern of the A&E is to achieve a low level of microbiological contamination or to reduce the existing level of microbiological contamination throughout all stages of capsule assembly and test so that the total microbial load exposed to the terminal heat cycle is within the kill capabilities of that cycle. Conceptual design was therefore predicated upon developing technically feasible and economical approaches to the control of microbial contamination in capsule assembly and test environment, operating personnel, tools, test equipment, and all materials and processes.

[1] "Criteria Development Studies for an Assembly, Test and Sterilization Facility," Daniel, Mann, Johnson and Mendenhall, Los Angeles, Calif.

Table 1. Summary of comparative equipment performance

Method	Effectiveness	First cost	Application	Reliability	Operating cost
Air filtration	2	2	1	1	1
Ionization	5	5	5	5	4
Gamma radiation	4	4	4	4	4
Ultraviolet	4	4	4	4	4
Electrostatic precipitation	3	3	5	3	4
Chemical air washers	2	2	2	3	3
Air washers	3	1	1	2	2
Air incineration	1	5	5	1	5

2. Control of Capsule Environment

Various methods of reducing or controlling microbial contamination in the capsule assembly and test environment were investigated. Results of these investigations are presented in Table 1. The analyses indicated that a combination system of air filtration and air washing offers the optimum approach. Air filtration in combination with chemical air washing is also effective although the costs are somewhat higher.

3. Horizontal vs Vertical Laminar Air Flow

The merits of vertical and horizontal laminar air flow in the air filtration concept were investigated to determine their relative effectiveness in this application. The horizontal approach offers some economy in first cost and a better solution to the lighting problems; the vertical laminar air flow approach offers a less contaminated room. The decision in favor of vertical flow is also influenced by the nature of the work to be performed and the anticipated fabrication processes.

4. Analog Studies

The selection of vertical laminar flow was not confirmed until the spatial requirements invoked by the potential work flow and area layouts were examined. Two-dimensional analog studies were conducted to evaluate the equipment and man/machine interfaces which might inflict serious cost penalties on the use of laminar flow or even demonstrate the impracticability of establishing and maintaining the laminar flow.

Figures 10 to 14 illustrate five configurations which can be considered representative of the interfaces which may be encountered in the Assembly Test and Sterilization Facility. The studies demonstrate the following summarized findings:

1. Vertical laminar flow can be obtained (or recaptured) approximately 5 ft below the ceiling level using a light module about 2 ft wide.
2. Eddies can be expected near and slightly below the bottom edge of the capsule, where it rests on the assembly pad.
3. If the assembly pads are spaced no closer than 6 to 8 ft from each other, no special problems in turbulence are interjected into the design considerations.

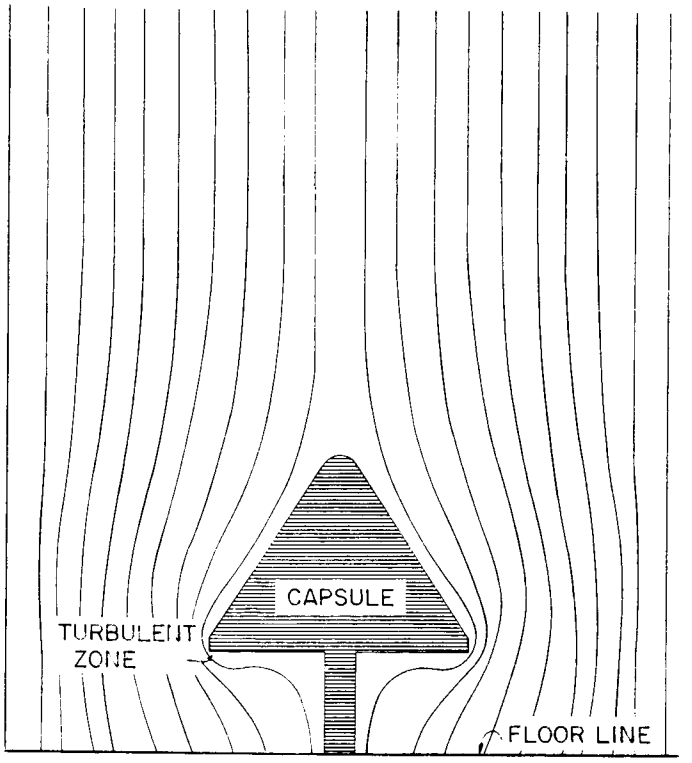


Fig. 10. Analog study: isolated capsule

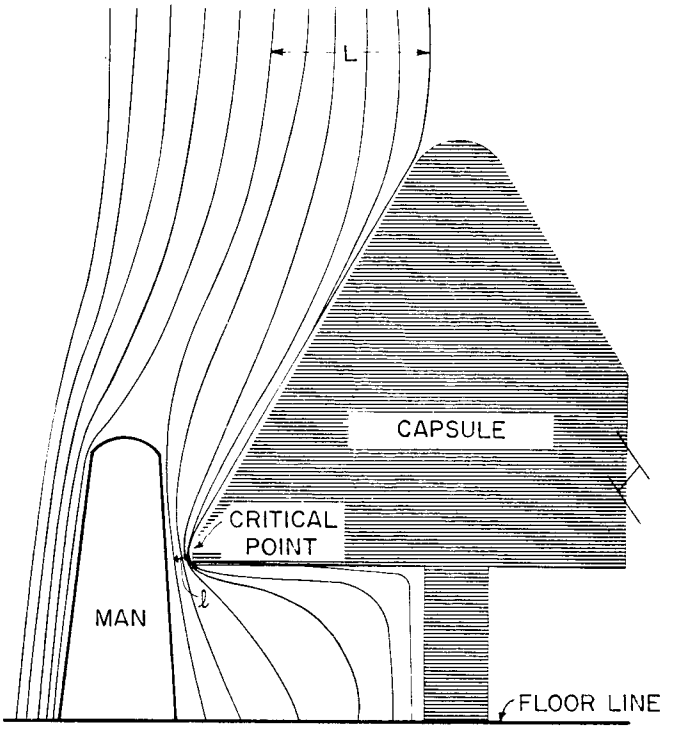


Fig. 12. Analog study: man/capsule

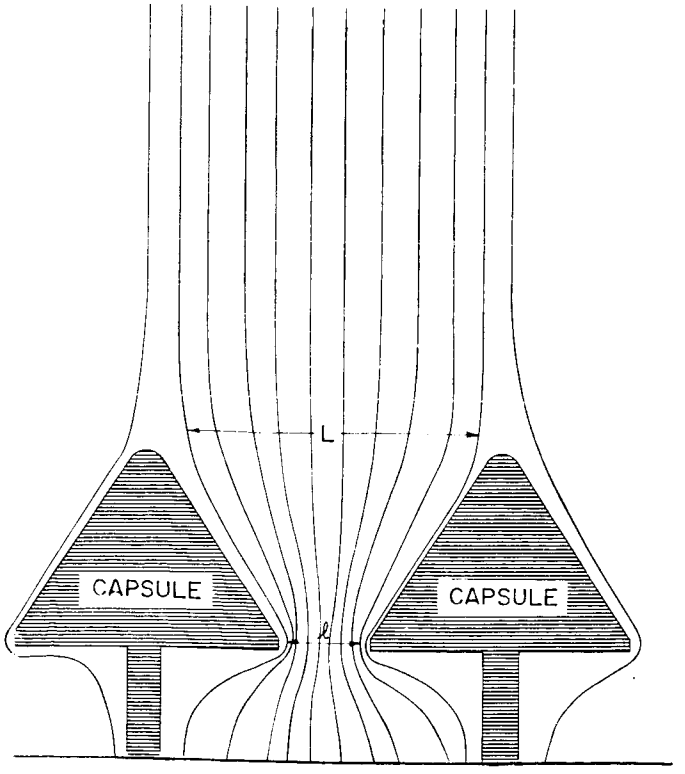


Fig. 11. Analog study: capsule/capsule

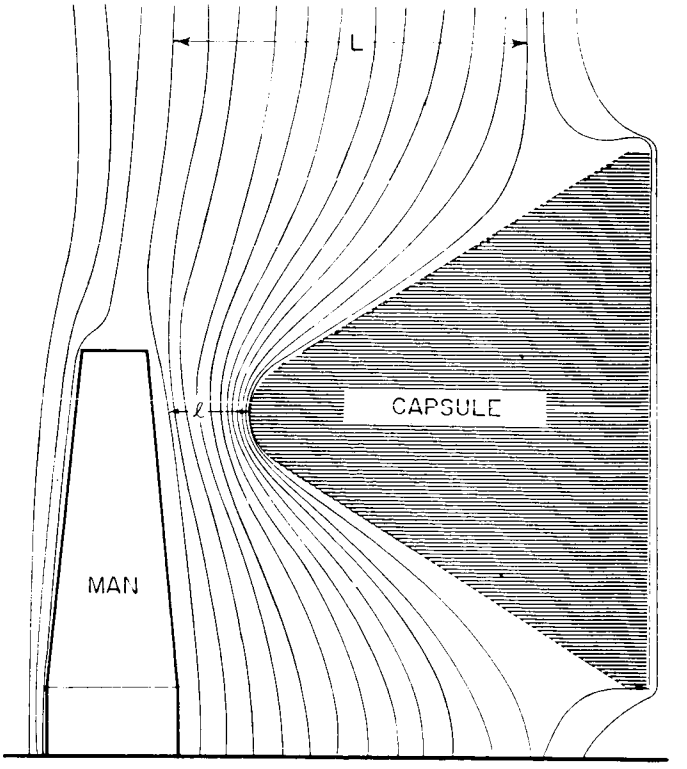


Fig. 13. Analog study: horizontal capsule/man

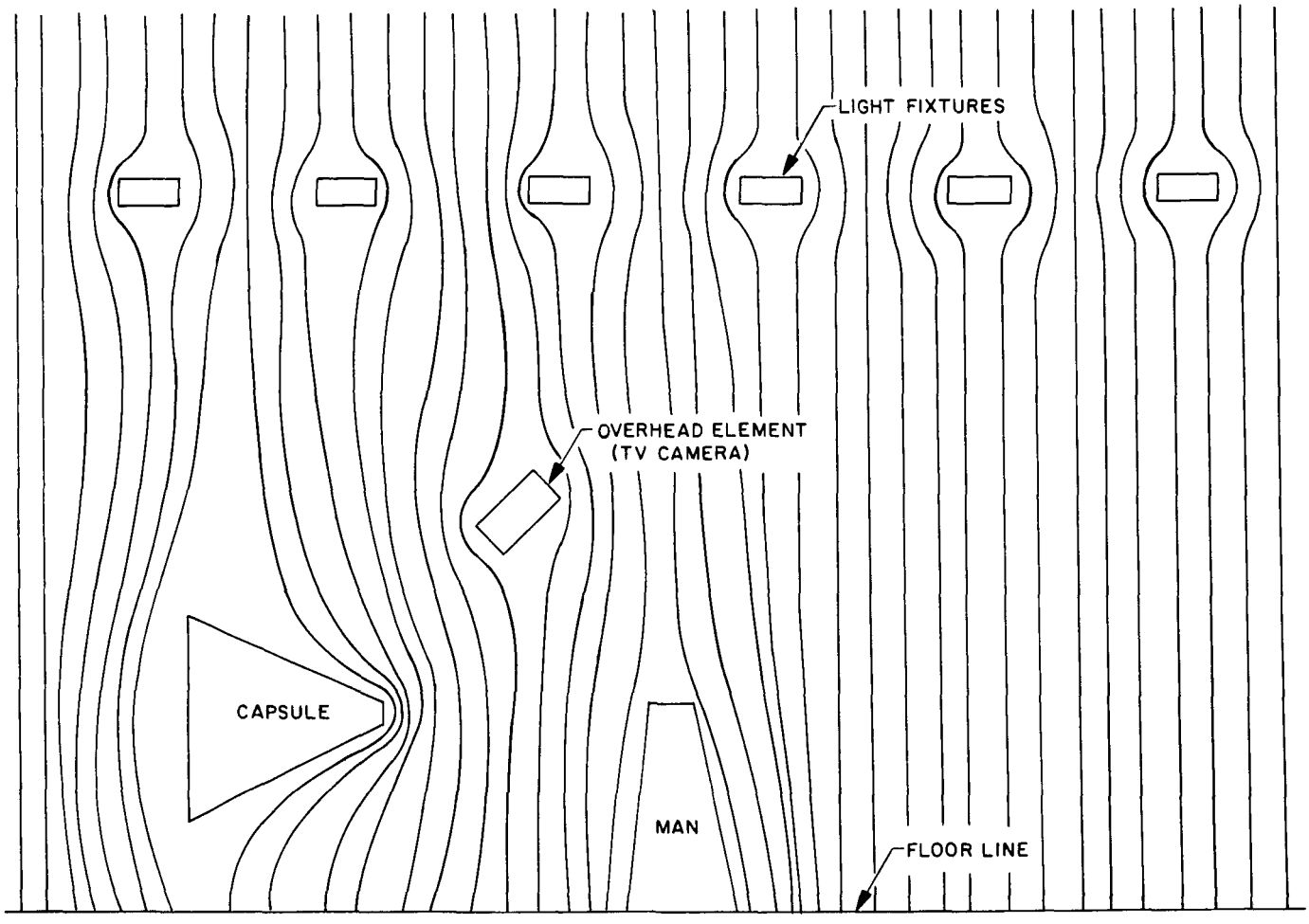


Fig. 14. Analog study: light fixtures

4. The interface between the technician and the capsule will result in excessive turbulence and loss of laminar flow above the bottom edge of the capsule as the distance between the two is decreased. The critical distance here is on the order of 1½ ft. If the technician performs his operations from a point beyond this distance, little interference can be expected.
5. In considering the possibility of tilting the assembly pad to facilitate certain capsule assembly operations, the previous problem is accentuated by the configuration of the capsule itself. Excessive turbulence and loss of laminar flow will occur.

The studies also indicated that isolated instances of turbulence (such as those encountered at the base of the capsule) can be controlled by means of fairing devices. Similarly, adjustments in the work habits of the technicians can prevent the build-up of turbulence around a capsule throughout the assembly process.

From the standpoint of facility design the primary requirement is to assure a basic approach that will neither interpose costly spatial requirements (vertically or horizontally) nor prevent operating personnel from performing their duties in an efficient manner.

5. Integrated Modular Design

A ceiling module integrating the mechanical, lighting, and structural requirements would offer several advantages, particularly in reduced construction costs and assurance of quality. The seal problem surrounding the superinterception filters is a special concern. Figures 15 to 19 illustrate an integrated modular design concept which resolves most of the problems. Any concept should recognize all the problems: namely, lighting needs, structural loads, laminar flow requirements, maintainability, accessibility, costs, problems in field fabrication and installation, and quality control.

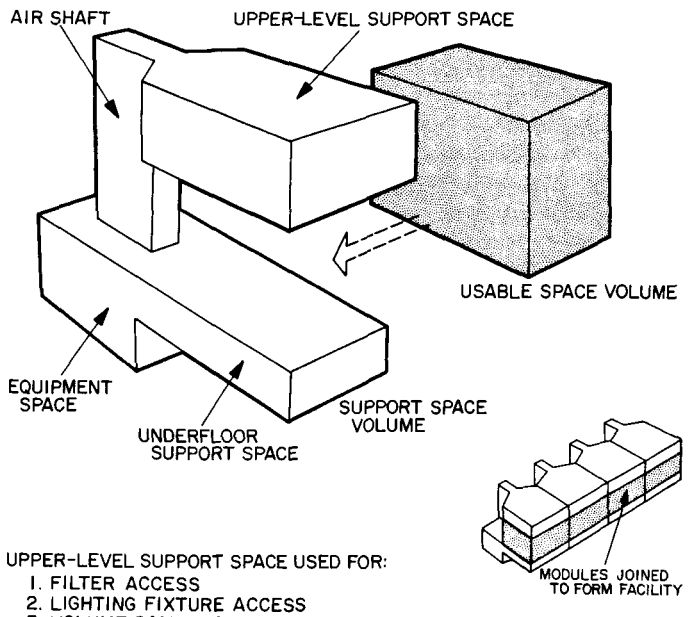


Fig. 15. Laminar downflow module: volume relationships

6. Process Control

The facility design problem in the control of all processes is primarily a resolution of the human factors in the fabrication and test operations. In general, a good facility design approach means that adequate space and proper equipment are provided for the anticipated operations, and that flexibility, versatility, and growth are built into the design at the very start. The optimization of these areas was obtained by means of a formalized functional analysis of all operations. The functional analysis was performed on a reiterative basis as criteria were developed and implemented.

7. Material Control

Since numerous vendors will supply the various capsule components and subassemblies which will be assembled and tested in the AT&SF, positive means of contamination control must be considered from the time material is received. Particular attention must be given to ensure that all materials, including tools and test equipment, are properly received, documented, and decontaminated before entering the clean areas of the facility. Essentially this means rigorous procedural controls, interlocks, and well-planned layout and equipage of supporting as well as primary work areas. Basic layouts and material flow patterns which meet the criteria are illustrated in Figs. 20, 21, and 22. The decontamination arrangement is shown in Fig. 23.

8. Personnel Control

Personnel are a major source of particle contamination as well as microbiological contamination. Data indicate that the average person sheds 3.5 viable particles per minute. The data in this area may be refined as results from the U.S. Public Health Service micro-bio-tank experiments at the Communicable Disease Center in Savannah become available. The microbial contamination shed by the individual represents, at present, the greatest problem to the facility designer.

Solutions to personnel contamination problems are many and varied. Initially, extensive consideration should be given to reducing the manpower requirements in the basic assembly and test procedures. However, there is a practical limitation because of the very nature of the work, which is not a high-volume repetitive assembly line effort but is more properly classified as special crafting operations by highly skilled technicians. As indicated earlier, formal functional analyses with emphasis on the human factors can assist greatly in obtaining a true definition of manpower requirements.

Subsequent personnel control efforts may include the following:

1. Preselection among technically qualified personnel.
2. Medical examination and daily health surveillance.
3. Introduction of adjunctive personnel hygienic practices (such as use of special soaps).
4. Reduction of personnel traffic by means of:
   a. Optimum facility layout, as illustrated in Fig. 24.
   b. Adequate and flexible communications.
   c. Good visibility between and among operating areas.
5. Indoctrination and training in work practices and procedures.
6. Close supervision and monitoring of procedures by:
   a. Film cameras.
   b. TV cameras.
   c. Visual observation.
7. Proper dress of all personnel.

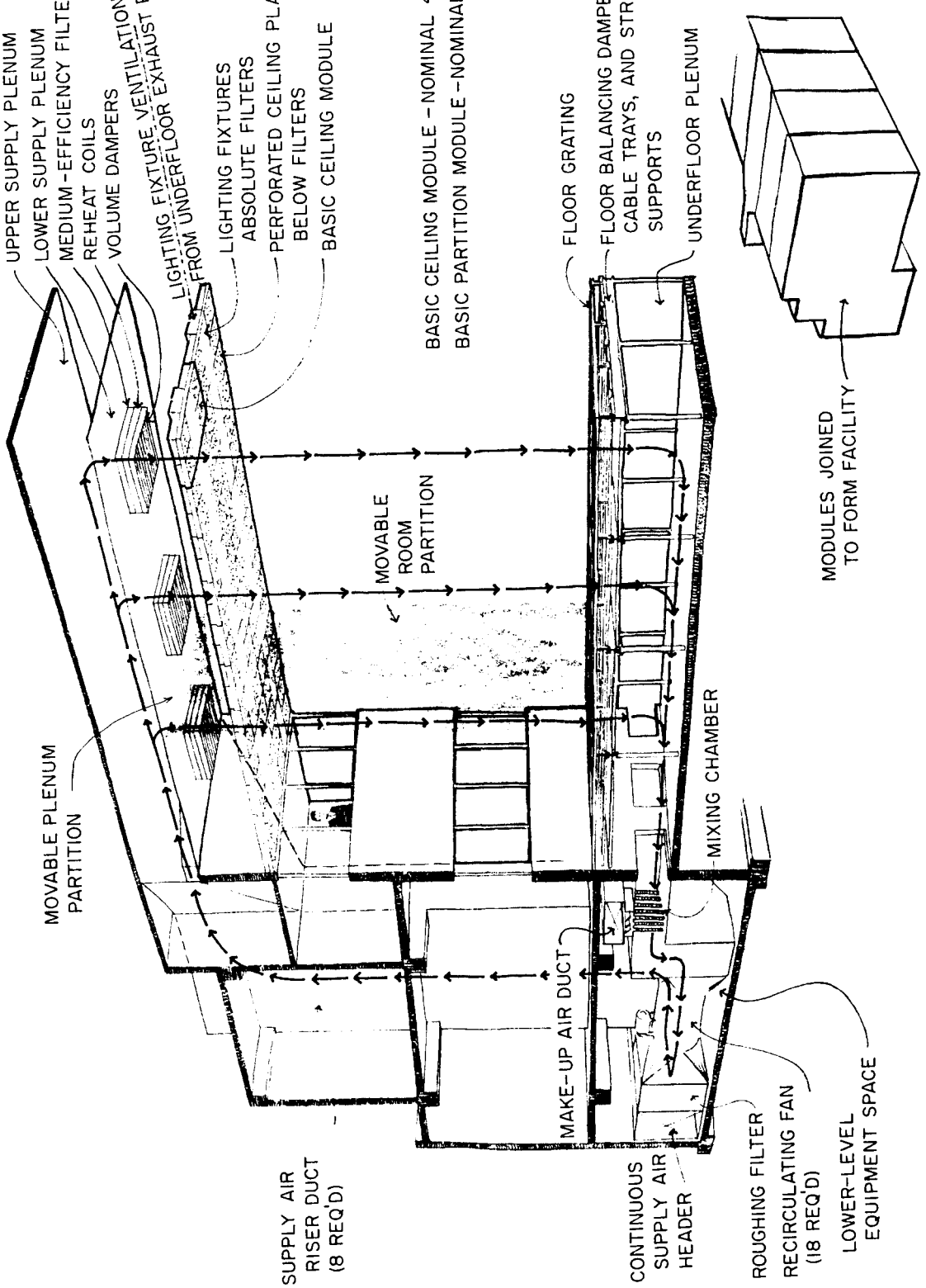


Fig. 16. Laminar downflow module

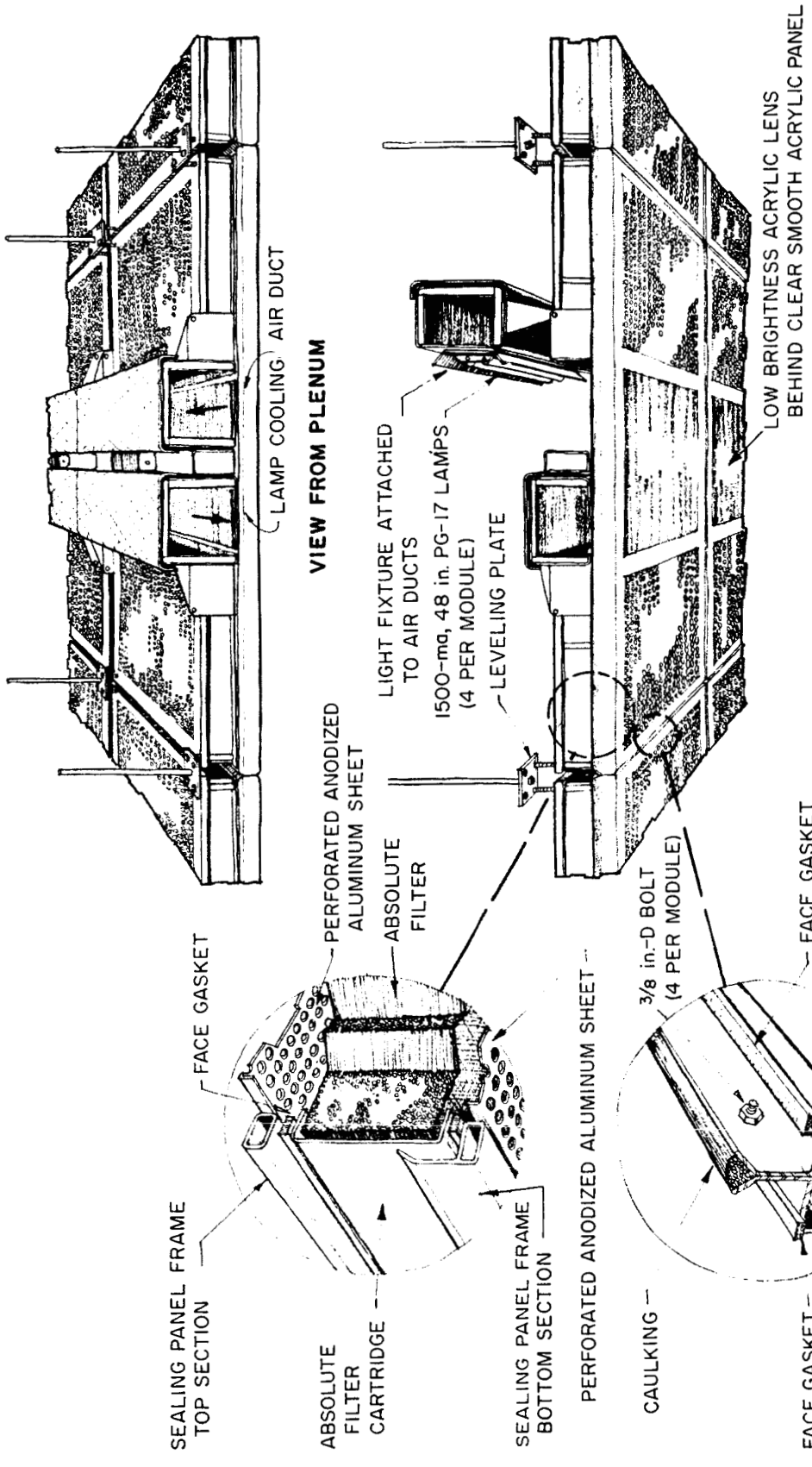


Fig. 17. Integrated lighting: absolute filter ceiling module

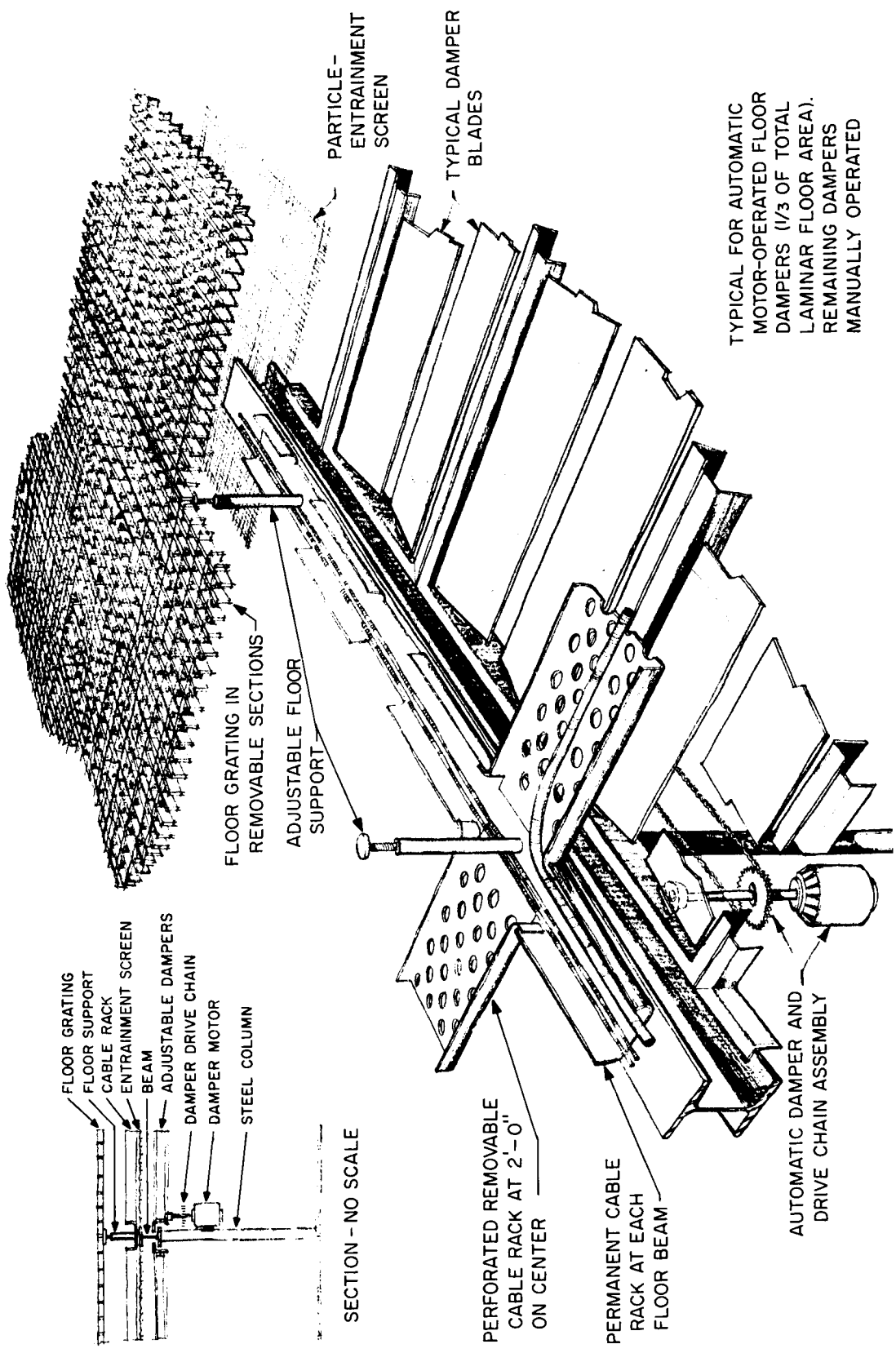


Fig. 18. Laminar floor assembly details

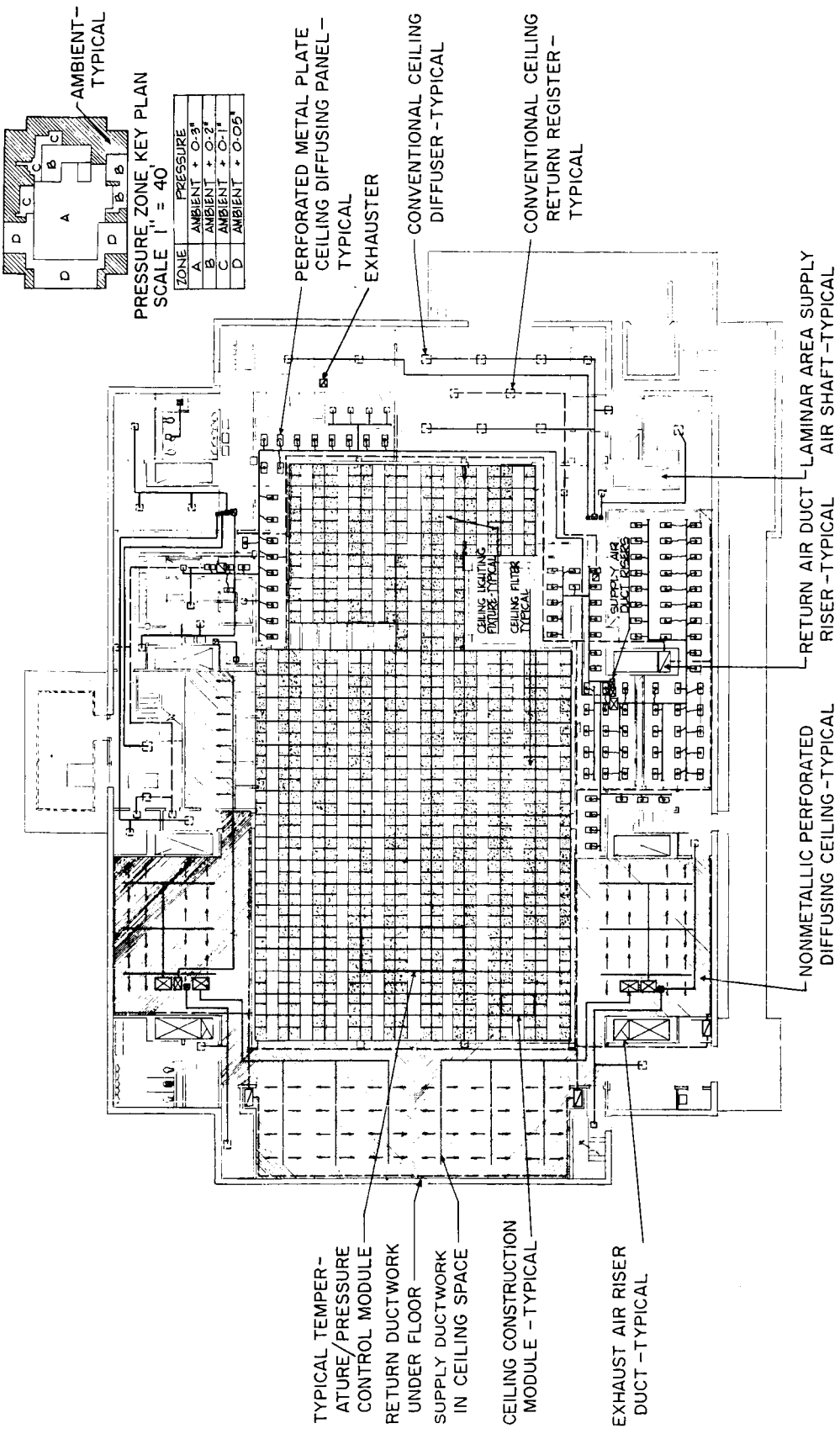


Fig. 19. Criteria drawing: mechanical systems (main level plan and air distribution and mechanical equipment layout)

LEGEND

1. SHIPPING AND RECEIVING
2. MAIN AIR LOCKS
3. SURFACE CLEANING AND DECONTAMINATION
4. CAPSULE DECONTAMINATION – ETO
5. SUBASSEMBLY DECONTAMINATION – ETO AND STEAM
6. SUBASSEMBLY AND QUALITY ASSURANCE – BENCH AREA
7. SUBASSEMBLY – OVENS
8. CAPSULE ASSEMBLY
9. SPECIAL OPERATIONS – ASEPTIC ASSEMBLY AND DISASSEMBLY
10. OPERATIONAL SUPPORT EQUIPMENT
11. MAIN ENTRY
12. MEDICAL OFFICE
13. EXAMINATION AND FIRST AID
14. WOMEN'S TOILET AND SHOWERS
    L – LOCKERS D – DRESSING
    S – SHOWERS T – TOILET
15. MEN'S TOILET AND SHOWERS
    L – LOCKERS D – DRESSING
    S – SHOWERS
16. BIO-ASSAY LABORATORY
17. MEDIA PREPARATION
18. MEDIA DISPENSING
19. STERILE TRANSFER
20. SAMPLE INCUBATORS
21. GLASSWARE WASHER
22. OSE DOCK
23. CAPSULE AND SUBASSEMBLY DOCK
24. TERMINAL STERILIZATION OVEN
25. OVEN CONTROLS
26. MEN'S TOILET
27. COATS
28. OSE READY SUPPLIES
29. SUPERVISORY OBSERVATION
30. TELEPHONE EQUIPMENT
31. JANITORS' CLOSET
32. LAMINAR RETURN DUCT RISER
33. SERVICE ELEVATOR
34. EXIT
35. CLEAN AIR LOCK
36. PRESSURE CONTROL LOCK

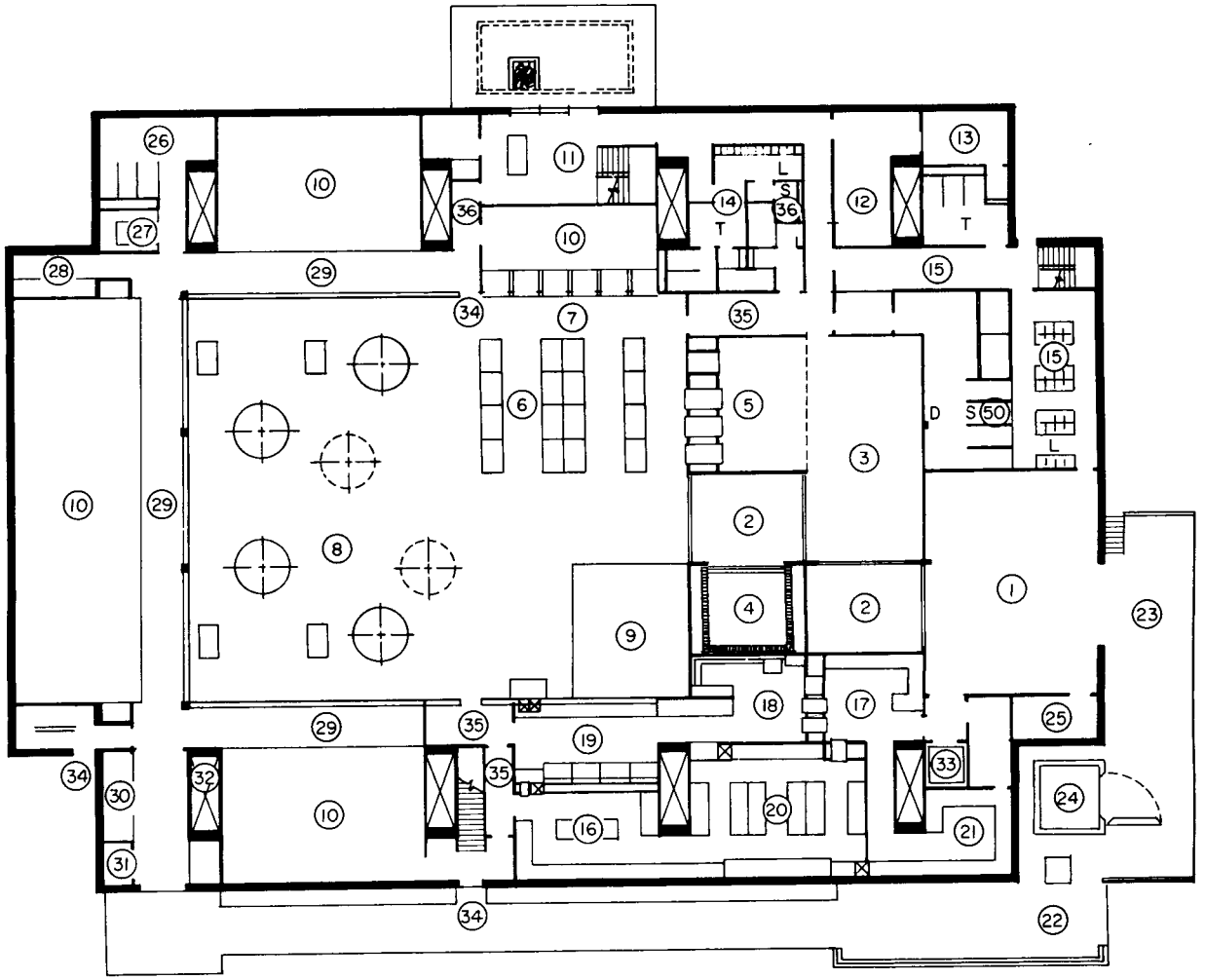


Fig. 20. Criteria drawing: main level floor plan

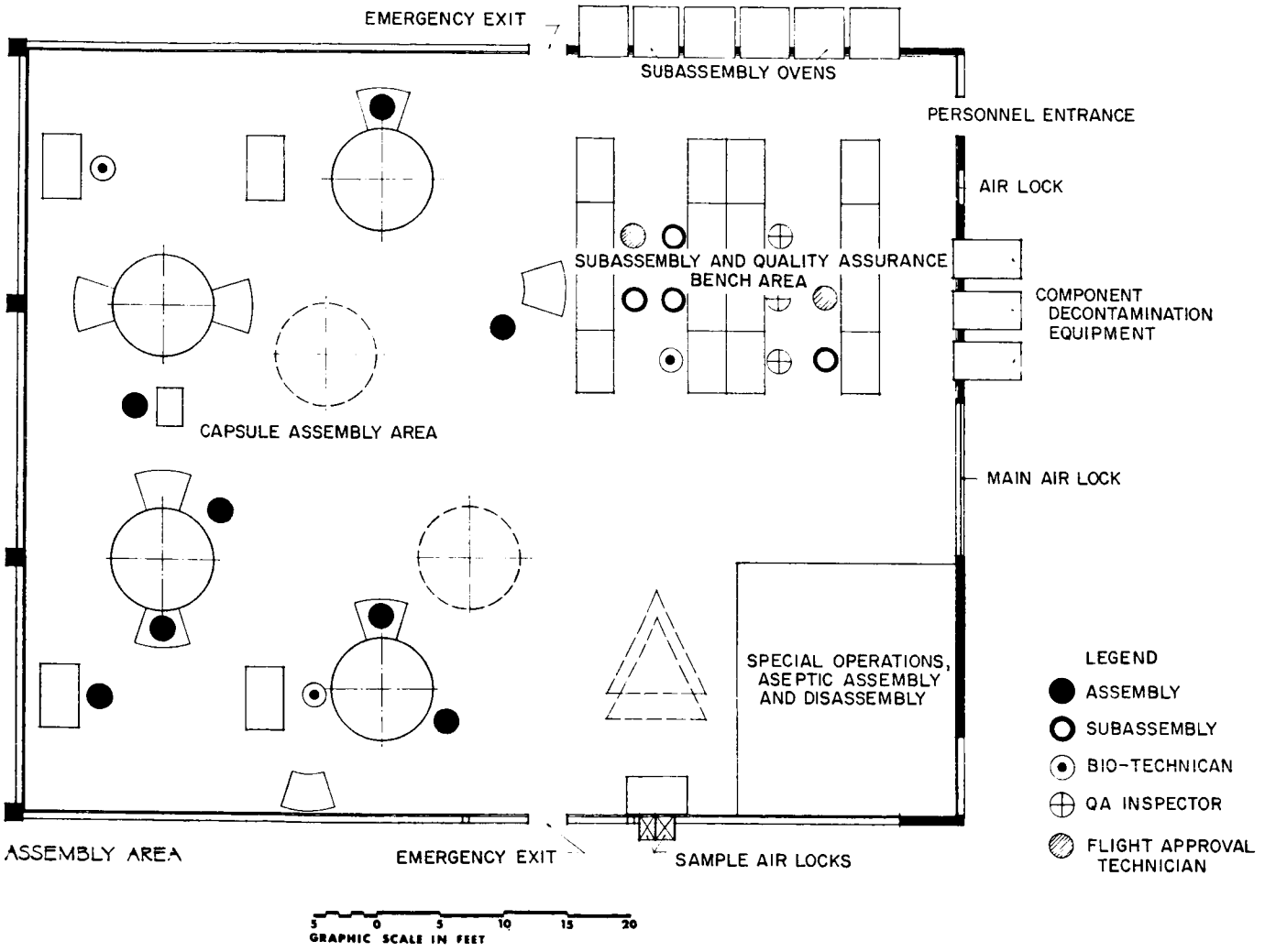


Fig. 21. Criteria drawing: assembly area layout

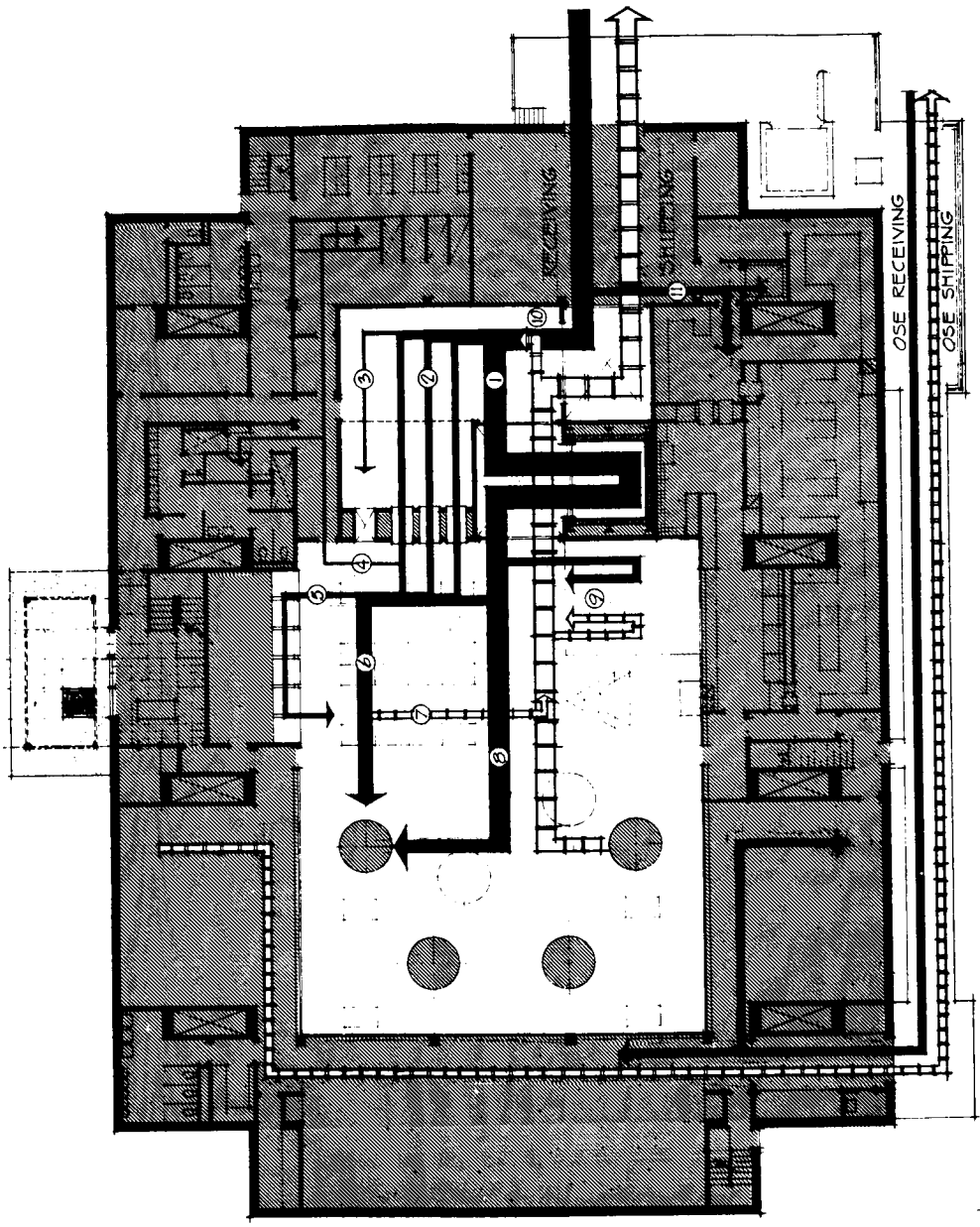


Fig. 22. Concept study: final concept, material flow diagram

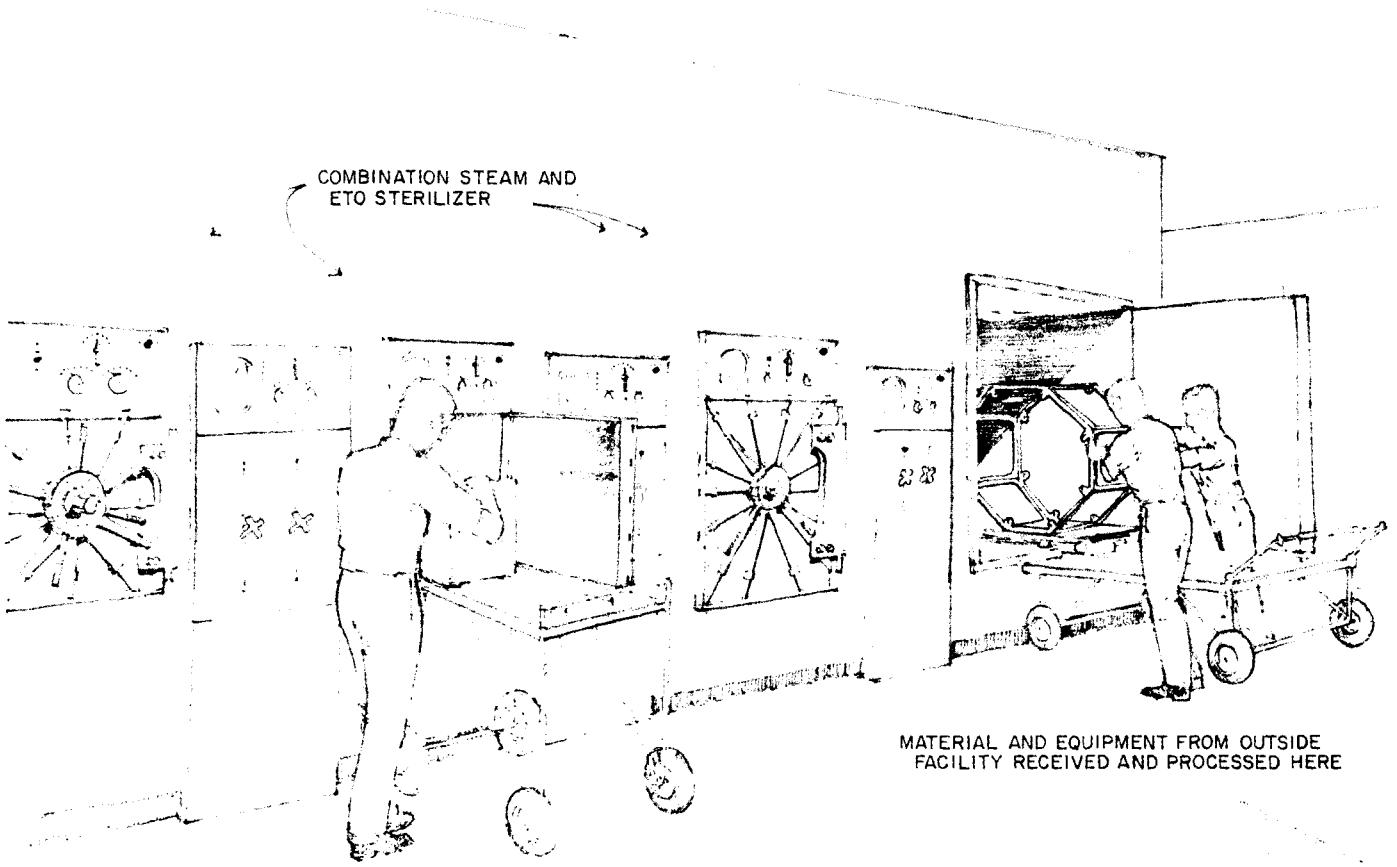


Fig. 23. Typical pass-through decontamination bank from receiving department into clean areas

The selection of proper dress is an extremely pressing concern. On the one hand, personnel could be dressed in the equivalent of a diving suit to completely isolate them from the surrounding environment. On the other hand, they could be dressed in ordinary street clothing and rely on the laminar air flow to remove the microbiological contamination from the area. The former approach appears to be too rigorous and might severely undermine the motivation and performance of the employee. The latter approach appears to be more optimistic than prudent.

A reasonable middle-ground approach which is effective in controlling microbiological contamination from personnel but does not adversely affect the comfort, convenience, and morale of personnel appears to be the answer at this time.

Personnel head coverings are of particular interest since millions of microorganisms may abound in the oral discharge of individual employees. Figures 25 and 26 illustrate typical approaches which have been considered.

The ultimate selection of attire may also be influenced by the development of a capability to decontaminate articles of clothing by passing them through a steam or ethylene-oxide autoclave.

9. Microbiological Assay and Control

A unique feature of the AT&SF will be the constant monitoring of all spacecraft assembly operations for evidence of microbiological contamination. An average of approximately 25 samples per hr may be required to achieve the proper confidence level in operational procedures and techniques. Each of these 25 samples will, in general, be cultivated in two different media (one for bacteria and one for mold), under two different oxygen tensions (aerobic and anaerobic), and incubated at three

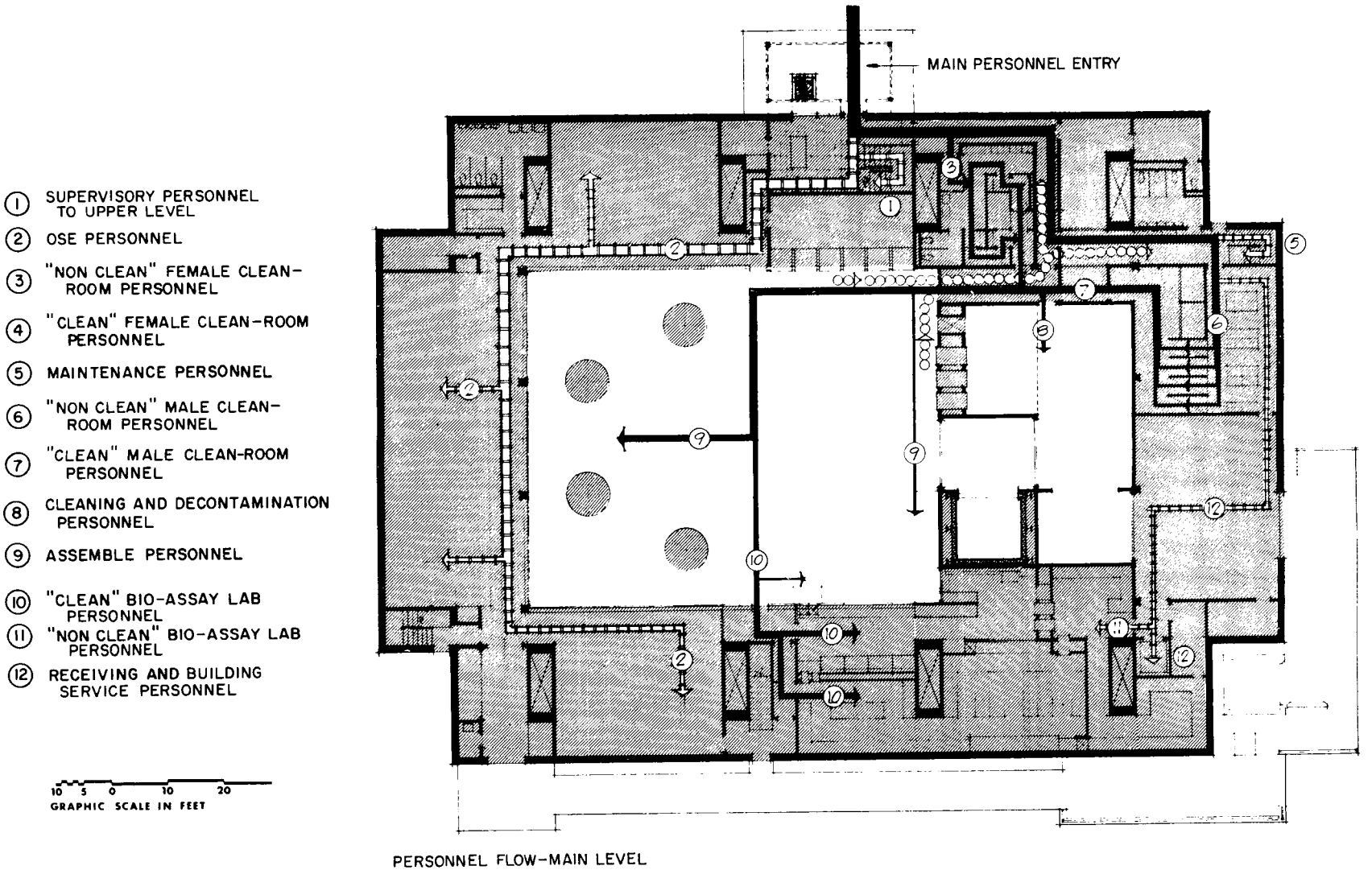


Fig. 24. Concept study: final concept, personnel flow diagram

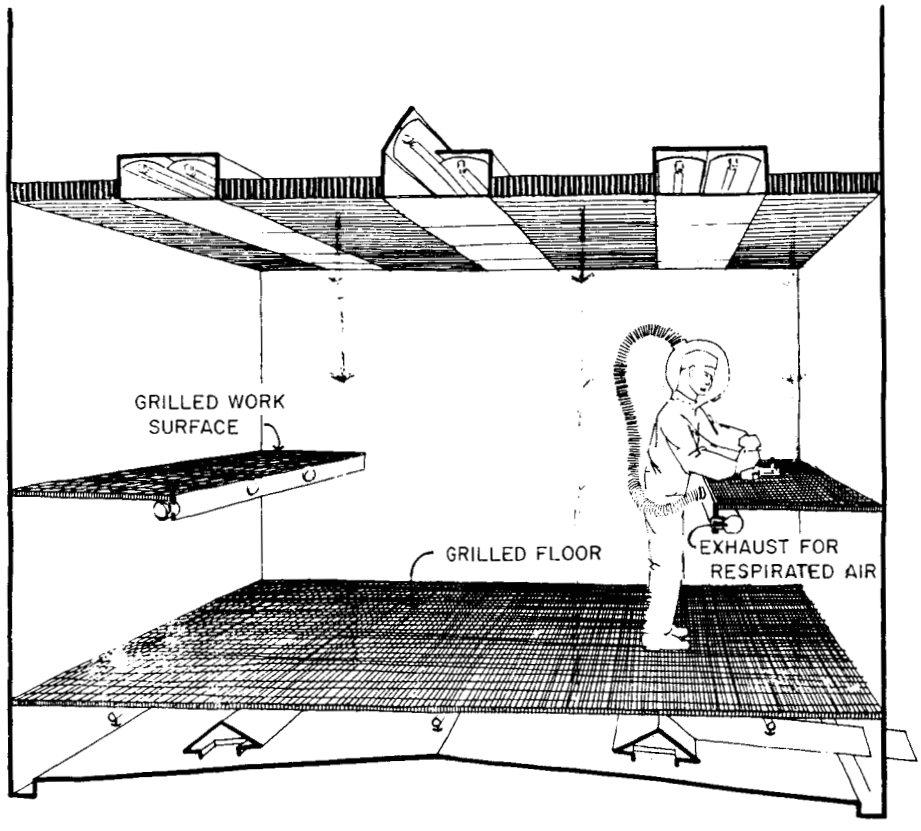


Fig. 25. Personnel ducted helmet

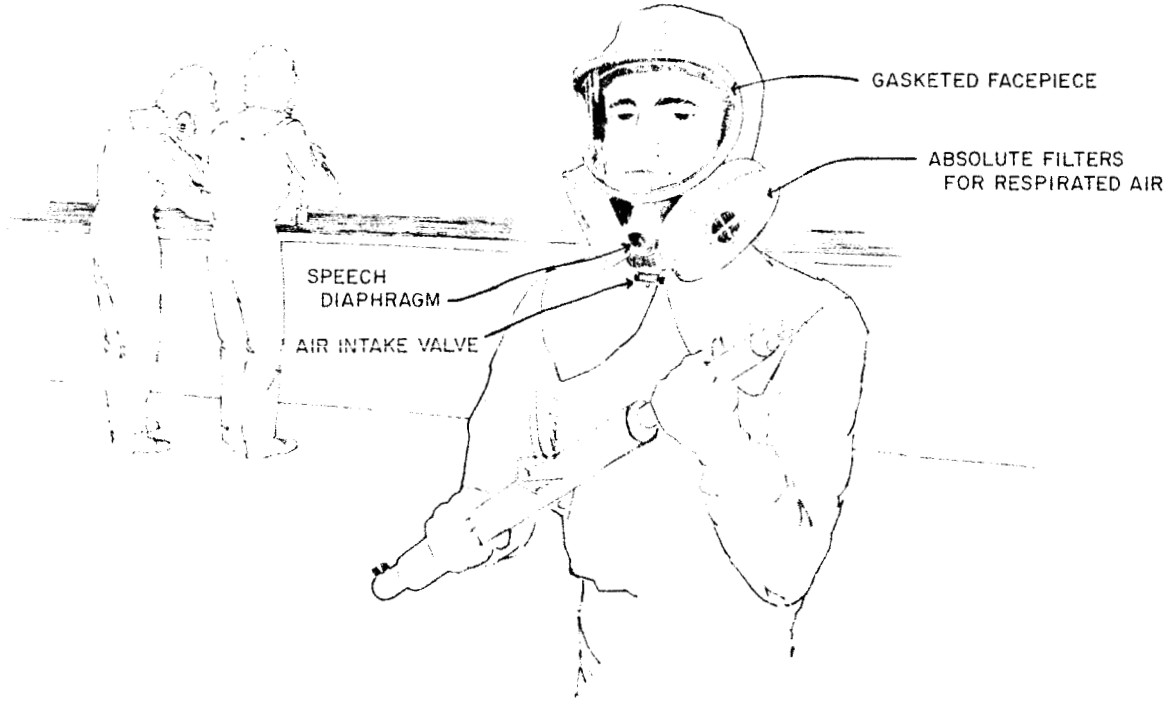


Fig. 26. Personnel respirator mask

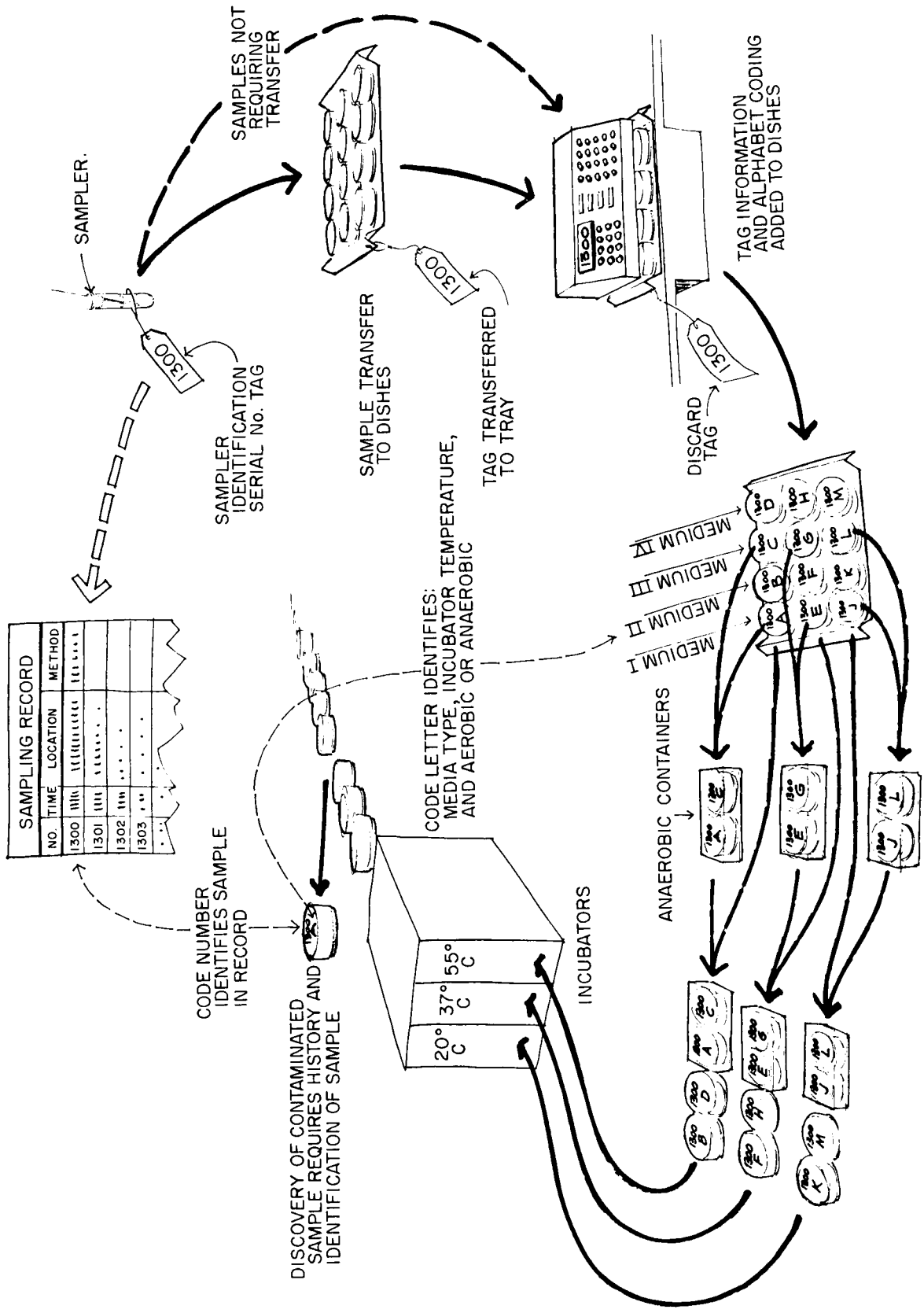


Fig. 27. Identification of culture dish

different temperatures (20, 37, and 55°C). The net result is that for any 8-hr day as many as 2400 specimens may be handled. In addition, samples must be removed from the incubators at 24-hr, 48-hr, and 7-day intervals for examination to determine the presence of microorganisms. Accordingly, an extensive, well-equipped and thoroughly planned microbiological assay laboratory is an inherent part of an AT&SF.

Figures 27 through 32 illustrate concepts of the typical work flow in the laboratory operations and are presented herein to highlight the volume and significance of the work.

10. Recommendations and Conclusions

The design and construction of an AT&SF which will contribute to the ultimate sterility of planetary landers can be demonstrated. The facility can be constructed from off-the-shelf hardware at reasonable costs. While some special crafting of individual items is indicated, no breakthroughs or special hardware developments are required to meet the essential facility criteria.

Conservative calculations made during the studies indicate that a typical *Mariner* capsule could accumulate approximately 1.8×10^6 viable particles during assembly and test operations in the proposed facility.

The following areas appear to need improvement and further development:

1. Automated microbiological assay laboratory devices.
2. Improved design, sizing, and pricing of laboratory equipment.
3. Smaller (instantaneous readout) particulate counters (readily portable).

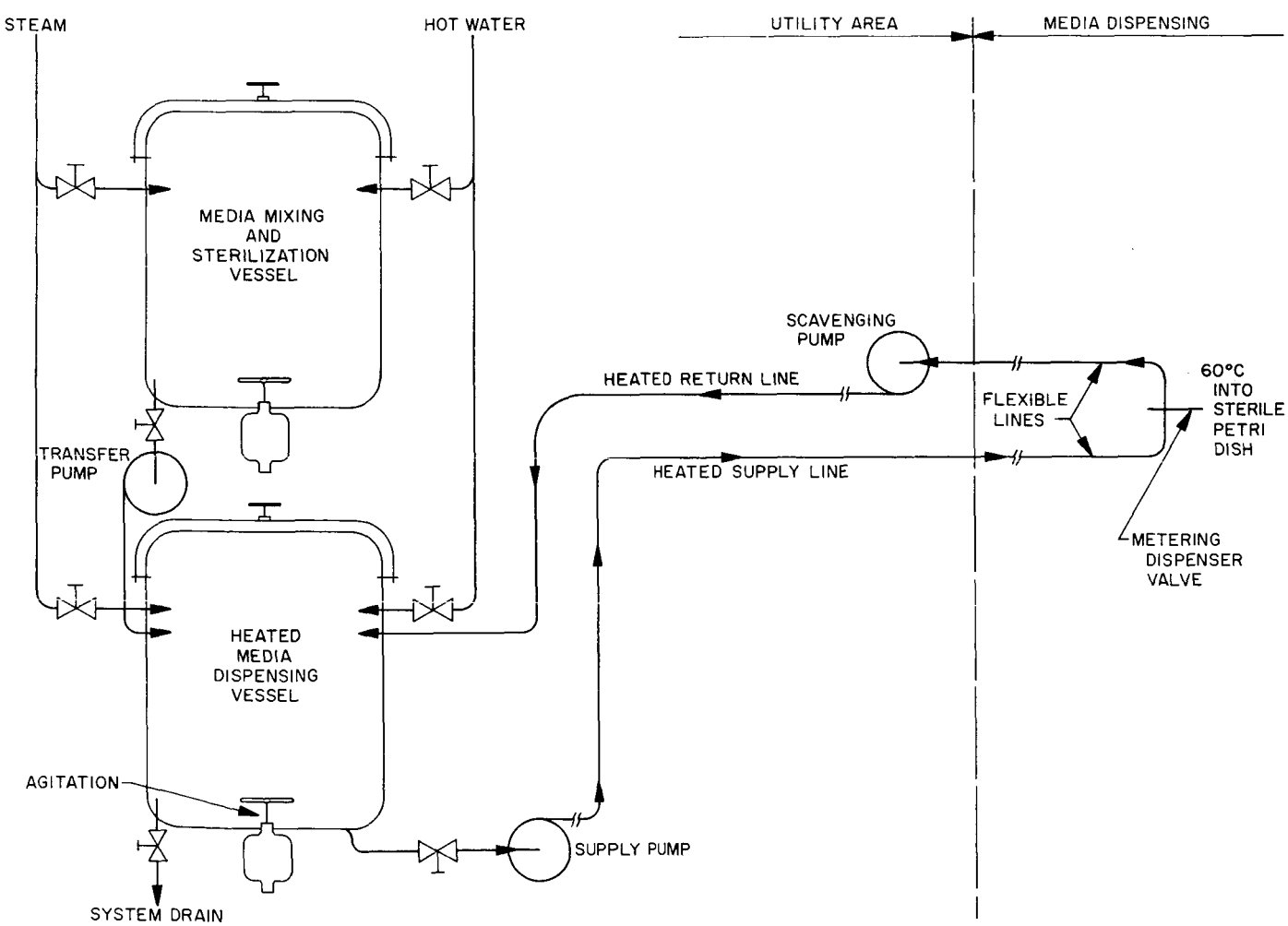


Fig. 28. Temperature-controlled recirculating media dispersing system

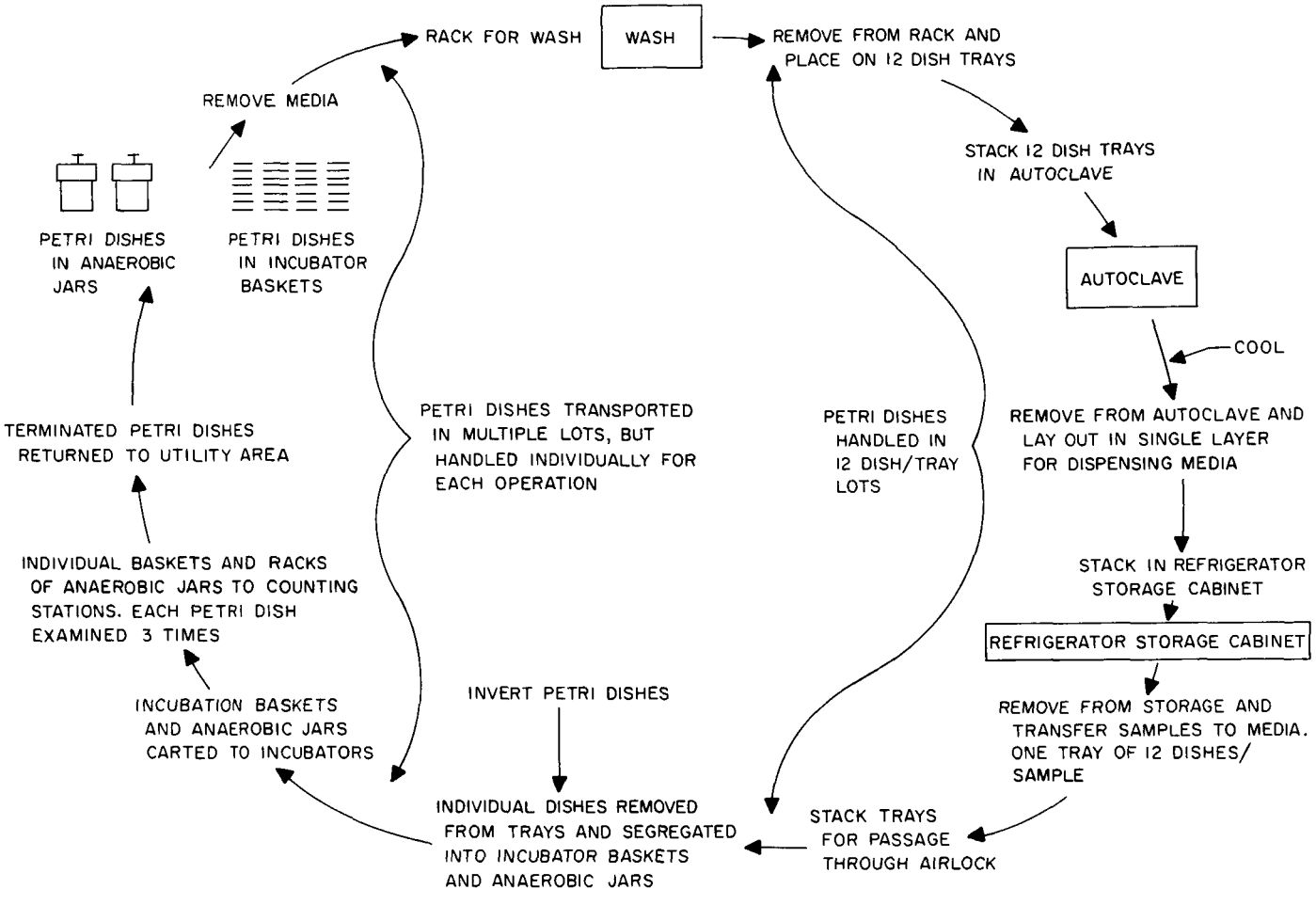


Fig. 29. Petri dish flow diagram "A"

4. Improvements in all clean-room clothing from the standpoint of sterility requirements and with attention to the capability to withstand steam or ETO autoclaving.
5. Improved headgear with emphasis on microbiological contamination problems.
6. New and improved techniques for microbiological sampling and assay work.
7. Additional functional analyses to further develop human factors in man/machine interfaces in spacecraft sterilization technology.

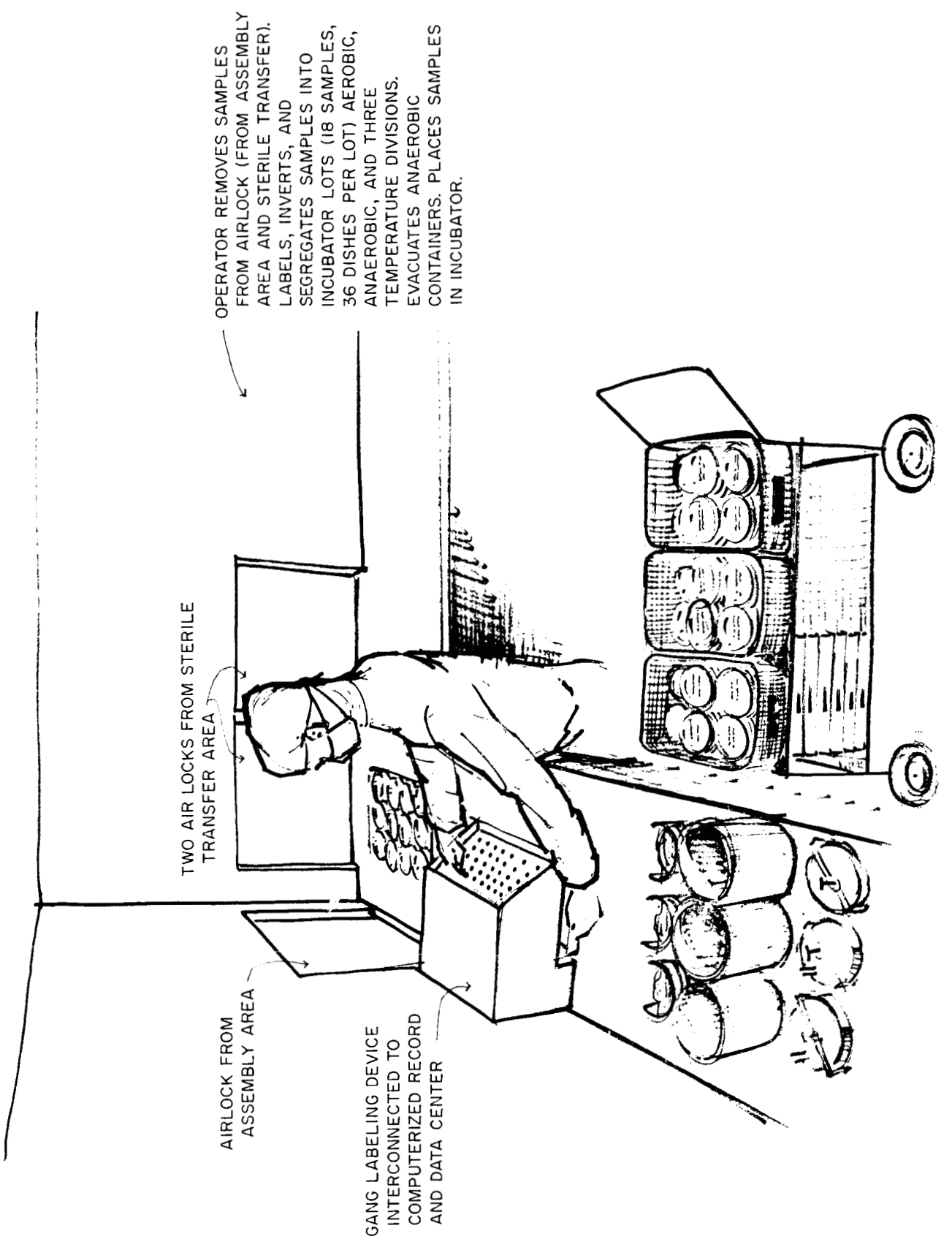


Fig. 30. Segregation and logging

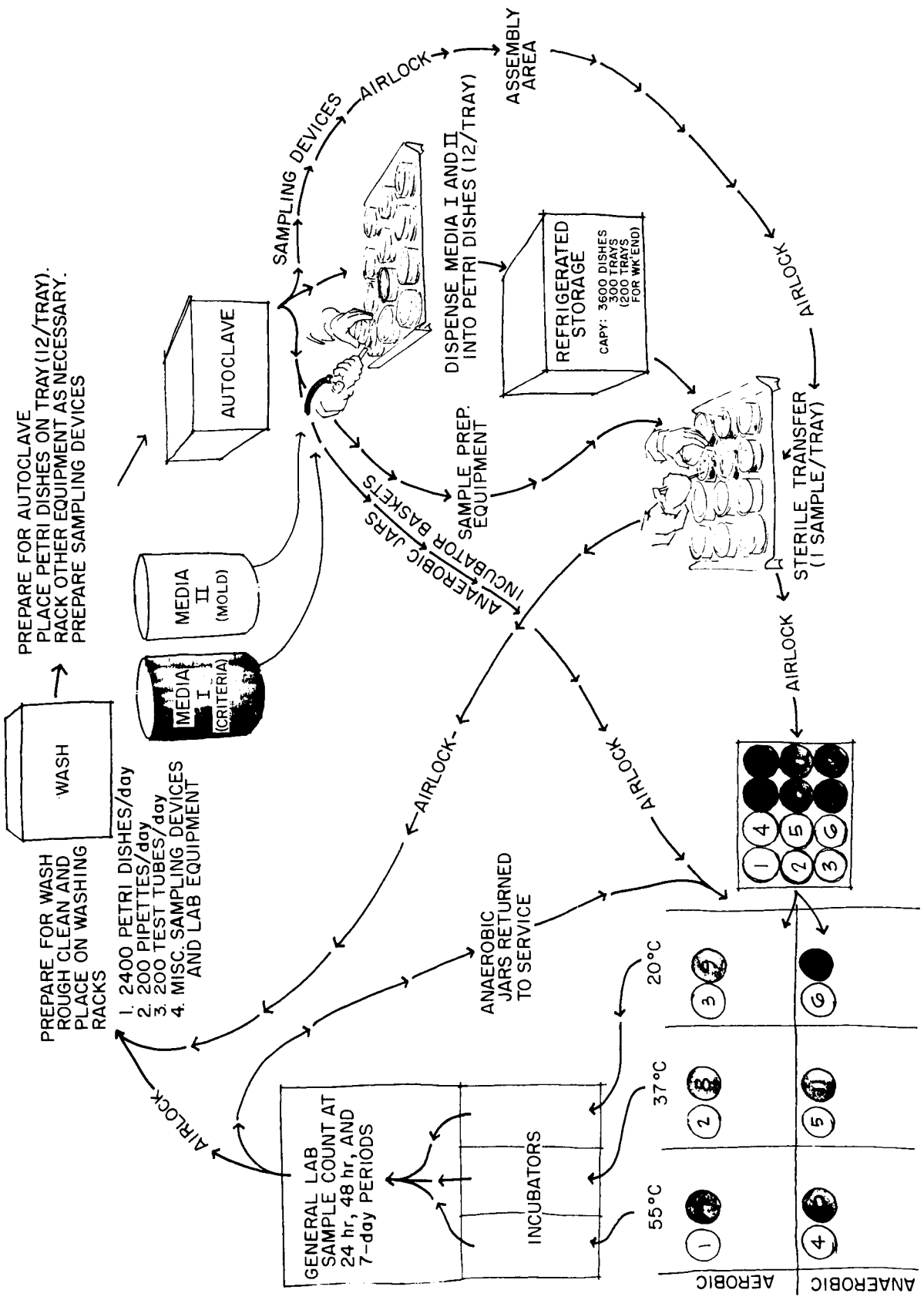
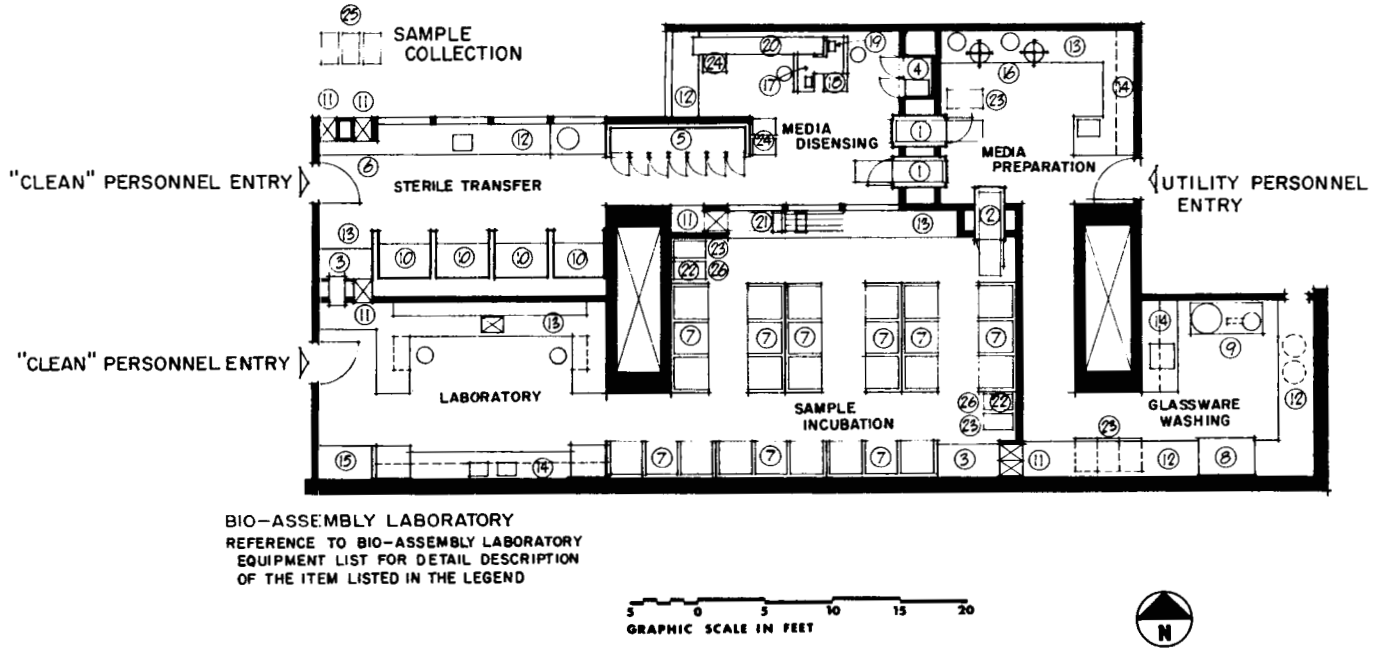


Fig. 31. Petri dish flow diagram "B"



(1) STERILIZER—STEAM VACUUM
(2) STERILIZER—STEAM ETO COMBINATION
(3) STERILIZER—ETO PASSCLAVE
(4) PETRI DISH DRYING AND COOLING CABINET
(5) PETRI DISH REFRIGERATOR
(6) SAMPLER REFRIGERATOR (UNDER COUNTER)
(7) SAMPLE INCUBATORS
(8) LABORATORY GLASSWARE WASHER
(9) WATER STILL; 20 gph, 100 gal STORAGE
(10) LAMINAR WORK STATIONS
(11) AIR LOCKS (APPARATUS PASS THROUGH)
(12) SS COUNTER
(13) BASE CABINETS AND DRAWERS
(14) WALL CABINETS
(15) UTILITY STORAGE CABINET
(16) MEDIA PREPARATION SYSTEMS
(17) LAMINAR DISPENSING STATIONS
(18) TRAY FEED MAGAZINE
(19) AUTOMATIC PIPETTERS
(20) TRAY CONVEYOR
(21) PETRI DISH MARKING AND SORTING UNIT
(22) AUTOMATIC PETRI DISH SCANNER
(23) UTILITY CARTS
(24) TRAY TRANSFER CARTS
(25) SAMPLE COLLECTION CARTS
(26) SCANNER CARTS

Fig. 32. Criteria drawing: bioassay laboratory floor plan

IV. PROJECT ENGINEERING

As part of its activities, the Environmental Requirements Section provides microbiological and systems engineering capabilities for the study, implementation and execution of project policies concerning sterilization requirements. These capabilities have been developed by the section's sterilization group, consisting of professional microbiologists and systems engineers. The group is supported by a unique sterilization laboratory designed exclusively for research and development related to spacecraft sterilization problems. The sterilization group also maintains liaison with other specialists involved in sterilization techniques and research that are applicable to spacecraft sterilization.

A. Recovery of Microorganisms from Solid Materials, J. Iandolo

The elimination of microorganisms by sterilizing treatments presupposes an existing technique to detect contaminating bacteria. In the conventional materials ordinarily assayed by bacteriologists this requirement is easily met. However, the rapid advances in spacecraft technology, aimed at planetary exploration, have introduced sterilization requirements that present a challenge to the microbiologist. The time-tested and proven techniques, using high-pressure steam at 250°F, are no longer applicable. Rather, dry heat and gaseous fumigation techniques are being investigated. This change in approach is due to the need for sterilization of nonconventional materials.

Determining the presence of contaminating microorganisms in such materials as plastics, epoxy resins, and rocket propellant is essential for a proper evaluation of the sterilizing treatment. The surfaces of these solids can be examined by various means, but the presence of interior flora is considerably more difficult to realiably determine. To solve this problem, the Dynamic Science Corporation[1] under contract to JPL, has been developing methods for the detection of viable organisms in solid materials. Initially, several avenues of approach were utilized, encompassing physical as well as biological assay systems. These data were largely negative. Distinctions between the background material and viable and nonviable particles were not always possible with methods such as electron spin resonance, staining, fluorescent staining, electrophoresis and radioautography. Consistent results were produced by bacteriological culturing techniques, which were adopted for further study.

[1] 1900 Walker Ave., Monrovia, Calif.

The most immediate problem facing the researchers was the freeing of the microorganisms from encasement. Pulverization techniques involving drilling, ball mill, Waring blender, and mortar and pestle were investigated as well as techniques designed to solubilize solids. Although encouraging results were obtained with these techniques, none was found to be as effective as drilling, and this was eventually determined to be inferior to sawing the solid and examining the saw dust for bacteria.

Recovery of microorganisms from solids prepared to contain a standard count per unit volume was below expectations. Improvement was obtained by removing inhibitory substances present in the solids. Leaching with sterile distilled water was employed, after which the redox potential and pH were adjusted. The leached particles were then cultured. Using these techniques, viable organisms in quantities as low as 10^1–10^2/cc could be detected.

Future contracted activities planned for the Dynamic Science Corporation involve improvement of the recovery techniques now available and the development of new procedures consistent with the aims of the study. A detailed study of sawing methods and saw types for several classes of solids is necessary in order to improve the efficiency and applicability of the methods while simultaneously reducing any physiological damage to the entrapped organisms. Additionally, studies are to be made of the optimum conditions for leaching toxic substances from the pulverized solids. Further, there exists a need to devise methods by which the soluble inhibitors (leachates) and the inhibitors present within the solid (nonsolubles) may be neutralized or inhibited.

A broader, more detailed investigation leading to the development of culture media which optimize the recovery of injured organisms will be made. A study of this type will necessitate the consideration of several classes of substances capable of counteracting the injuring effects of pulverization and leaching. Growth factors and metabolic constituents not normally required by the organisms under other conditions will be studied. The trace metal content, the ionic strength, the pH, and the redox potential will also be considered.

The basic aim of the research plan is to define a practicable procedure for the recovery of entrapped microorganisms, regardless of their physiological condition,

from the interior of compatible solid materials used in spacecraft construction.

B. A Biological Sterility Indicator for Dry Heat Sterilization, A. Irons

The need to prove the sterility of spacecraft which will impact extraterrestrial surfaces requires the development of a biological indicator capable of proving, with a high degree of confidence and reliability, that sterilization has in fact been achieved. A biological test system is a valid method of testing for the adequacy of the proposed sterilization cycle. This work is under contract to the Wilmot Castle Company, Rochester, New York.

The indicators, when developed, could be placed in various locations about the spacecraft being sterilized, and after the sterilization process they could be retrieved and cultured to determine whether or not the process was successful in destroying all the microorganisms associated with the indicator. The organisms contained in the indicator would have been previously shown to be:

1. One of the most dry-heat-resistant type of organisms.
2. Present in numbers in excess of the number of organisms possibly present on or in the spacecraft.
3. Capable of being grown on the recovery medium being used.

At the start of the program, the state-of-the-art did not permit the fabrication of an indicator capable of surviving exposure to a temperature of 135°C for a period of approximately 20 hr, which was one of the requirements. Since then, an indicator has been made which meets our requirements of time, temperature, and exposure in an atmosphere of dry nitrogen.

The problem of heat resistance was solved by using vacuum-dried spore powder. The number of spores contained in each indicator approximates 1×10^{11}. This number of spores, in powder form, is compressed into a ¼-in. tablet under pressure of 30 tons per sq. in. after freeze drying. The problem of exposure of the organism to dry heat while in an atmosphere of dry nitrogen was solved by fabricating a Teflon tube, hermetically sealed, to contain the spore tablet in an atmosphere of N_2.

Resistance of the indicator organisms to 20 hr at 135°C has been erratic and variable. The erratic survival pattern is being investigated, and it is felt that this problem can be solved in one of several ways:

1. Production of a cleaner spore crop centrifuged in a refrigerated centrifuge to prevent incipient germination, with the resultant contamination of the spore powder with dead vegetative cells.
2. Thermal insulation of the spore tablet.
3. Use of a strain of *B. subtilis* spores which appear to have better heat-resistant characteristics than the WC18 strains now in use.
4. Agitation of cultures after inoculation with the exposed spores. (This has produced positive cultures where otherwise they appeared to be negative.)

Industry can benefit from the availability of a biological indicator which can be utilized for determining sterility at increased temperatures for extended periods of time.

C. Microbiological Filters for Liquid and Gas, A. Irons

Production of certain sterile spacecraft systems and the maintenance of sterility of these systems may require sterilization by filtration. For example, some of the liquids required for biological experiments may be degraded by the proposed dry heat sterilization cycle. Also, sterile assembly procedures which may include glove box systems should require some method of sterile filtration of air. Clean room assembly procedures accomplished under conditions of laminar-flow air required high-efficiency filtration to keep the airborne microbial load at a minimum.

This study was designed to make an impartial evaluation, under use conditions, of currently available filters. The intent of this study is to determine which filters, if any, are acceptable for use in the assembly and maintenance of sterile spacecraft and to compile a list of filters which can be used for absolute filtration of air or gas and/or sterilization of gas and liquid. This work is under contract to the Wilmot Castle Company, Rochester, New York.

This evaluation will cover four specific areas:

1. Evaluation of superinterception filters in a duct.
2. Evaluation of filters in a pressure gas-flow system.
3. Evaluation of filters in a pressure liquid-flow system.
4. Evaluation of filters in the filtration of small volumes of liquids under gravity flow conditions.

In all cases, liquids or gases will contain microbial contamination.

Certain problems were presented by this study such as:

1. Consistent contamination of the air stream used to challenge the superinterception filters.
2. Locating physical leaks which were detected by microbiological samplers.

These problems were solved in the following manner:

1. A system of nebulizing a given concentration of organisms (spores) suspended in nearby absolute methanol into a duct was developed which produced a consistent contamination level of 200,000 viable particles per min.
2. A "roving probe" was developed which is attached to a Reyniers air sampler. This procedure permits sampling the entire face of the filter and relating position on the filter face to time, microbial count, and velocity.

This study will result in a system of testing and evaluating filters that will permit more accurate evaluation of efficiency and reliability. Increased confidence in future selection and use will also result.

A "velocity profile" of superinterception filters which will indicate the homogeneity of the filter medium should be of importance to filter manufacturers.

D. Microbiological Profile of Clean Room, J. J. McDade

Information on the levels of microbial contamination within industrial clean rooms is not presently available. Such information, however, is necessary to determine the assembly and/or testing environment for spacecraft hardware that must meet the space quarantine requirements. A study in this area is being conducted by Douglas Aircraft Company under contract to JPL.

The study phase of this contract proceeded successfully, with no major problems or technical difficulties encountered. At the end of August 1965, the Douglas Aircraft Company had completed the sampling program as follows: Six weeks of microbiological sampling in the Class II clean room (three sampling sites within the clean room and one sampling site outside the clean room), the Class III clean room (one sampling site inside and one sampling site outside the clean room), and the Class IV clean room (one sampling site inside and one sampling site outside the clean room).

1. Air Sampling Studies

a. Class II clean room. The number of viable airborne particles ranged from 0.0 to 1.0 per cu ft of air (during lunch period when the room is empty) to 10.0 to 20.0 per cu ft of air (when the room is fully staffed and operated under "normal in-use" conditions).

b. Class III and IV clean rooms. The number of viable airborne particles ranged from 0.0 to 0.5 per cut ft of air (during lunch) to 0.5 to 2.0 per cu ft of air (when the room is fully staffed and operated under "normal in-use" conditions).

2. Surface Sampling Studies: Settling Strips

a. Class II clean room. 10^3 microorganisms (1000 to 9999) were recovered per sq ft of surface over an exposure period of 1 to 10 weeks.

b. Class III clean room. 10^2 microorganisms (100 to 999) were recovered per sq ft of surface over an exposure period of 1 to 10 weeks.

c. Class IV clean room. The recovery figure was essentially the same as described for a Class III clean room.

3. Other Studies

The results obtained with other samplers and procedures (Elliott, AGI, Andersen, settling plates, and human handling experiments) have not been reviewed, tabulated, and compared in sufficient numbers for comment. The final report of this study is scheduled to be submitted by the contractor during October 1965.

E. Sterile Assembly Techniques, J. J. McDade

The sterile barrier concept is not a new or unique approach to the control of microbiological contamination. The field of gnotobiology has developed and centered around the maintenance of sterile environments behind microbiological barriers. The techniques of gnotobiology have been developed to a point where it is now possible to bring a germ-free male animal from one geographical area of the United States to another and successfully mate this male with a germ-free female of the same species, ultimately resulting in a germ-free animal colony. This example is cited to suggest application to a different situation: the development of equipment and techniques

for the assembly of spacecraft materials and parts into a sterile unit within the confines of a microbiological barrier. The resultant small sterile units might then be assembled into larger units, perhaps resulting in the production of sterile spacecraft hardware. Thus, heat-labile units might be produced by the sterile assembly of small component items that are heat stable. Or, perhaps, sterile components can be placed into a sterile spacecraft by means of sterile assembly techniques. Alternatively, field repair situations might be encountered wherein a malfunctioning part of a sterile spacecraft must be replaced by a sterile, operationally functional replacement. Consideration of such operations can only be realized when it has been demonstrated that sterile assembly techniques are effective and reliable barriers against microorganisms.

A recent JPL subcontract with the Lockheed Missiles and Space Company (LMSC) has shown that it is possible to assemble a small electronic unit in a glove box, using glove box techniques in atmospheres of sterile nitrogen, sterile air, or a gaseous mixture of 12% ethylene oxide and 88% Freon 12 (ETO-F12). In general, assembly takes about three times as long in the glove box when compared with assembly on the bench. Hand soldering, dip soldering, staking, nut/bolt connections, and epoxy bonding and potting operations may be effectively accomplished in a 12% ethylene oxide, 88% Freon 12 gaseous mixture (ETO) in sterile nitrogen or in sterile air. Occasional ETO "skips" in decontamination were observed, with capacitors being the item most frequently involved in the "skips." The final study report was received during March 1965. The study was satisfactorily and successfully completed by LMSC.

Based on the results obtained by the contractor, it was decided to continue the evaluation of possible sterile assembly technique applications to spacecraft sterilization. Final results from the JPL/LMSC study were necessary for the development of a request for proposal to continue sterile assembly techniques. Completion of the task has provided this information. Emphasis will be placed on equipment development, actual use situations, and reliability of the barrier system to produce and maintain a sterile environment and product.

Assembly is to be conducted in a sterile environment established through use of a microbiological barrier system. Sterile assembly as well as sterile repair situations will be investigated. All operations will be conducted in a sterile environment (other than the ethylene oxide, Freon 12 mixture or sterile nitrogen). Emphasis will be placed on the microbiological aspects of getting tools and equipment into the sterile work area and removal of the sterile product in some type of protective envelope. The integrity of the barrier will be challenged both chemically (with Freon and/or helium gas) and microbiologically (with bacterial spores). All phases of the operation will be microbiologically monitored.

F. *Clean Room Prototype*, *J. J. McDade, W. Paik, and M. Christensen*

Preliminary reports on the microbiology of clean rooms have been published (Refs. 1 to 3). Yet, at present, data concerning levels of microbial contamination within industrial clean rooms are scanty or totally lacking. Information on the levels of microbial contamination within spacecraft assembly areas is not available either. In order to obtain more definitive information on the microbiology of clean rooms, the JPL Sterilization Group initiated tasks to: (1) determine the microbiological profile of clean rooms,[2] and (2) develop a clean room prototype as an experimental laboratory in which microbiological research and training could be conducted (Ref. 4).

The data obtained from the microbiological profile study of clean rooms will result in a firm estimate of the microbial contamination levels that exist in industrial clean rooms (Classes II, III, and IV, according to Ref. 4). However, the operational recommendations and requirements established in the "NASA Interim Requirements" (Ref. 5) have necessitated an updating in design for spacecraft assembly and test facilities. Therefore, in order to determine the efficacy of the specified procedures and to attain the allowable levels of microbial contamination as established in the "NASA Interim Requirements," it was necessary to construct and operate a facility according to these requirements (Ref. 1). To provide such a facility, the JPL Sterilization Group task entitled "Clean Room Prototype" merged funds and objectives with the *Voyager* Project Office to establish an experimental assembly and sterilization laboratory (EASL) at JPL.

Construction of the EASL facility began during February 1965 and was completed on July 1, 1965. The purpose of this facility is to conduct an investigation into some of the procedures and techniques for assembly and/or test of spacecraft hardware under full implementation of the "NASA Interim Requirements." Assembly and test will be at the component and modular levels.

[2] JPL/Douglas Aircraft Co. Contract No. 950920: "Microbiological Profile of Clean Rooms."

Simultaneously, a duplication of the work being done in the EASL facility shall be conducted under non-bioclean conditions or routine laboratory conditions and procedures. Scheduled microbiological assays of the hardware and assembly environment will be conducted throughout all operational states of both the bioclean and non-bioclean assemblies. The cost in time and money for both types of assembly will be compared, contrasted, and reported. Operational reliability of the assembled units will also be checked.

The rationale of microbiological monitoring, assay, and certification has been discussed in JPL Specification GMO-50470-GEN (Ref. 6); the procedures to implement this specification are described in Ref. 7.

The activities planned for the EASL facility by the JPL Sterilization Group include the following (items marked with asterisks are monitoring activities; all others are microbiological research studies):

1. Microbiological assays and monitoring.* Microbiological monitoring routines shall be established and applied to demonstrate the reliability of the facility to conform to the requirements set forth in the "NASA Interim Requirements" standard. The following activities constitute the microbiological studies to be conducted in EASL:
   a. Microbiological sampling of air.
   b. Sampling of air for a total particulate count.
   c. Microbiological sampling of environmental surfaces.
   d. Microbiological sampling of spacecraft hardware.
   e. Microbiological sampling of clothing and packaging.
   f. Microbiological sampling of personnel.
2. Visual assay.* (See Refs. 6 and 7.)
3. Parametric measurements.* (See Refs. 5 and 6.)
4. Terminal heat sterilization.*
   a. Dry heat in:
      1) Nitrogen atmosphere.
      2) Dry air atmosphere.
   b. Supporting data required: Instrumentation of sterilization oven and hardware with thermocouples connected to a recorder.
5. Microbiological research*
   a. Modification of old, or development of new, microbiological sampling equipment and/or procedures.
      1) Air sampling.
      2) Surface sampling.
   b. Determination of the survival curves for surface-exposed microorganisms in laminar flow (horizontal and vertical) air streams.
      1) Naturally contaminated surfaces.
      2) Artificially contaminated surfaces.
         a) Fungal spores.
         b) Bacterial spores.
            (1) Members of the genus *Bacillus.*
            (2) Members of the genus *Clostridium.*
         c) Bacterial vegetative cells.
            (1) *Staphylococcus aureus.*
            (2) *Escherichia coli*
            (3) *Proteus vulgaris.*
            (4) *Pseudomonas sp.*
         d) Viruses (bacteriophage), *E. coli T* series.
6. Evaluation of personnel attire on dissemination of microorganisms: ranges from fully clothed (bunny suit, facial mask, surgical gloves, booties) to no protective clothing (street clothes).
7. Evaluation of germicides for in-use application situations.
   a. Facility decontamination.
   b. Decontamination of handled or dropped tools.
   c. Decontamination of handled or dropped hardware.
   d. Any other situation requiring sterilization or decontamination.
8. Microbial recovery from surfaces.
   a. Routine procedures.
      1) Contact methods.
      2) Direct plating.
      3) Rinse methods.
   b. Sonic removal: effect of sonic cleaning or microbial contamination of hardware, tools, other assembly items, and artificially contaminated surfaces.
9. Recovery of microorganisms from hardware interiors.*

10. Other application situations as operational problems are uncovered. Scheduling of these activities is presented in Fig. 1 and the following activity schedule:

Activity	Frequency
Microbiological	Daily
Visual assay	Daily
Parametric measurements . .	Daily
Microbiological research . .	Daily, or as operating schedule permits

Currently, the EASL facility is scheduled to have a hardware assembly series for one month, followed by a 2- to 3-week evaluation of results and a preparation period for the next hardware assembly series. Monitoring activities will be conducted throughout all phases of EASL operation (including terminal stages of construction and balance and checkout, as well as during actual operation by the Engineering Mechanics Division after phase-out of the facility group). Microbiological research activities will be conducted during the assembly phases of EASL operation as scheduling permits. Following completion of a hardware assembly series, and during evaluation and preparation of a new hardware assembly series, specific, detailed microbiological research activities will be conducted.

The materials and methods for implementing the monitoring activities have been described (Ref. 6 and 7). The technical implementation of the research activities is beyond the scope of this report.

G. Microbial Certification of the Experimental Assembly and Sterilization Laboratory

J. McDade, W. Paik, M. Christensen, D. Drummond, and V. Magistrale

The EASL microbiological support and research team has conducted a number of environmental sampling activities during June 1965. It is the purpose of this report to provide a summary of the results obtained during this sampling program and to provide the documentation necessary for microbiological certification of the EASL.

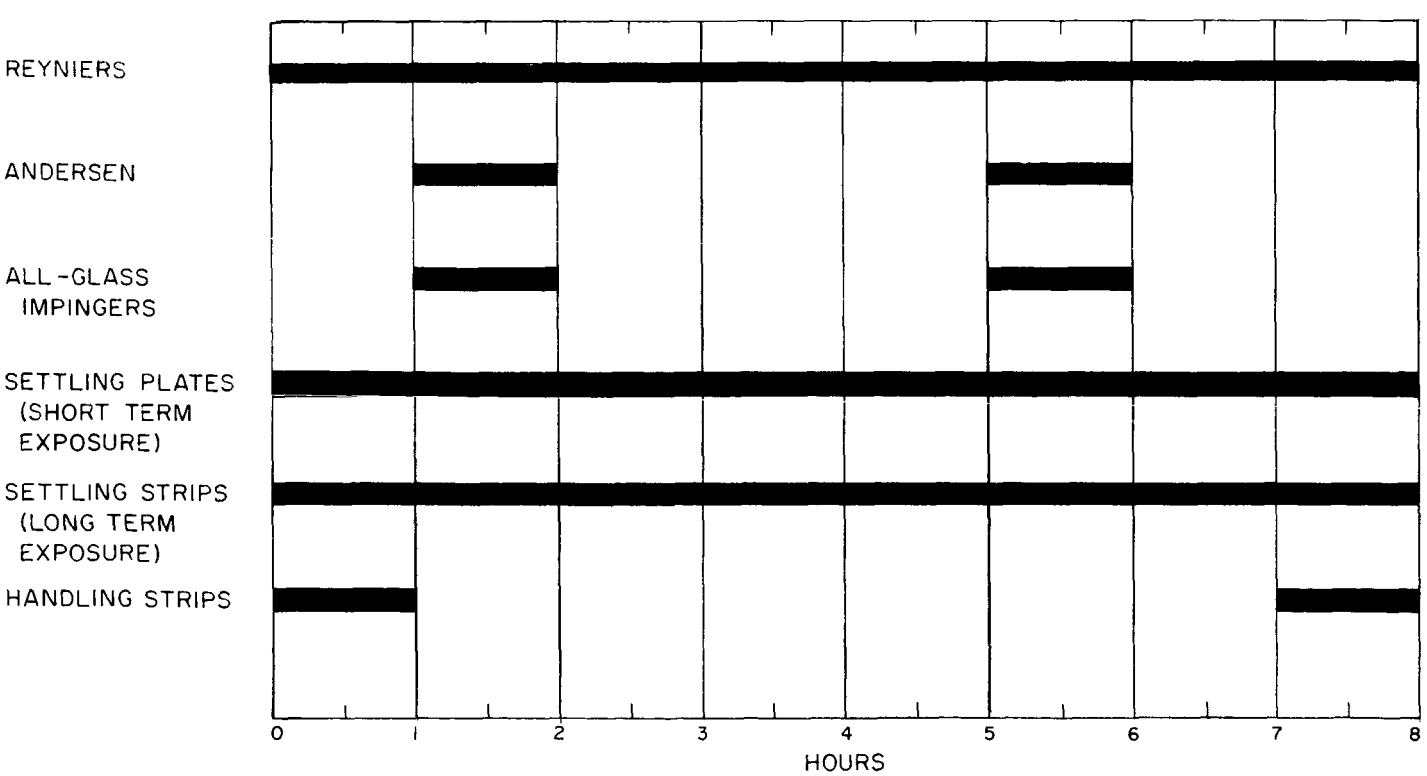


Fig. 1. Typical microbiological sampling schedule

1. Microbial Requirements to be Met for Certification of the EASL Facility

The microbiological requirements for bioclean facilities have been described elsewhere (Ref. 5). A recapitulation of these requirements is as follows:

a. Level of airborne microbial contamination (Ref. 5). "The concentration of viable particles in the room air shall not exceed an average of 2 viable particles per cubic foot of bioclean room air for any 10 successive samples."

b. Level of total particle contamination (Ref. 8). "A Class 100 clean room shall contain no more than 100 particles 0.5 microns and greater in diameter and no particles 5.0 microns or greater in diameter."

c. Level of microbial contamination on surfaces (Ref. 5). "The degree of microbial contamination accumulating on surfaces shall not exceed an average of 200 viable particles per square foot of surface."

1. Materials and Methods

a. Culture media. Trypticase soy agar was used to collect all volumetric air samples. Sterile 1.0% peptone water was used to remove the microbial contamination deposited onto stainless-steel strips during environmental exposure of the strips. Aliquots of this rinse fluid were plated on trypticase soy agar. All samples were incubated at 32°C for 72 hr.

b. Microbiological sampling of air. Volumetric air samples were collected with a Reyniers slit sampler. All samplers were equipped with a 1-hr clock motor. Each sampler was calibrated to operate at a sampling velocity of 1 cu ft of air per min.

c. Total particle sampling. A Royco particle counter (Model 202) was used to obtain the number of total particles per cu ft of intramural air. The machine was programmed to sample on two of its fifteen individual channels (recording a count of particles 0.3 microns and larger in diameter on channel 1 and a count of particles 0.5 microns and larger in diameter on channel 3). Scan rate was for a 10-min period at a sample collection velocity of 0.01 cu ft per min.

d. Microbial fallout sampling. Stainless steel strips 1.0 × 3.0 × *ca.* 0.06 in. were used as microbial fallout sampling surfaces. For use, the strips were washed with nonionic detergent, rinsed with distilled water, rerinsed with isopropyl alcohol, and then terminally rinsed with ethyl ether and allowed to drain dry. The clean strips were arranged as a monolayer on sheets (*ca.* 10.5 × 14.5 in.). Finally, an outer layer of aluminum foil was used to wrap each tray containing clean stainless-steel strips (35 strips per tray). Trays wrapped with aluminum foil were sterilized by exposure to dry heat (175°C for 3.0 hr). Following sterilization and prior to use, five strips were removed from each tray and assayed as sterility control check items.

e. Environmental exposure of the sterile stainless-steel strips. An aluminum-foil-wrapped tray was placed at each of the microbial fallout sampling sites shown in Fig. 2. At each site the aluminum foil was carefully removed from the tray, and, if necessary, the sterile strips were rearranged as a monolayer with sterile forceps. Trays were placed at work height, i.e., on a level comparable to the height at which hardware will be assembled.

f. Procedure for assaying the number of viable particles contained on stainless-steel strips after environmental exposure. For each microbiological assay, five exposed stainless-steel strips were randomly selected from each of the microbial fallout sampling sites. Each strip to be assayed was placed into a separate, sterile bottle. A sterile screw cap was placed on each of the bottles. The strips were then taken to the sterilization laboratory for microbiological assay. To assay each set of five strips from their respective sampling sites, 25.0 ml of sterile 1.0% peptone water was added to each bottle containing an environmentally exposed stainless-steel strip. Next, each bottle containing peptone water and stainless-steel strip was mechanically shaken for 5 min. After shaking, the samples in each series were processed as follows:

1) The liquid content of the bottle was asceptically transferred into a sterile, 150-mm-diameter Petri plate; 25 ml of sterile, double-strength Trypticase Soy agar was added to the strip rinse liquid, and the content of the petri plate was carefully and thoroughly mixed. After mixing, the plates were allowed to stand at room temperature until the agar solidified (*ca.* 15 min).

2) All plates were incubated at 32°C for 72 hr. A preliminary count was made after 40 hr of incubation and a final count made after 72 hr of incubation at 32°C.

2. Results

a. Phase I sampling. A general outline of the floor plan of the EASL is presented in Fig. 2, which shows a number of microbial sampling sites where air and surface samples were collected.

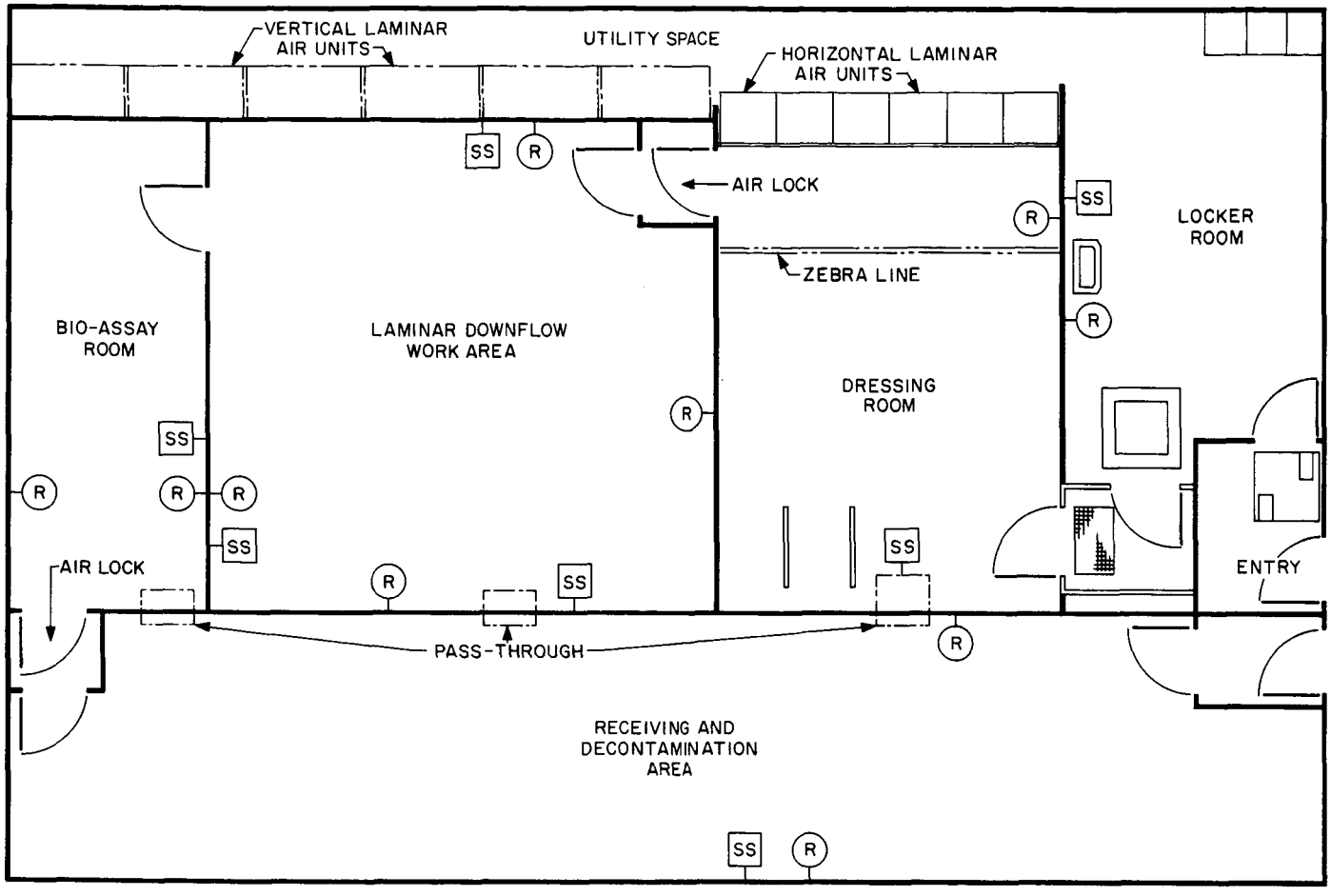


Fig. 2. Location of microbiological sampling sites within the EASL (R: sampling site Reyniers slit sampler; SS: location of tray containing microbial fall-out sampling strips)

The present study was conducted in two phases. Phase I sampling was done during the final stages of construction of the EASL. Strict personnel procedures for entrance, dress, and exit were not enforced (Ref. 5). During Phase I, the velocity of the vertical laminar air flow was varied. During Phase II, the final settings for air-flow velocity, temperature, relative humidity, and pressure were established for both the horizontal and vertical flow rooms.

Microbial contamination accumulating on surfaces. Sterility controls indicated that all of the stainless-steel test strips were sterile. Tables 1 through 3 present the results obtained from the microbial fallout sampling studies carried out during Phase I. A number of interesting observations may be made from the information presented in these tables. It may be seen that both the horizontal laminar air flow room (dressing room) and

Table 1. Results of microbial fallout sampling studies in EASL

Area sampled	Average number of viable aerobic mesophiles recovered per sq ft of surface[a]	
	6-hr exposure	72-hr exposure
Locker room	2323	1092
Dressing room	24 (1219)[b]	10
Laminar flow work area	0	10
Bioassay station	14	29
Receiving/decontamination area	408	600

[a] Five stainless-steel strips assayed to form average.
[b] One strip accidentally contaminated by workman in area. Data from assay of this strip not included in assay.

Table 2. Results of microbial fallout sampling studies in EASL

Area sampled	Average number of viable aerobic mesophiles recovered per sq ft of surface[a]	
	6-hr exposure	72-hr exposure
Locker room	10560	1968
Dressing room	365	182
Laminar flow work area	29	10
Bioassay station	29	19
Receiving/decontamination area	2064	154

[a] Five stainless-steel strips assayed to form average.

Table 3. Results of microbial fallout sampling studies in EASL

Area sampled	Average number of viable aerobic mesophiles recovered per sq ft of surface[a]	
	6-hr exposure	72-hr exposure
Locker room	Not done	58
Dressing room	Not done	10
Laminar flow work area	Not done	0
Bioassay station	Not done	10
Receiving/decontamination area	Not done	855

[a] Five stainless-steel strips assayed to form average.

the vertical laminar air flow rooms (laminar flow work area and bioassay station) had the lowest levels of microbial surface contamination. Furthermore, it would appear from the data in Tables 1 and 2 that the locker room had the highest level of surface contamination. During one of the sampling periods the horizontal laminar flow dressing room failed to meet specifications; i.e., it exceeded a microbial surface contamination of 200 microorganisms per sq ft of surface (Table 2). These increased levels of microbial contamination may be attributed to an increase of personnel in the area during the dioctylphthalate (DOP) "smoke test" demonstrations which were conducted within the vertical laminar flow work area. To witness these tests, spectators (in groups of 10 to 15 people) entered the dressing room through the locker room and viewed the smoke test through a window in the dressing room. This increased number of people, clad in street clothes, probably contributed greatly to the surface contamination in the locker room and the dressing room.

Table 4. Results of microbiological sampling of air within the EASL

Area sampled	Time sample collected	Volume of air sampled, cu ft	Number of viable particles per volume of air samples	
			Average per cu ft	Range per cu ft
Locker room	Not done[a]	—	—	—
Dressing room	Not done[a]	—	—	—
Laminar flow work area	0905–1305	300	0.05	0.03–0.07
Bioassay station	0900–1300	300	0.07	0.03–0.15
Receiving/decontamination area	0850–1250	300	1.75	0.73–2.86

[a] Vacuum system insufficient to support five samplers.

Table 5. Results of microbiological sampling of air within EASL during a dioctylphthalate (DOP) smoke test (area sampled June 19, 1965)

Area sampled	Time sample collected	Volume of air sampled, cu ft	Number of viable particles per volume of air samples	
			Average per cu ft	Range per cu ft
Locker room	Not done			
Dressing room	0940–1440	300	0.003	0.00–0.01
	1440–1450	10	0.00	0.00
	1450–1505	15[a]	2.67	2.40–3.00
	1505–1540	35	0.00	0.00
Laminar flow work area	0945–1445	300	0.003	0.00–0.01
	1445–1450	5	1.00	0.00–5.0
	1450–1500	10[a]	6.70	6.40–7.00
	1500–1545	45[b]	0.60	0.00–3.80
Bioassay station	0950–1450	300	0.00	0.00
	1450–1505	15[a]	5.20	4.25–7.60
	1505–1550	45[b]	0.00	0.00
Receiving/decontamination area	0955–1055	60	0.50	0.00–1.80
	1055–1155	60	0.68	0.00–2.40
	1155–1255	60	0.30	0.00–1.00
	1255–1355	60	0.23	0.00–1.60
	1355–1455	60	1.30	0.40–4.25
	1455–1555	60	1.00	0.20–2.40

[a] Laminar air flow off; room filled with DOP smoke.
[b] Laminar air flow turned back on.

The last observation to be made from Tables 1 through 3 concerns the degree of surface contamination attributable to human handling of the surface sampling strips. Handling of a sample strip (Table 1) by a construction worker increased the average surface contamination per square foot approximately 50 times (24 to 1219 microorganisms per sq ft). On another occasion, a complete sampling experiment was invalidated through several mishaps, such as construction tools being mislaid on trays of sampling strips. This point is mentioned to emphasize that personnel education and application of facility rules is essential to proper operation of the EASL. Had the accidents gone undetected, increased levels of microbial surface contamination would have been recorded. The unexplained increased level of surface contamination might then have initiated corrective action resulting in an unnecessary delay of facility certification.

Air sampling studies. Tables 4 and 5 present the results of microbiological sampling of air within the EASL. Table 4 indicates that the number of airborne viable particles in a vertical laminar flow air stream is extremely low (*ca.* 0.05 viable particles per cu ft of air), considerably below the acceptable level of 2.0 viable particles per cu ft of intramural air (Ref. 5). Table 5 contains the air-sampling results obtained during a DOP smoke test. These results need little explanation. During the period when the laminar air flow was turned off (*ca.* 1450 to 1505), the number of airborne viable particles increased sharply. However, when the laminar air flow was resumed, the number of airborne viable particles decreased and stayed at an apparently normal level that appears to be very low. The fluctuation of the number of airborne viable particles during another DOP smoke test is illustrated in Fig. 3; the data is self-explanatory. Results such as these have been obtained in repeated samplings during other DOP smoke tests.

b. Phase II sampling. Tables 6–8 contain the results obtained during the period when the final settings for facility air flow, temperature, pressure, and relative humidity were established.

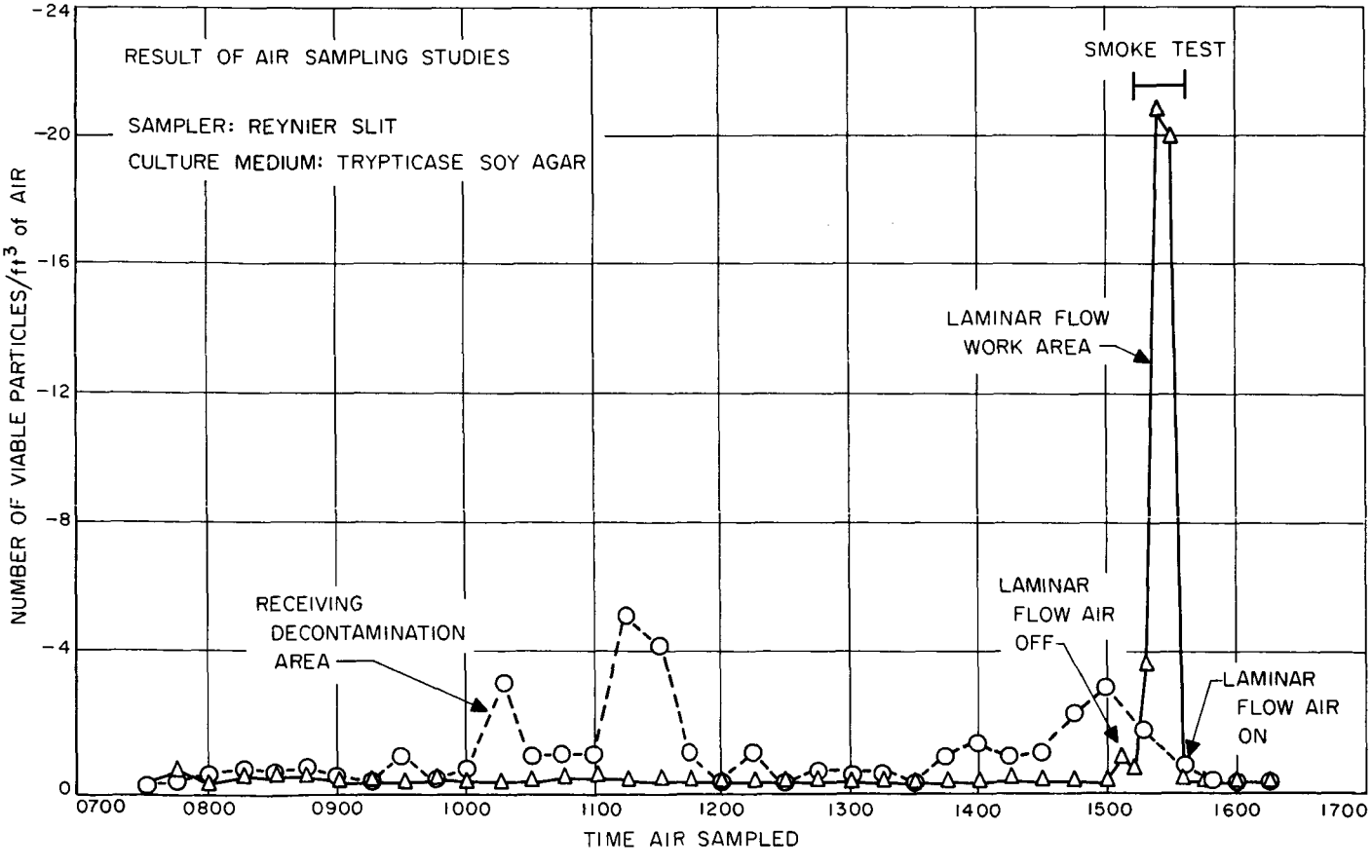


Fig. 3. Air sampling studies within EASL during a "smoke test"

Table 6. Results of microbial fallout sampling studies in the EASL: final setting of facility air flow, temperature, pressure, and relative humidity (strips placed in EASL June 30, 1965)

Area sampled	Average number of viable aerobic mesophiles recovered per sq ft of surface[a]				
	Exposure				
	6-hr	24-hr	48-hr	120-hr	144-hr
Locker room	230	4224	1680	1056	1932
Dressing room	10	10	19	0	58
Laminar flow work area					
Site 1	0	0	19	10	19
Site 2	0	0	10	0	0
Site 3	12 (12,336)[b]	0	0	10	0
Bioassay station	0	0	0	19	0
Receiving/ decontamination area	0	172	134	67	96

[a] Five stainless-steel strips assayed to form average.
[b] One strip was accidentally touched by a worker. Count for this strip was 12,336 microorganisms per sq ft. Violated strip results not included in average.

Table 7. Results of microbiological sampling of air within the EASL: final settings for facility air flow, temperature, pressure and relative humidity (area sampled June 30, 1965)

Area sampled	Time sample collected	Volume of air sampled, cu ft	Number of viable particles per volume of air samples	
			Average per cu ft	Range per cu ft
Locker room	0910–1510	360	0.92	0.00–9.00
Dressing room	1120–1520	240	0.01	0.00–0.20
Laminar flow work area				
Site 1	0905–1505	360	0.006	0.00–0.01
Site 2	0910–1510	360	0.003	0.00–0.01
Bioassay station	0915–1515	360	0.009	0.00–0.03
Receiving/ decontamination area	0905–1505	360	0.11	0.00–1.60

The results shown in Table 6 are similar to those previously discussed (Tables 1–3). When contamination from human handling is eliminated, the level of surface contamination within the laminar flow rooms remains well within the microbiological level necessary for certification. This also holds true for the air sampling study results presented in Table 7. Table 8 contains results that indicate the intramural air of the EASL also meets specifications for a class 100 clean room (Ref. 8) during the sampling period.

Table 8. Results of total particulate sampling (Royco) of air within the EASL: final setting for facility air flow, temperature, pressure, and relative humidity (area sampled June 30, 1965)

Time laminar flow work area sampled with Royco instrument	Volume of air sampled, cu ft	Particulate count per cu ft of air	
		0.3-μ-diameter and larger particles	0.5-μ-diameter and larger particles
0921–1021	0.60	0	0
1021–1119	0.50	0	0
1119–1129[a]	0.10	10	Not scanned
1129–1139[a]	0.10	Not scanned	10
1139–1149[a]	0.10	10	Not scanned
1149–1159[a]	0.10	10	Not scanned
1159–1259	0.60	0	0
1259–1359	0.60	0	0
1359–1409	0.10	0	Not scanned
1409–1419	0.10	Not scanned	10
1419–1513	0.57	0	0

[a] Welding technician was in the room during this period.

3. Discussion

From the results obtained, it is the technical judgment of the EASL cognizant microbiologist that under the test conditions the levels of microbial contamination within the EASL are below those considered acceptable for microbiological certification of a bioclean facility (Ref. 5). However, certain important conditions remain to be investigated. The results obtained in this study represent data from a limited number of samples collected during a time when the test facility was not in normal operation. The influence of operating personnel on the levels of microbial contamination within EASL remains to be determined. Thus, the results contained in this report must be regarded as provisional and preliminary. For example, during the last series of samplings (June 30 to July 6, 1965) the air-conditioning system was inoperative for several days (July 5–7, 1965). During this period, temperature within the facility reached the inoperable levels of 84 to 89°F, with comparable fluctuations in relative

humidity. The effects of environmental exposure on surface contamination have been thoroughly investigated (Refs. 9 through 15). The fluctuations of temperature and relative humidity within the EASL may have had some effect on the subsidence of surface contamination. At this point, such an observation can only be speculative and requires considerable study and documentation for confirmation. In the absence of confirmative data, the facility should be regarded as operational, and the microbiological support and research work should be allowed to proceed as rapidly as possible.

Finally, the value of personnel training cannot be overemphasized. The few mishaps noted in this study provide at least three conclusions:

1. Handling of sterile objects by improperly dressed personnel can increase the levels of microbial contamination surfaces.
2. The microbiological sampling techniques used in EASL appear to be fairly sensitive and reliable. When contamination is present the sampling techniques appear adequate to allow its detection.
3. Proper operating procedures are necessary and require a training program for proper implementation.

4. Conclusions

The preliminary results obtained in this study indicate that the EASL facility can be certified microbiologically as meeting the requirements imposed on any bioclean facility (Refs. 5 and 8). It must be emphasized that the EASL facility was unoccupied during a large portion of the study. The influence of operating personnel on the levels of microbial contamination within EASL remains to be determined.

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ABSTRACTS

• Reed, L., "Ethylene Oxide Sterilization Studies," *Space Programs Summary No. 37-22, Vol. IV*, pp. 6–8, Jet Propulsion Laboratory, Pasadena, California, August 31, 1963.

Ethylene oxide vapors have been used in the terminal sterilization of the *Ranger* series of spacecraft; during tests of spacecraft components, occasional skips or nonsterile results have appeared. Under a contract with Dynamic Science Corporation of South Pasadena, California, JPL is conducting investigations which it is hoped will shed light on the causes of these skips. The present areas of investigation are:

1. The effect of dust on ethylene oxide sterilization success and the resistance of isolated dust microorganisms.

2. Specification of a satisfactory test organism for use with ethylene oxide.

For the dust studies, floor dust from the JPL Spacecraft Assembly Facility was used. In Phase I of the study, the dust was evaluated physically, and with respect

to its microbiological population. In Phase II, the resistance of dust samples was compared to the resistance of standard *Bacillus subtilis*, var. *niger* spores under various conditions of exposure. The conclusions of this study were as follows:

1. Floor dust is more difficult to sterilize than pure culture of microorganisms.
2. The skips which have appeared in ethylene oxide sterilization tests are due in large part to a highly variable and uncontrolled microbiological population in the dust.
3. To increase the reliability of decontamination with ethylene oxide, exposure conditions must be as severe as can be tolerated.

In view of the high resistance of floor dust to ethylene oxide sterilization, an experimental program was designed to isolate the most resistant microorganisms from the dust to determine:

1. Whether the resistance of the microorganisms is dependent on the organisms themselves or is a shielding effect of the dust.
2. If the former, whether one of these isolants would prove a more satisfactory test organism for ethylene oxide sterilization than the organisms presently in use. After evaluation, the most satisfactory organism appears to be a sample of *Bacillus subtilis* var. *niger* spores in diatomaceous earth obtained from the U.S. Army Biological Laboratories, Fort Detrick, Maryland. These spores are most suitable because:
   a. They show a high level of resistance.
   b. Their colony pigmentation makes them easy to identify.
   c. They are relatively easy to maintain and reproduce.
   d. They germinate rapidly and at a high percentage level.
   e. A large body of information is available about the effect of various conditions of exposure to ethylene oxide on the viability of this organism.

The floor dust isolants showed a much lower resistance to ethylene oxide in the pure culture state.

- Reed, L. L., "Sterilization Studies," *Space Programs Summary No. 37-24, Vol. IV*, pp. 5–7, Jet Propulsion Laboratory, Pasadena, California, December 31, 1963.

To demonstrate the reliability of a spacecraft sterilization cycle, reliable analytical techniques are necessary. Since the conditions of analysis are much different from previous sterility test conditions, considerable research is necessary in order to develop proper test techniques. The research task has been divided into four phases as follows:

1. The testing of methods for the detection of small numbers of viable microorganisms. This phase includes evaluation of various methods of micronization and disintegration of materials and evaluation of the possibilities of other detection mechanisms such as staining, electrophoresis, autoradiography, and electron-spin resonance.
2. The further development of Phase 1 techniques to increase efficiency and to give quantitative reliability figures.

3. The demonstration of the applicability of the developed method or methods to the assay and certification of spacecraft sterility.
4. The documentation and evaluation of the reliability and efficiency of these methods.

In the microbiological literature, it is established that microorganisms exposed to the sterilizing influence of heat die at an approximately logarithmic rate; it is apparent, then, that if the number of microorganisms on a spacecraft or other body can be decreased, the sterilizing time can also be decreased.

This line of reasoning indicates that assembly of Mars landing capsules or spacecraft in clean rooms may help to reduce the microbiological load and thereby relieve the burden on the terminal sterilization cycle. To evaluate the real benefits of clean room assembly of spacecraft, a program will be initiated to obtain a microbiological profile of present industrial clean rooms.

With the realization that the present dry heat terminal sterilization cycle is a rigorous environment for spacecraft hardware, a feasibility study has been undertaken to determine whether some pretreatment might be applied to reduce the thermal resistance of bacterial spores, the most resistant life form with which we must deal. The present study will consist of a literature search and interviews of appropriate individuals to evaluate the desirability of further investigations in this area.

- Irons, A., "Development of a Biological Sterility Indicator for Dry Heat Sterilization," *Space Programs Summary No. 37-29, Vol. IV*, pp. 7–8, Jet Propulsion Laboratory, Pasadena, California, October 31, 1964.

This task calls for the development of a biological sterility test system for dry heat in accordance with certain specified constraints. In general, this will include the use of an organism sufficiently resistant to dry heat, the determination of the proper substrate and carrier, the establishment of sufficient survival-kill data to indicate and guarantee an efficient sterilization cycle at the specified time and temperature, and the establishment of a reasonable form of the system for practical use.

The Wilmot Castle Company, under contract to JPL, is attempting to develop a biological sterility indicator for a dry heat sterilization cycle of 135°C for 24 hr in an atmosphere of dry nitrogen.

- Irons, A., "Evaluation of Microbiological Filters for Liquids and Gases," *Space Programs Summary No. 37-29, Vol. IV*, pp. 16–18, Jet Propulsion Laboratory, Pasadena, California, October 31, 1964.

The assembly, test, sterilization, and launch of a sterile spacecraft will require filters of high microbiological efficiency. Filters will be required for air-conditioning systems, pressure equalization across the spacecraft biological barrier, and perhaps for gases and heat-labile liquids aboard the spacecraft. The Wilmot Castle Company, under JPL contract, is studying the microbiological efficiency and reliability of commercially available filters. Controlled challenge aerosols of known organisms will be used, and, if the filters are not 100%

efficient, an attempt will be made to determine the cause of failure. Details of the test procedure and failure analysis are discussed.

- McDade, J. J., "An Experimental Study of Sterile Assembly Techniques," *Space Programs Summary No. 37-29, Vol. IV*, pp. 13–16, Jet Propulsion Laboratory, Pasadena, California, October 31, 1964.

  The use of dry heat appears to be the method of choice for the terminal sterilization of a planetary landing spacecraft. However, at present, certain spacecraft items cannot withstand the dry heat cycle without serious degradation in reliability. Therefore, if an entirely heat-stable spacecraft is not possible, reliable sterile assembly techniques must be developed and be ready for use to incorporate heat-labile components into sterile spacecraft.

  The present study is being conducted by Lockheed Missiles and Space Corporation to evaluate the potential of obtaining a sterile electronic unit through use of a procedure in which each assembly step takes place in a glove box containing an ethylene oxide atmosphere.

  A small electronic module will be assembled in the glove box. The microbiological efficiency of the assembly operation will be monitored and evaluated to assure that sterility is maintained. In addition, the operation itself will be studied to determine the quality of the product and requirements for special tools or devices and to assess the extra time required. Details of the procedures to be used are discussed.

- McDade, J. J., "The Microbiological Profile of Clean Rooms," *Space Programs Summary 37-29, Vol. IV*, pp. 8–13, Jet Propulsion Laboratory, Pasadena, California, October 31, 1964.

  The probability of obtaining a sterile spacecraft by any sterilization procedure is enhanced by keeping the initial microbial contamination to a minimum level. Reduced levels of microbial contamination may be obtained through proper environmental control of the spacecraft assembly area during assembly and checkout. Such an environment can also afford the secondary benefit of improved reliability.

  At present, only a limited amount of data exists concerning the microbiological contamination of industrial clean rooms. Therefore, a microbiological sampling study was initiated to determine the levels of viable particulate contamination that exist within the different classes of clean rooms during various conditions of operation and activity. The nature of the study is explored and sampling techniques are discussed.

- McDade, J. J., "An Experimental Study of Sterile Assembly Techniques: Midterm Progress Review," *Space Programs Summary No. 37-31, Vol. IV*, pp. 37–43, Jet Propulsion Laboratory, Pasadena, California, February 28, 1965.

  Work to date has been performed in a glove box in an ethylene oxide–Freon 12 gaseous environment. The glove box has been modified to provide a workable and efficient system. A toxicity problem has been detected with some of the items

subjected to sterility testing. From the tests conducted to date, both hand and dip soldering processes appear to be feasible in an ETO environment. However, on occasion, more than one dip was necessary to completely fill the tracks on printed circuit cards. Also, hand soldering in ETO must be done with a soldering iron having a sealed heating element to prevent failure of the iron. It should be noted that some of the items inoculated with spores of *Bacillus subtilis* var. *niger* and processed as sterility check items were contaminated. An explanation was offered, and corrective action was taken; however, the efficiency of this corrective action remains to be determined. Mechanical operations (e.g., staking and nut and bolt connections), epoxy bonding, potting, and breadboard assembly also appear to be feasible in an ETO atmosphere.

- Reed, L. L., "Microbiological Analysis Techniques for Spacecraft Sterilization," *Space Programs Summary No. 37-32, Vol. IV*, pp. 35–42, Jet Propulsion Laboratory, Pasadena, California, April 30, 1965.

  Methods for the detection of small numbers of microorganisms have been investigated and evaluated. From the results, it is concluded that the most reliable means of differentiating between viable and nonviable microorganisms is the use of the culturing (growth) technique. The physical methods (staining, electrophoresis, autoradiography, and electron spin resonance) did not reliably differentiate between viable and nonviable cells as required in sterility testing. In addition, they present numerous problems that make them impractical for general use.

  Of the four techniques studied for comminuting solid materials, it must be concluded that the drill technique gives the best recovery of viable cells. However, the drill technique as used in this investigation still leaves much to be desired since maximum recovery is only at the 1% level. The reliability and applicability of this technique to a broader range of solid materials are to be evaluated.

  The data collected in this investigation indicate that there are several problem areas which must be resolved; therefore, continuing investigations to improve techniques for the recovery of viable cells from solids are required if practical and reliable sterility test procedures are to be developed to support certification of spacecraft sterility.

- McDade, J. J., Irons, A. S., and Magistrale, V. I., "A Microbiological Survey of Hughes Aircraft Company Facilities Involved in the Assembly and/or Testing of Surveyor Spacecraft," *Space Programs Summary No. 37-32, Vol. IV*, pp. 25–35, Jet Propulsion Laboratory, Pasadena, California, April 30, 1965.

  The study reported here represents an accelerated program initiated by JPL at the request of NASA to obtain an estimate of the levels of microbial contamination present within the HAC facilities involved in the assembly and/or testing of the *Surveyor* spacecraft. The requested information was obtained together with other information which should serve as guidelines for future microbiological surveys.

Because of the urgent need for the results, a temporary lack of microbiological facilities at JPL, difficulties in transporting a large number of samples to the U.S. Public Health Service Field Station at Phoenix, Arizona, and personnel limitations, only a limited number of microbiological samples were collected. Therefore, the results obtained in this study must be regarded as preliminary information. Definite conclusions cannot be drawn from the data obtained. However, a few general statements can be made concerning the surface-sampling phase of the study:

1. The total number of viable aerobic mesophiles recoverable from test surfaces appeared to stabilize and fall within the range of 10^3 (1000 to 9999)/ft^2 of surface over the study period.

2. The total number of viable aerobic spore-forming mesophiles recoverable from a limited number of the test surfaces appeared to stabilize and fall within the range of 10^2 (100 to 999)/ft^2 of surface over the study period.

The number of airborne viable particles appeared to be dependent, at least in part, on the number of personnel and type of activity occurring within the area. However, no conclusions should be made from the very limited amount of air-sampling data obtained during this survey.

V. SPACE SCIENCES

The Space Sciences Division supports the scientific aspects of the space exploration program and carries out independent work ranging from research and development through fabrication and testing. In the sterilization area, the Division is concerned with providing sterilizable scientific instruments for planetary landing missions. Current programs include the development of instruments and associated sensors that can withstand sterilization and planetary landing environments. Significant results obtained during investigations are reported in this section; previously published findings are abstracted.

A. Sterilizable Image Sensor, *L. R. Baker*

The purpose of this investigation is the development of an image sensor capable of withstanding gas and dry heat sterilization and the extremely high shock-levels expected during a Mars landing.

The image sensor presently used in space photography is the vidicon, a rugged, reliable, fairly sensitive device that has been extensively used in commercial and industrial television for over fifteen years. Since commercial vidicons were primarily used in stable environments, they were not required to withstand severe vibration, shock, or high temperatures. With the advent of the space program, vidicons used in space-borne experiments were required to survive vibrational launch environments, and in some cases were required to survive heat sterilization.

For programs which may land experiments on Mars, advances in vidicon design must be made to ensure the sensor's surviving higher sterilization temperatures as well as landing impacts on the order of 3000 to 5000 Earth *g*.

To improve the vidicon to this extent, new concepts for electron gun and tube envelope design and new techniques of photoconductor and signal electrode application must be developed. The Radio Corporation of America Electron Tube Division at Lancaster, Pennsylvania, has contracted to develop a ruggedized, sterilizable vidicon with the following characteristics:

1. Electron gun using electrostatic focusing and deflection.
2. Resolution comparable to commercial vidicons.
3. Sensitivity comparable to commercial vidicons.
4. Operation in the slow-scan mode (image storage).
5. Spectral response in the visible region.
6. Dynamic range and signal-to-noise ratio comparable to commercial vidicons.
7. Low-power filament.
8. Capable of surviving gas and dry heat sterilization.
9. Capable of surviving launch environment shock and vibration.
10. Capable of surviving ± 3000-*g* terminal sawtooth with a 3-millisec rise time.

The following problem areas in the development of the above device are expected:

1. Photoconductors are essentially semiconductors and are therefore sensitive to heat. Storage at high temperatures can cause permanent damage to the crystalline structure of the photoconductor or shifts in the operating characteristics.
2. Photoconductors may become detached from the substrate under high-*g* impacts. Investigations in this area have not yet been carried out, but a potential problem is recognized.
3. The present vidicon glass envelope cannot tolerate stresses which would be applied under high-*g* loading; to withstand high-*g* impacts, a new envelope material such as a ceramic must be employed. So far, vidicons have not been made with other than a glass envelope.
4. Critical components such as the heater-cathode structure must undergo extensive design changes and rigorous testing to assure that electron gun components can withstand high-*g* impacts.

At the conclusion of the program, the newly developed vidicon will be tested further to establish new design goals and fabrication techniques. An image sensor with the described features should perform satisfactorily on nearly any type of mission assignment.

At this writing, the program has been underway at RCA for four months. The results thus far indicate that the photoconductor has the capability of surviving both heat and gas sterilization.

B. Sterilizable RF Crystals, *F. T. Barath*

The objective of the current program is to establish a source of supply for high-sensitivity radio frequency crystal detectors and high-performance crystal mixers for space applications. The crystals should be heat- and gas-sterilizable with demonstrated high reliability and suitable for use in scientific instrumentation operating at wavelengths shorter than about 15 mm (frequency of 20 Gc).

Prime emphasis is on the development of RF mixer crystals. Mixer crystals, essentially devices to detect and measure microwave signals, are used in a variety of RF or microwave instruments such as radar and radiometric receivers. The crystals are frequency converters inasmuch as incoming microwave energy is converted to a much lower frequency by heterodyning with a locally generated microwave source within the nonlinear crystal diode element. The output frequency or intermediate frequency (IF) is the difference between the signal frequency and the local oscillator frequency and is normally chosen to be low enough (in the megacycles) to be amplified in conventional low-noise amplifiers.

Secondary emphasis is on crystal detectors, straight square-low rectifiers that yield a DC signal proportional to the RF energy incident upon them. They are noisier than mixers but are often used in place of mixers when there is adequate signal strength or time for long integration and when extreme system simplicity is essential (no local oscillator required).

As both mixer and detector crystals are used on RF/microwave receivers associated with radars and radiometers, the types of spacecraft that would carry such instruments include capsules (radar altimeters, atmospheric constituent determination instruments) and orbiters and flybys (radar and passive microwave imagers, microwave spectrometers, and surface and subsurface temperature mappers).

Detector and mixer crystal diodes have been important devices since the advent of radar in World War II. Gradual improvements have been made in the noise figures of mixers and the minimum detectable signal of detectors. Concurrently, the reliability of both diodes has also been improved.

Two recent fabrication techniques indicate substantial improvements in noise figure, burnout, bandwidth and mechanical reliabilities for these diodes. The techniques consist of the use of epitaxial silicon layers and fused-glass pin-beads in coaxial diodes. The epitaxial silicon gives the designer increased control of the RC time constant of the junction, resulting in improved noise figure, burnout, and sensitivity of detector diodes. The glass pin-bead provides a marked improvement in mechanical reliability and bandwidth.

The first detector and mixer crystals were made from highly doped silicon and a tungsten point. In England during World War II, it was found that a heat treatment of 15 min or more at a temperature of approximately 1000°C gave the silicon then used a substantially superior surface for the fabrication of a point-contact mixer. This improvement resulted from the formation of a relatively high-resistivity layer upon the low-resistivity substrate. Because the impurity distribution in this layer is determined by the diffusion constants for the dopant involved, it is not possible to obtain the most desirable impurity distribution within the layer by means of this process. In 1960, a new processing technique suited to the production of thin, high-resistivity layers on low-resistivity substrates was discovered. This method, epitaxial growth, allows the designer to select layer thickness, doping profile, and layer doping. The designer then can determine the optimum layer characteristics for optimum noise figure or sensitivity at any given frequency. This improvement is greatest at frequencies above 10 Gc, where the sensitivity or noise figure of the diodes is limited by the RC time constant of the junction rather than by the rectification efficiency. The importance of the ability to optimize the material increases with an increase in frequency.

High-temperature pin-beads for coaxial design diodes are a second design improvement incorporated in the current fabrication technique. Coaxial design diodes that employ a metallized ceramic pin-bead to withstand sterilization temperatures exhibit mediocre reliability and definitely constricted bandwidth. This is attributed to the relatively high dielectric constant and the rather inferior mechanical reliability of ceramic metallizing in the required shape. By employing a fused-glass pin-bead, which has a much lower dielectric constant and superior strength, the bandwidth as well as the mechanical strength of the diodes can be greatly improved. Because the glass can be melted into voids, it is easier to fabricate an undercut pin-bead with its improved bandwidth. The design has been proven in a series of diodes designed for operation above 15-mm wavelengths (20 Gc).

Present commercially available mixer and detector crystals for wavelengths down to about 3 cm (frequencies

up to 10 Gc) are fabricated with the described techniques. They exhibit satisfactory electrical characteristics and reliability and the capability of withstanding heat and gas sterilization as well as spacecraft environmental conditions.

However, as most if not all microwave instruments proposed for future spacecraft are to operate at wavelengths shorter than 3 cm, a development program was initiated to obtain sterilizable crystals in shorter wavelengths incorporating the advanced techniques used for longer-wavelength crystals. Three types of mixer crystals and two types of detector crystal diodes appear adequate to meet the foreseeable needs of potential instruments. The required electrical characteristics of the mixer diodes are presented in Table 1, and the detector diodes are listed in Table 2. The objectives of the development program are to fulfill these requirements as well as heat and gas sterilization, environmental, and reliability requirements.

The approach for the development of the five diodes listed in Tables 1 and 2 will consist of perfecting the following:

Diode 1
: A diode with the same mechanical and physical dimensions as the standard 1N 26 coaxial diode but designed for a center frequency of 22 Gc and with a glass pin-bead as well as epitaxial silicon.

Table 1. Mixer diode characteristics

Diode	Frequency, Gc	Bandwidth, %	Conversion loss, db	Noise figure, times at 16 Gc	Noise figure, db	IF impedance, ohm
1	22	±10	6.5	1.3	8.8[a]	250 ±50
2	35	±10	6.5	1.4	9.0[a]	250 ±50
3	75	±10	10.0	3.0	15.3[b]	250 ±50

[a] Calculated with the inclusion of an IF amplifier noise figure of 1.5 db and bandwidth of 100 Mc centered at 60 Mc.
[b] Calculated with the inclusion of an IF amplifier noise figure of 3.0 db and bandwidth of 10 Mc centered at 1000 Mc.

Table 2. Detector diode characteristics

Diode	Frequency, Gc	Bandwidth, %	Figure of merit, M	Video impedance, k ohms
4	75	±15	180	10 ±5
5	90	±15	150	10 ±5

Diode 2
: A diode identical in outline to the standard 1N 53 coaxial diode but also with a glass pin-bead. Upon completion of the electrical design, the optimum epitaxial silicon will be determined for minimum noise figure and maximum burnout resistance.

Diode 3
: A diode that will probably be a ridged, integral waveguide structure with the silicon element sealed with glass windows.

Diode 4
: Detector diodes similar in structure to (3) but tuned to the individual frequencies and with the appropriate impedance transformers.

Diode 5
: There is the possibility of building a single structure with sufficiently wide characteristics to cover the frequency range of diodes 4 and 5 with minor adjustment.

Commercial diode manufacturers have expressed considerable interest in this program as it presents the opportunity of developing a technology for short-wavelength, high-performance, high-reliability RF crystal diodes. Such devices would have appreciable commercial value since the inferior-quality diodes presently used in the development and construction of RF instrumentation affect the total design and result in reduced performance or increased complexity.

A set of specifications for the development of the required mixer and detector diodes has been generated, and a number of companies responded with detailed quotations. Some preliminary discussions were held with one of the potential vendors to better understand the state of the art. No further activity has been undertaken, however, and none is planned for the near future. It was determined that the application of these devices is rather remote (1973 or later) and that the available funds could be used to greater advantage in other areas of the sterilization program.

C. *Sterilization of Atmospheric Instruments,*

L. D. Bowman

The evolution of gas chromatography into one of the most powerful analytical tools in chemistry makes it a prime contender for use on missions intended to investigate planetary composition. Briefly, a gas chromatograph (GC) is a device that separates a gas sample into its constituents by circulating the sample through a sorbent

such as a molecular sieve or silica gel. The time it takes each constituent gas to pass through the sorbent is peculiar to that particular gas and, hence, identifies it. The magnitude of response of the instrument to each detected gas serves as a measure of quantity of each constituent in the sample.

Since gas chromatography can be used to analyze any material that can be converted into the stable gaseous state, it is ideally suited for both planetary atmospheric and surface analyses. As a surface analysis instrument, the gas chromatograph can obtain both geological and biological information.

As the gas chromatograph will be used for planetary landings, it will have to be sterilizable and capable of surviving high-g impact. The development of a flight-type gas chromatograph has been directed toward perfecting the individual components so that they can withstand both the required sterilization and three mutually orthogonal planes of impact at 10,000 g.

Evaluation of the electronic components in the GC is being carried out primarily by the JPL Component Parts Qualification Group (Section II). Preliminary investigations indicate that tantalum and metallized mylar capacitors will need more extensive evaluation and possible development to assure that they meet the environmental requirements. Mylar capacitors, although considerably larger, are an acceptable electrical substitute for metallized mylar and are currently being used in the GC. The lower-mass capacitor is highly desirable for an impact-resistant instrument. No other electronic components appear to present unusual environmental problems.

All mechanical GC components have to be specially developed for a flight instrument because commercial components either do not exist or will not pass either high-g impact or sterilization tests. The primary problem has been to design components that will not leak after impact or during and after the heat sterilization cycle. Leaks in the GC gas-handling components can, of course, alter instrument performance, but, further, they could admit gases from the ethylene-oxide sterilization cycle which would contaminate the system. The fabrication of a new sample injection valve, carrier gas regulator, and cross-section ionization detector with special consideration to the leakage problem has been completed. In addition, Teflon-insulated hookup wire, coax, and high-temperature lacing cord have been used to fabricate the instrument cables. Teflon has also been substituted for other synthetic materials such as that used in the connector potting boot.

The effects of heat sterilization on the H_3 ionization source used in the detector have been the subject of a NASA-funded investigation by James Lovelock. His report indicates that although the source life is shortened at elevated temperatures, it would not be decreased appreciably during the heat sterilization cycle.

Early in the development of the gas chromatograph it appeared that the dynamic capacitor would require particular attention because of the expected effects of the heat cycle on the capacitor's contact potential. The capacitor was developed to perform satisfactorily after sterilization by a manufacturer under contract to JPL. (The detailed procedure is discussed in the following section.)

A prototype Mars atmospheric gas chromatograph has been fabricated. Upon completion it will receive the complete type-approval sterilization cycle. The design of the instrument, its development, and results of the tests will be reported at a later date.

D. A Heat-Sterilizable Dynamic Capacitor,

John R. Locke

The dynamic capacitor is used as a modulator for carrier-type electrometer applications. It converts low-level direct currents (as low as 10^{-15} amp) into a sinusoidal voltage at the vibrational frequency. The device has been developed during the past two years by the Kinelogic Corporation[1] under contract to JPL. During the past six months, Kinelogic's efforts have been directed at perfecting the device so it could undergo heat sterilization without adverse effects.

A similar device, but more than 10 times as heavy, was used in the solar plasma analyzer flown aboard *Mariner II*. The dynamic capacitor has many additional potential space applications such as in gas chromatography, mass spectroscopy, low-level nuclear dosimetry, ultraviolet and infrared measurements, and any other experiment requiring the capability of measuring very small currents.

In addition to flyby and orbiter uses, recent high-g impact testing indicates that the capacitor could be used aboard a hard-landing capsule.

The conversion of a small direct current into a sinusoidal voltage is accomplished by sinusoidally varying the separation distance between two parallel circular metal disks charged with the current being measured. One of the disks is fixed in position and the other is supported

[1] 29 S. Pasadena Ave., Pasadena, Calif.

only at its center. The disk supported at its center is free to vibrate in its fundamental mode at its natural resonant frequency when electrostatically driven by a voltage of that frequency. The voltage is provided by a tracking oscillator.

The dynamic capacitor can be characterized by six parameters: the natural resonant frequency, conversion efficiency, input impedance, output impedance, transducer pickup voltage, and contact potential.

The natural resonant frequency is determined by five factors: the mode constant (a constant dependent on the manner in which the vibrating disk is supported), the modulus of elasticity of the vibrating disk, the density of the disk, its thickness, and its effective radius. The frequency of the output voltage of the dynamic capacitor is that of the natural resonant frequency.

The conversion efficiency is a measure of how effectively the dynamic capacitor can convert dc voltage at the input to ac voltage at the output.

The input and output resistance and capacitance are, respectively, determined by insulation resistance and disk spacings.

The pickup transducer voltage is a feedback voltage which is independent of the input signal to the dynamic capacitor. It is provided for the tracking oscillator that oscillates at the resonant frequency of the vibrating disk and provides the electrostatic drive voltage. The transducer voltage is developed by utilizing the variable reluctance path of the vibrating disk and a permanent magnet and by a coil to detect the changing magnetic flux.

The final parameter, contact potential, is the result of dissimilarity of metals and can be depicted (for purposes of circuit analysis) as a virtual battery connected between ground and the vibrating disk. Although this voltage is typically less than 100 mv, its stability as observed at the electrometer output is of great importance in determining the lowest-level current a vibrating-reed electrometer can reliably measure.

The six previously described parameters are all thermally sensitive. Changes of temperature cause expansion and contraction, thus affecting spacing, stress patterns, and geometry. For example, a change in the 1-mil spacing between the fixed and vibrating plates will change the input and output capacitances and conversion efficiency; a change in the stress on the vibrating-disk support can change the mode constant and thereby the resonant frequency. Changes in temperature affect the modulus of elasticity of the vibrating disk, the permeability of the magnetic path of the pickup voltage transducer, and the field strength of the permanent magnet. These areas, it was believed, might be adversely affected by heat sterilization. Testing performed by Kinelogic indicated this was not the case.

The contact potential, however, did present a problem. It was found that when the dynamic capacitor was heated, cooled, and then returned to its original temperature, the contact potential would assume a new magnitude. In conjunction with this, it has also been observed that a larger contact potential magnitude causes a corresponding increase in the thermal coefficient ($\Delta V_0/\Delta T$). In light of the earlier discussion regarding contact potential and its effect on the output voltage of the electrometer, this is an intolerable condition if the lower-level output voltages from the electrometer are to be meaningful.

Kinelogic has completed its study of the contact potential problem. Experimentation with an unsealed unit in a vacuum chamber indicates that Kinelogic has achieved a successful control procedure based on the following two basic considerations:

1. Contact potential results because of difference in work functions when dissimilar metals are in contact.
2. The work function of a metal is strongly influenced by its surface condition.

To obtain a homogeneous surface, the disks and adjacent assembly were given a gold finish. Kinelogic concentrated on the development of controlled cleaning procedures, electrochemical gold plating, and thin-film vacuum gold deposition to achieve physical and chemical uniformity of the finish.

It has been experimentally determined that one of the most important factors affecting the magnitude and stability of contact potential is the degree of outgassing of the assembled dynamic capacitor. Contact potential modification is attributable to the modification of the work function of the surfaces of the disks and adjacent assembly through gas adsorption. The quantity of adsorbed gas varies with the temperatures to which the dynamic capacitor is subjected, and the contact potential changes correspondingly. The outgassing procedure employed consists of heating the assembled unit to 140°C and pulling a continuous vacuum (maintained at less than 1 micron of mercury) for a minimum of 50 hr.

An outline of the procedure used by Kinelogic for controlling the contact potential is as follows:

1. Electrochemical plating.
   a. Ultrasonic cleaning.
   b. Vapor degreasing (using trichloroethylene).
   c. Electrocleaning (using Oakite 90).
      1) Emulsification.
      2) Electrotransport suspension.
   d. Water rinse (for removal of salts).
      1) Tap water.
      2) Deionized water.
   e. Acid dip (for removal of oxides).
   f. Nickel plate (gold will not plate directly on stainless steel).
   g. Water rinse (removal of salts).
      1) Tap water.
      2) Deionized water.
   h. Gold plate (Orosene 999 plating solution).
   i. Tap water rinse.
   j. Dry with dry nitrogen.
2. Vacuum deposition (performed immediately after gold plating).
   a. Desiccate (using phosphorous pentoxide).
   b. Ionic bombardment cleaning.
   c. Deposition by evaporation.
      1) Vacuum lower than 1 micron of mercury.
      2) 24-carat gold used.
3. All gold-finished parts stored in a desiccator until assembly.
4. Assembly; avoid direct handling of gold-finished parts.
5. Post-assembly.
   a. Evacuate for a minimum of 50 hr.
      1) Temperature: 140°C.
      2) Pressure: 1 micron of mercury or less.
   b. Vacuum tube sealed.

E. Sterilizable Solid-State Memory System,

R. H. Nixon

A sterilizable, solid-state memory system that will withstand the high-g impact of hard landing is presently under development. The memory system will be part of the data automation equipment that processes the planetary science data prior to transmitting it to Earth. High-capacity memories without the hard-landing requirement are also under consideration.

1. Design Criteria

Criteria for a hard-landing capsule memory include the following:

1. Meeting the environmental characteristics including sterilizability and shock.
2. Low-power operation.
3. Modest operating speed.
4. Minimum weight.
5. Very high reliability.

2. Problem Areas

Significant problem areas subject to survey, investigation, and development are as follows:

1. Selecting a memory element that can withstand sterilization and shock.
2. Selecting a packaging scheme for this memory element. (Available packaging materials and techniques may strongly influence the selection of the memory element.)
3. Designing reliable, low-power electronic circuits. (Most commercial and military memories have emphasized high-speed operation to the detriment of low power consumption.)
4. Developing a packaging technique for both the memory element and the electronics, especially the technique for interconnections between the two. (Depending on the memory capacity, there may be hundreds of these connections; they represent the most significant potential source of failures.)
5. Overcoming the general effects of the emphasis in commercial technology (and many satellite projects) on low cost and/or other factors detrimental to the necessary quality and reliability.

3. Approach

a. Survey. To ascertain the state-of-the-art of memory development, an industry survey was undertaken. The development status of standard as well as new memory approaches was determined for a portion of the industry.

The completed survey will include a study of memory systems that have been built specifically to withstand high-g shock and/or high-temperature environments. Although aside from the military there existed no general need for these design criteria, considerable information can be obtained from such sources as the Harry Diamond Fuse Laboratories and the Atomic Energy Commission, which have built impact-surviving systems. The JPL Packaging Section and the Material Research Council of Canada, both provided with high-g impact facilities, are considered to be sources of information, and it is planned to cooperate with these agencies and any others similarly engaged to gain insight and to avoid obvious pitfalls.

b. Investigation of design trade-offs. The requirements for data and electrical organization (random-access, serial first-in, first-out, etc.) are presently under discussion with systems engineering personnel. System trade-off factors that involve buffers or memories include weight, power, and reliability. With a selected memory organization and more than one memory element packageable to withstand shock and sterilization available, the final selection of the memory element must include suitable trade-offs with and between the following factors:

1. Complexity of the required electronics.
2. Reliability of the interconnections between the memory element and the electronics.

The data capacity of the memory will also affect the trade-offs; thus, small and large memories may conceivably benefit from different technologies. The omission of the high-g impact requirement could also affect the choice.

c. Hardware investigations by JPL. Samples of several memory elements are being evaluated by JPL; contracted assistance will be sought where warranted. Suitable packaging materials are being investigated for their mechanical, electrical, and chemical suitability, and packaging techniques will be developed in coordination with the JPL Advanced Packaging Group. In particular a detailed study of all possible memory-electronics interconnection techniques will be carried out.

Reliable low-power circuits must be developed, and feasibility studies are in process in order to support the development of packaging and interconnection techniques. Some work has already been accomplished in this field (including a previous JPL-sponsored study contract); further contracted assistance will probably be sought.

d. Full flight-model prototype. Fabrication of a full flight prototype is pending the completion of present development work and is contemplated for FY 1967.

F. Sterilizable Solid-State Radiation Detectors,

R. A. Wengert

Sterilizable, ruggedized, and highly reliable solid-state radiation detectors for use in the detection and measurement of alpha particles and protons and for counting beta particles are being developed. These detectors may find application in biological instrumentation, in instruments used in the study of trapped radiation belts and the distribution of ionizing radiation in the atmosphere, and in the determination of specific elements of planetary atmospheres.

Possible application of these detectors is for instruments in the payloads of planet-orbiting and/or soft-landing spacecraft. Further testing, however, may prove them usable on hard-landing vehicles.

Four types of detectors are included in this program; the processing has been developed to the point where the resulting detectors are sterilizable, but work remains to be done to improve their characteristic behavior following the environmental testing.

1. Problem Areas

The following problem areas were encountered during development:

1. Bonding of the signal lead to the silicone wafer.
2. Detector signal degradation as a result of heat sterilization.
3. Mechanical degradation caused by gas decontamination.
4. Metal-to-metal surface contact resistance.

2. Discussion of Problems and Solutions

The following procedures were implemented to solve problems encountered:

1. A signal lead must be brought from the active area of the detector for use in the electronic circuitry. An attempt to accomplish this by bonding a 1-mil gold wire to the detector face with a silver doped epoxy resulted in a very noisy contact after heat sterilization. Another attempt to bond the wire, this time by ultrasonic means, was unsuccessful.

The problem was successfully solved by depositing a protective insulating epoxy ring on the detector front surface and evaporating a thin gold layer over it, establishing contact with the active area and a spring contact between the gold layer and the signal lead. This arrangement successfully passed all sterilization and environmental tests.

2. All sterilization and environmental testing was subcontracted to a testing laboratory. After the first cycle of heat sterilization, the detectors were observed to have a black material deposited on 30% of their surface area, and had suffered a 60% increase in leakage current and a 10% degradation in resolution. The trouble was traced to contaminating materials in the oven. When the detectors were cleaned and retested in clean ovens, no degradation was encountered.

3. When the detectors using various contact bonding epoxies were subjected to gas decontamination, the material became soft and suffered mechanical deformation. The problem was solved after a thorough study of the various epoxy materials A mixture of Armstrong epoxy C-7 and activator W in a 70-to-30 ratio proved satisfactory for this application.

4. The detector assembly contains a number of metal-to-metal surface contacts used to transmit the detector signal to the output connector. It is believed that proper surface plating will reduce the contact resistance and eliminate measurement changes introduced when the detector is subjected to the environmental testing.

This task is being performed by the Special Products Division of the Technical Measurement Corporation, San Mateo, California, under contract to JPL. The principal investigator is Mr. Louis Wang.

G. *Sterilizable Inorganic Scintillation Crystals,*

R. A. Wengert

A current task consists of the development of inorganic scintillation crystals which will be unaffected by both heat sterilization and gas decontamination, ruggedized to withstand environmental requirements, and highly reliable. The crystals will be suitable for application in determining planetary surface composition (by neutron activation experiments) and specific elements in planetary atmospheres and in gamma ray detection and spectroscopy.

Crystals that satisfy the sterilization and environmental requirements have been designed and tested and are suitable for application in instrumentation which may be included in the payload of planet-orbiting or soft-landing spacecraft. Further testing and/or improved packaging may extend their use to hard-landing vehicles.

Initial testing was performed with thallium-activated sodium iodide and cesium iodide crystals. Early in the program it was decided to concentrate on the NaI(Tl) crystal because of its much better pulse-height resolution. Although this crystal is more fragile than CeI(Tl) and is very susceptible to water vapor, it was believed that proper packaging could overcome these problems and allow taking advantage of the better resolution of NaI(Tl).

1. Problem Areas

The following problems were encountered during the performance of this task:

1. Coupling of an exit window, both mechanically and optically, to the crystal package.

2. Crystal fracture during both dry heat sterilization and environmental testing.

2. Discussion of Problems and Solutions

Commercially available crystal packages contain the crystals in an aluminum housing with a glass exit window. For maximum transfer of light to the sensor being used, an optical coupling material is placed between the crystal and the glass window. All coupling material used introduced signal degradation as a result of heat sterilization. An Epon cement changed to a yellow color and caused crystal fracture. A silicon vacuum grease turned brown and caused loss of the crystal-window interface. Further testing used a dry coupling and resulted in a slight degradation but much more predictable characteristics than when coupling material was used. When the environmental requirements were considered, maintaining the glass window in position and in good condition became a problem. To protect the fragile crystals, the entire package was potted with RTV615. Because of the good optical characteristics of the potting material, the final configuration, as now envisioned, will not have a glass window.

Initial testing before and after heat sterilization indicated that the crystal characteristics did not deteriorate

as a result of temperature cycling if the crystal did not fracture. A number of crystals, however, did crack, and investigation revealed that the effect of thermal shock was more evident in units having internal stress and crystal multiplicity. This was also evident during environmental testing in that units with internal stress fractured during testing. Proper inspection, selection of single crystals, and improved packaging have eliminated this problem. Initial heat sterilization testing of bare crystals produced the requirement that the 145°C temperature be applied and removed at a rate no greater than 25°C per hr. Because of the heat-transfer lag introduced by the RTG615 potting compound previously discussed, the reduced rate of heat application is no longer required.

3. Further Investigations

Although major program emphasis was placed on the improvement of available and proven crystal types, additional work is also being performed on others. Europium-activated calcium iodide crystals are being grown in an attempt to produce sterilizable crystals with more favorable characteristics than those of NaI(Tl) developed during this task. Since this phase has only been started recently, no result can be reported at this time. If sterilizable, however, these crystals may be expected to have pulse heights 150 to 200% better than NaI(Tl) and correspondingly improved energy resolution characteristics.

This task is being performed by Isomet Corporation[2] under contract to JPL. The principal investigator is Dr. Warren Ruderman.

H. Sterilizable Geiger-Müller Counter Tubes,

R. A. Wengert

Heat- and gas-sterilizable, ruggedized, and highly reliable Geiger-Müller tubes are under development. The tubes are of two types: thin sidewall and mica end window. Possible application of these detectors includes their use in biological instrumentation and in instruments used to study possible trapped radiation belts and the distribution of ionizing radiation in the atmospheres of other planets. The detectors as presently designed and tested may find application in soft-landing capsules and planet-orbiting spacecraft. Further testing may prove them usable aboard hard-landing vehicles.

[2] Palisades Park, N. J.

1. Problem Areas

The following problems were encountered during the performance of this task:

1. Leaks developed in the stainless-steel sidewall of the thin-wall detector tubes.
2. The relative plateau slope for both tube types had a relatively high value.
3. The mica end window incurred damage during gas decontamination.
4. The test data exhibited poor reproducibility.
5. The tube operating characteristics drifted.

2. Discussion of Problems and Solutions

The thin-wall tubes are made of stainless-steel tubing machined to an effective wall thickness of 30 mg/cm^2. Following machining and cleaning, the tubes are tested with a mass spectrometer to detect any leak through the wall material. The first group made showed an unusually high number of rejected parts. A microscopic inspection detected flaws in the tubing material which when machined could develop into pinholes and cracks. This problem was easily solved by finding another source of the material and closer incoming inspection.

Commercial tubes, similar in most respects to the desired types, have relative plateau slopes considerably greater than the 5% specified. The nominal value for the relative plateau slope of the commercially available thin-wall tube is 10% per 100 volts and 20% per 100 volts for the end-window tube. To accomplish improvement of this characteristic, the following investigations and modifications were made.

The standard version of the thin-sidewall tube did not maintain the anode under tension but held firmly only at one end while the other end was centered by being retained in a rather loose-fitting boss. It was determined that the value of slope could be best improved by better centering of the anode, thereby maintaining a uniform field along its length, and by holding it more rigid to eliminate sag or deformation caused by the required environmental testing. This improvement was made by designing a new retaining member for the far end of the tube that would better center the anode and permit it to be held under tension. This modification improved the

value of the slope to an average of 7.5%—still short of the desired 5%.

The end-window tube first selected satisfied all requirements with the exception of the plateau slope, which was 20% per 100 volts. Because of the end window, the anode was cantilevered and was not firmly held in a centered position or in tension. The first attempt at improvement consisted of using a slightly larger tube with a supported ceramic sleeve to hold the anode in a centered position. The attempt was unsuccessful because the anode wire was distorted during environmental testing, and the larger tube was abandoned. The problem was solved when a heavier anode was used in a tube similar in size to the first one selected. The average value of relative plateau slope for tubes of the final configuration is 3.5% per 100 volts, surpassing the desired goal.

After gas decontamination of the end-window tubes, it was discovered that a number of tubes failed because the mica windows were damaged during the pumping out of the gas sterilizer. At this time, the pressure on the outside surface of the window is less than that on the inside, and the resulting outward force fractures the mica. This problem was solved by adding a stainless-steel reinforcing strongback over the window. Two designs were fabricated and successfully used on experimental tubes. The first was a mesh design resulting in an open area of 51%; the second consisted of two crossbars with an open area of 80%. There appears to be no detection degradation in either case.

In the early phase of the program, testing to determine the plateau points used a counting rate of 100 counts per sec during a 1-min period. It was found that because of the random nature of the radiation from the source, the desired repeatability could not be obtained. Statistical analysis indicated that in order to measure true tube changes the testing should be performed for 3-min intervals. This change was initiated, and as a result any change in the measured values indicates a true change in the tube characteristic.

The problem of the drift of tube operating characteristics is still unsolved. The change was attributed to the chemical reaction between the filling gas mixture and the stainless-steel base material of the cathode. To avoid this reaction, the cathode surface was plated with a metallic material which does not react with the gas. However, the problem was not alleviated, and it is possible that the plating may sometimes be porous and therefore penetrated by the gas mixture. Improved plating techniques are being investigated to solve this problem.

This task is being performed by the EON Corporation, Brooklyn, New York, with Dr. Gustav Weinberg as principal investigator.

VI. TELECOMMUNICATIONS

The Telecommunications Division is responsible for the research, development, and support of radio tracking, radio communications, and interstation data transfer for all JPL programs. Further Division responsibility includes supplying space-borne elements and associated ground support equipment as required to meet the tracking, telemetry, and command needs of the various Laboratory programs, with the exception of special data handling equipment peculiar to space science instrumentation and computers peculiar to guidance and control functions.

Additionally, tracking and telemetering data is provided as required at the ground stations and for post-flight tracking and telemetering data in reduced form as needed at the Laboratory.

The Division is also responsible for the Deep Space Instrumentation Facility, the technical communications net, ground support equipment associated with spacecraft communications, and those technical aspects of missile-range radio instrumentation which affect Laboratory programs.

To implement the sterilization requirement, the Division is engaged in research and development work on those elements of space-borne communication instrumentation which use nonstandard component parts. The sterilization development program is presently applied to tape recorders and pressure transducers.

A. A Sterilizable Magnetic Tape Recorder,

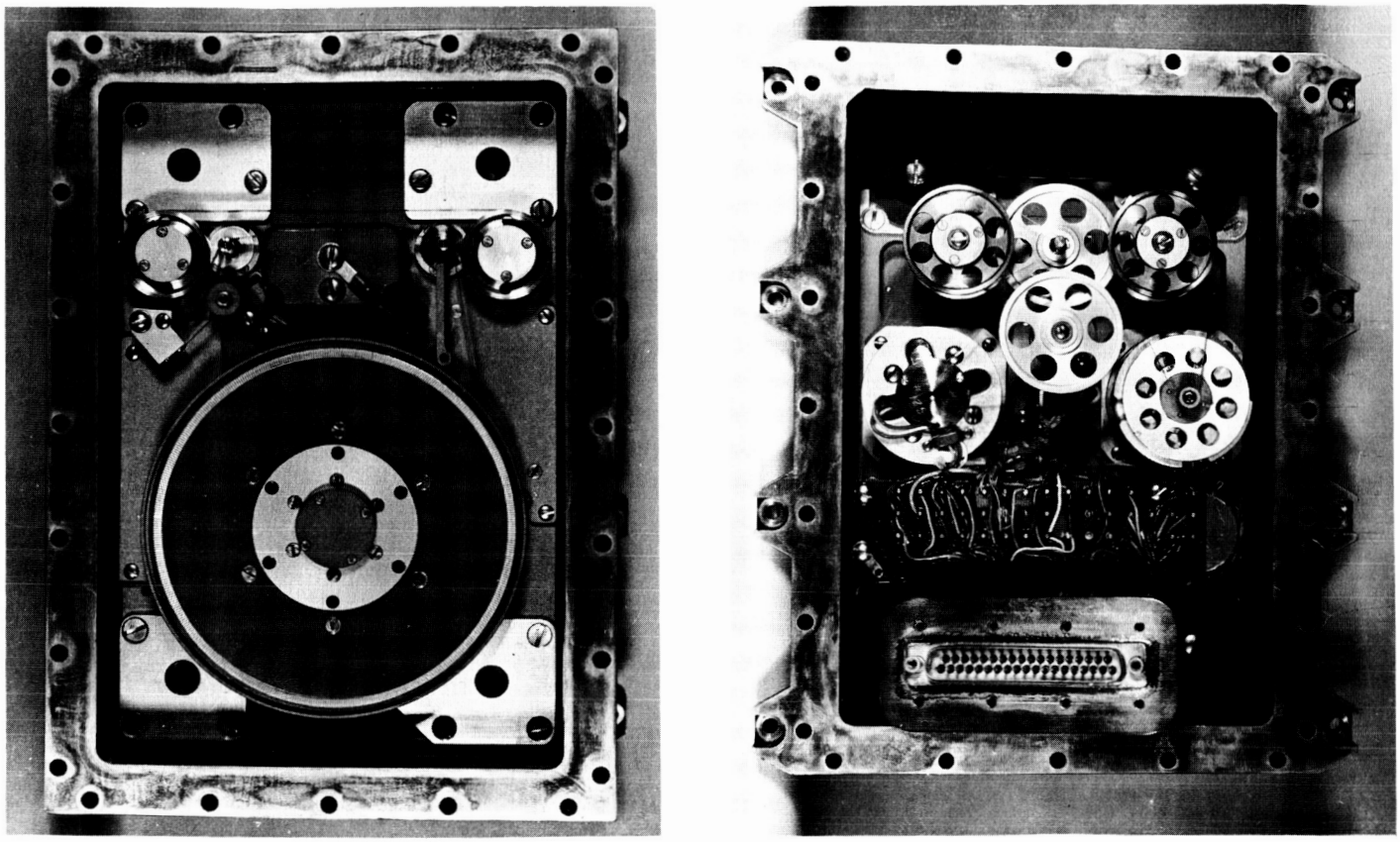
A. R. Lowe

The sterilization of magnetic tape recorders to be used for data storage on planetary missions has been investigated. In addition to the regular environmental requirements—vibration, RF interference, and magnetic field—spacecraft-mounted tape recorders will be required to withstand 24 hr of 135(+4, −0)°C in inert atmosphere for flight acceptance; prototypes will be required to withstand three 36-hr cycles at 145±2°C for type approval. An additional requirement consists of soaking the instrument in an ethylene oxide atmosphere at 35% RH and at 40°C for 24 hr. This process is considered a decontamination rather than a chemical sterilization and is less reliable than heat sterilization. However, in the case of the tape transport, it is simpler to implement than heat sterilization, as a sealed pressurized box is used; the only concern is the possibility of sealing material degeneration under the specified condition. No degeneration occurred in the Parker Gask-O-Seal made of DuPont Viton rubber; nor was there any evidence of ethylene oxide inside the box or a significant increase of leak rate after the decontamination tests.

The major portion of the effort was carried out at the Raymond Engineering Laboratory, Connecticut. Fabrication of a heat-sterilizable transport was begun by separately testing and, when necessary, developing individual components such as motors and bearings with a view toward later assembly and unit testing.

The transport plate was one of the first components to be considered. Despite expansion due to heating, a certain amount of plate warpage is not considered serious. The plate flattens again after the heat cycles, and plate-mounted parts are realigned. Since the motors, idler, and capstan shafts are all perpendicular to the plate, the only effect of plate warpage is a varying of the spacing. When this deformation is combined with the effects of the thermal coefficient of expansion of the belt material, however, a net change in belt tension will take place. It is hoped that this change will not exceed the elastic limit. In the case of the *Mariner C* sterilizable tape recorder (Fig. 1), the plate is made of AZ31B magnesium and the drive and reduction belts are made of DuPont polyimide film (the so-called "H-film"). The coefficient of expansion for magnesium is greater than that for H-film; for example, as the length of the drive belt was twice that of the pulley centerline distance, thermal changes were very nearly cancelled out. This was not true for the reduction belts, and, as a consequence, loss of tension of from 15 to 25% had occurred and was compensated for by presetting these tensions to the high side of their original tolerances.

Aside from the sterilization requirements, the problem of size and tolerance of bearings required for this application is complicated. The approach to this problem consisted of measuring the run-out and coast-down time of all of the rotating components of the *Mariner C* recorder, running them through the heat cycles, and repeating the measurements with a disassembly inspection. In most cases, changes were insignificant. However, one of the six assemblies so tested, a capstan, had a run-out increased by a factor of 12, and a coast-down increased by a factor of 2. This assembly (Fig. 2) was investigated to determine the cause of failure. The excess run-out was apparently



FRONT VIEW REAR VIEW

Fig. 1. *Mariner* C tape transport

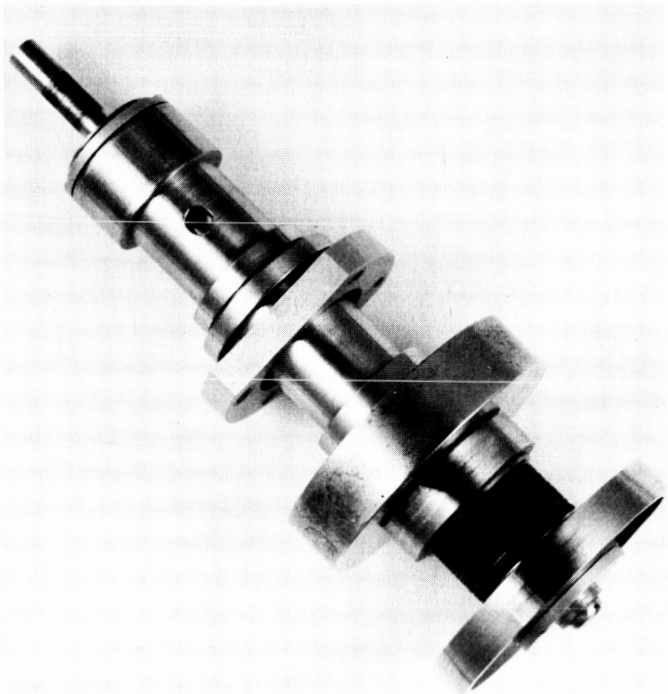


Fig. 2. Upstream capstan

caused by a relaxation of the preload as well as by certain borderline tolerances which compounded into negative results such as surface flatness, parallelism, and length of separators. Another possibility is that this capstan was of material from a lot having a different heat-treatment history from that used for capstans that exhibited no problems. The faulty capstan was reworked, resterilized, and used again. This process was also employed with other items by subjecting them to an environment slightly in excess of the required one and reworking them to correct any shifts.

In the case of the record and reproduce motors, the laminations were spray-varnished, assembled, bored, "fluidized" with an epoxy compound, and baked for several cycles, the most extreme of which was 185°C for 4 hr. Any volatiles from the varnish or Hysol used to cement the laminations were thus driven off by the fluidizing process, and the stack was good for continuous duty at 170°C.

These motors were designed by H. C. Rotors, Kew Gardens, N. Y., and produced by Raymond Engineering

Laboratory; they are quite efficient for their size—about 25 to 30% —and quite stable. The sterilization cycles had very little effect on them. The only changes that occurred were on the order of a 10% reduction in stall torque at −10°C in the playback motor and an increase in both synchronization and stall torque of the record motor (at room temperature) of the same order of magnitude. Slight changes were also noted in the bearing running torque, but they were not considered significant. In general, bearing torque tests are not repeatable to within ±10%, and greater reliance is placed on life tests. The tested motor bearings are still in operating condition.

Magnetic tape heads presented the most troublesome problems to be solved. The first heads, produced by Applied Magnetics Corporation (AMC), Goleta, Calif., were made on the basis of substitution of high-curing-temperature (400°F) bonding materials such as Union Carbide R-610 silicone varnish and others. Although these heads—there were three of them—survived initial sterilizations at AMC, they had not been mounted nor had tape pressure been applied against them. They all failed eventually after being mounted on the transport. At this point, consideration of possible failure mechanisms led to speculation that temperature coefficients had to be matched, and possibly a head design peculiar to this application would have to be employed (such as the inclusion of a retaining spring to force the pole pieces to return to their original position after reduction to ambient temperature).

Compatibility of thermal expansion coefficients was obtained by fabricating molds of RTV rubber, laying a strip of metal in the bottom, and pouring an epoxy over it. After cure, the end of the strip of metal and epoxy was clamped and the relative curl vs temperature was measured by means of a set of rulings in millimeters placed at the other end of the strip. Figure 3 shows two excellent results, exemplified by the minimum amount of slope. Figure 4 shows two curves of greater slope that, in addition, show a change in slope in each curve reflecting an undesirable characteristic. An indication of relative peel strength may also be obtained from this type of testing.

The aforementioned spring arrangement was unsatisfactory and did not solve the common problem of pole piece "stepping." While the main problem in heat sterilization of the head is that of gap opening, slight asymmetries in head construction lead also to terminal misalignment of the pole pieces in the plane of the tape. So far, the most adequate approach to this problem is care in assembly. The best set of heads sterilized to date still had a small step of about 20 microinches, which was burnished away after sterilization. The technique used with this set, and three other more or less successful ones, consists of filling the back volume of the head with a "poor match" epoxy so that as expansion takes place, the back gap tends to open, forcing the front gap to close, with a fulcrum being formed in the vicinity of the assembly screws. The position and expansion coefficient of these screws also becomes important. Earlier assemblies used two screws, one in each core half; later assemblies use one screw per head, so that one core half is more or less "floating." In addition, the overall fill for the volume between the core and shell is now a relatively resilient rubber, rather than epoxy, so that some of the forces may be absorbed.

At this point, it appears that design technique may be more important than actual coefficient "matching." Four sets of heads (each set containing one record and one reproduce head) have passed the 108-hr tests, all using different shell materials: aluminum, aluminum-bronze, KR monel, and brass. Of these, the aluminum head appeared to be the most successful and was mounted on the

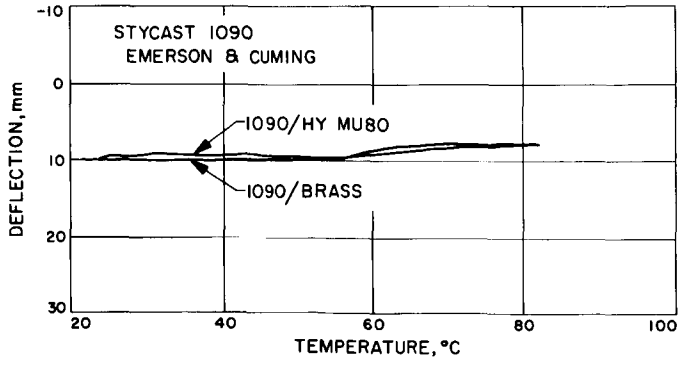


Fig. 3. Thermal expansion coefficients

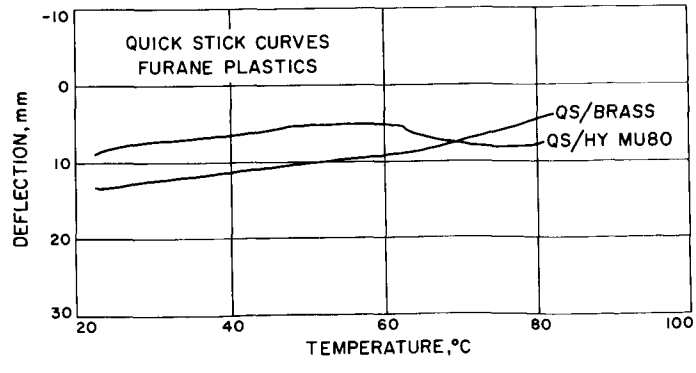


Fig. 4. Thermal expansion coefficients

transport for the overall sterilization tests. This head performed well functionally after an additional 108 hr at 145°C (total of 6 heat cycles); however, visual examination then revealed that loosening of a slight amount of potting compound was evident, and a segment or splinter had formed at one side of track 2, which could have been serious had it formed in the track path. The heads were bright, with no sign of seepage of interlamination adhesive and no head-to-tape stickiness. High-power microscopic examination has not yet been made.

Mariner C (Mars mission) type approval of 80°C (later downgraded to 75°C) had caused some concern over mylar belts and magnetic tape, since their survival is marginal at these temperatures. During the *Mariner C* tape recorder development, JPL personnel tried to obtain and use H-film as a substitute for mylar in these applications. Because of limited time before the launch date, the effort was abandoned, and development was completed with the polyester (mylar) material. During the sterilization project, however, there was time to pursue the use of polyimide (H-film) for belts and tape. To begin with, some comparisons were made at the sterilization (type approval) temperature, and it was found that (at zero tension) a mylar belt shrank over 5%, while an H-film belt had negligible dimensional change. For the H-film belt, tensile modulus was degraded by 2.5%. The mylar belt tensile modulus was degraded by 18%, and this belt tried to revert back to its original flat shape. It was also found that if the belts were mounted and under tension, the mylar belt lost as much as 61.5% of its initial tension, while the H-film belt lost initial tension in the range of 16 to 25% as a result of sterilization. This tension loss was found to be acceptable if the belt tensions were initially set to the high side of their tolerances. Tensions would then not drop below their low boundaries after sterilization. The first H-film belts were found to be wrinkled. However, this problem was circumvented by using constantly rotating mandrels during shaping and curing at approximately 600°F.

Some consideration had been given to the use of H-film for magnetic tape on the *Mariner C* program; in fact, consideration had originally been given to producing a Mars-mission tape recorder which would withstand a sterilization temperature of 125°C. A number of brands (and types of brands) of mylar- and metal-backed tape were investigated, since at that time only two companies were found that could produce even sample lengths of H-film-backed tape. In the case of mylar, it had been found that its 2.5 to 5% shrinkage could be alleviated by preshrinking or curing, and it was planned to do further work with mylar in the event of failure elsewhere. The metal tapes were too low in output for the slow, 0.01 in./sec tape velocity, and they were found to curl as a result of passing over the short-radius rollers. One company (Reeves Soundcraft) was able, on request, to produce a 6-ft sample of polyimide-backed tape which appeared quite promising in initial tests.

It is evident that the binder-backing system needs to be compatible, and in this case, the particular Reeves binder is compatible with H-film but not with mylar. A number of binders have been tested with respect to lubricity or stickiness with the head and the backing of either plastic (single-sided) or oxide-binder (double-coated) tape. Because the difference in expansion coefficients between the binder system and the backings would tend to produce a curl, a double-sided (coated on both sides) version of polyimide backing with the Reeves proprietary binder was selected. First attempts at sterilizing an entire pack met with failure; the pack had "blocked," and the conditioning processes that had become necessary for other portions of the transport were again employed. This conditioning (or curing) consisted of 24 hr at 150°C in a dry nitrogen wash, or flow of gas, over a loose tape pack. A tape-burnishing and cleaning process followed, because of lubricant (graphite) shedding due to an excess of graphite rather than any deterioration of the binder system. The burnishing and cleaning were carried out with two successive grades of the burnishing tape normally used for polishing heads while mounted on the transport. Two tape drives were arranged so that the magnetic tape and the polishing tape passed over a common roller of about 6 in. in half-circumference in opposite directions. This common path was adjustable in that a greater or lesser wrap angle could be obtained. After as many as 50 burnishing passes, an additional 10 or 12 passes were made with unoiled telegraphic paper tape to clear off the residual graphite. With a pack of 330 ft of ¼-inch tape, weight losses of about 0.2 g of the total weight of about 70 g were noted.

Polyimide-backed tape with Reeves proprietary binder and lubricant treated in this fashion was found to be sterilizable in the endless-loop reel with no starting difficulties. An observable physical change was evident — the tape became stiffer, or "crinkly." This change might be expected to lead to an improved flutter characteristic, as no flutter deterioration was noted. There was, however, some degradation in the amplitude modulation envelope of the recorded signal, probably because of reduced compliance of the head wrap. Also, a more intimate adhesion between the backing and the binder

was noticed; this occurred to such an extent that the usual solvents such as methyl ethyl ketone, butyl acetate, trichloroethylene, and nitric acid failed to remove the cured oxide binder. The tape was therefore spliced by the usual means using DuPont PM 1200 polyimide high-temperature binder solution, except that the splice was made over the oxide. Tensile strength tests showed the splice to be stronger than the adjacent material.

The entire transport, with two preamplifiers, is contained in a case which is hermetically sealed with Parker Viton rubber Gask-O-Seals. In the past, a great deal of work had been done on the sealing problem with respect to the actual *Mariner C* transport. The objective in this case was to extend sealing to withstand the higher temperatures. Leak testing is not particularly accurate, since tests under similar conditions yield three different ranges of values at three different locations. Ardel Corporation figures are in the vicinity of 7 cc/in./yr; Raymond figures are around 4.5 cc/in./yr; and the University of Rochester obtained about 3 cc/in./yr. Indications are, however, that the leak rate doubles when the case temperature is raised to 145°C but returns to normal after cooling to ambient. From a 21-psig gas pressure at room temperature, a pressure loss of about 0.8 psi occurs over the 108-hr period but is not considered critical. The cover (8½ by 6 in.) exhibited a certain amount of bulging (about $\frac{3}{32}$-in.), which indicates a need for a stronger cover. Ethylene oxide decontamination testing was also accomplished. The first test was a 24-hr soaking at room temperature in 35% RH, 500 mg/liter of 88% C_2H_4O, and 12% Freon-12. The second test is similar but with a temperature of 40°C. No change in the leak rate could be observed as a result of these tests, and no evidence of ethylene oxide osmosis could be found in the magnesium case. No known problem exists with present materials and techniques.

The end-of-tape sensing used in this *Mariner C* transport consists of metallic fingers contacting an area of the tape which has been made conductive by some means. The gold-plated mylar–aluminum laminate cannot withstand the sterilization temperature because, in addition to more obvious reasons, the gold edges tend to break down. An attempt to bond 0.3-mil gold leaf to the oxide side of the H-film was not successful because of nonadherence and cracking. Two other approaches were tried using silver: one was a deposition by the Brashear (mirror silvering) technique; the other was by spraying with a silver lacquer. Upon heating, the Brashear-deposited silver volatalized, but the silver lacquer remained, giving a resistivity of about 200 milliohm/in. At the low tape speeds involved (0.01 in./sec), this method is too noisy and further investigation is needed. Gold deposition or some optical method might be alternate approaches.

The culmination of the described preparatory work was to assemble the entire transport with its reject electronics and subject it to the sterilization cycles. Upon subsequent attempted operations, it was found that the transport started normally with no sign of sticking or binding, but that the electronics for one channel had failed, and the other channel was operating at reduced gain. The head outputs were normal, as were flutter and envelope amplitude modulation for cured tape. Digital coding was well defined, showing good high-frequency response. About 25 passes of the tape were made prior to opening the case. When the case was opened, a number of things were observed:

1. A black stain on the Dow 17 finish adjacent to the Viton seal rubber.
2. Whitish crystals also adjacent to the seal but toward the inside of the case.
3. A soft grey deposit on the reel housing which soon volatalized, reminiscent of an earlier experience with tape binder outgassing.
4. A surface phenomenon on the beryllium copper clutch spring and capstan "true-arc" (C-clamp) which gave the appearance of fine diamond dust.
5. A crack in the epoxy of one of the AMC heads, but not located in the tape path. (The gap remained constant, with no shifting or stepping evident under nonmicroscopic examination.)
6. An etched, or dulled appearance was imparted on the brass flywheel, the clear anodized aluminum pulleys, and all solder connections.

In trying to ascertain the reason or reasons for the corrosive effects noted, it was found that one item used in the routine assembly of the *Mariner C* transport had not been tested — the Dow Corning No. 55 silicone grease used as an aid to hermetic sealing. Previous leak check testing on the case before, after, and during heat sterilization and chemical decontamination had not employed this grease, and the tests had been successful. Unfortunately, it was applied in the test of the assembled and sealed transport and is believed to be responsible for the whitish crystals (which analysis show to be mainly silicon and magnesium). The scintillating surface phenomenon observed on the beryllium copper surfaces was believed to be a sulphur or chlorine compound; accordingly, the

Viton–elastomer and the grease were tested for chlorine and sulphur. Viton is a fluorohydrocarbon and does not contain sulphur or chlorine either in formula or as contaminants. The No. 55 pneumatic grease contained not only a chlorinated aromatic silicone but lithium oleate plus some diphatic hydrocarbon additives. Furthermore, the total volatile material was found to be over 16% when the grease was heated to 110°C for 23 hr. The manufacturer claims this product to be heat-stable and serviceable to 350°F (177°C).

Upon examination of the transport, a 3-week period of life testing was undertaken. A program of slow and fast tape velocities (0.01 and 12 in./sec) was established as well as an attempt to check bit errors. The bit error test produced no significant results due to faulty instrumentation. A capstan bearing failed after five days. That is, it became noisy and resulted in out-of-spec flutter. Another failed after ten days. These failures were assumed to be due to insufficient lubricant but could also be due to contamination of the lubricant.

Future work in tape recorder sterilization should include bearing lubricants, bearing and capstan design, more work on head design and materials testing, and the development of a new tape binder system. Contamination due to the outgassing of epoxies and cements used in miscellaneous places should be investigated more thoroughly, even though the manufacturer claims a high-level temperature range for his product. Control of the temperature history of metal as well as high-temperature end-of-tape sensing (possibly optical) should also be investigated.

Although this project resulted in a transport which failed to meet specifications after 5 days of life testing subsequent to type approval sterilization, a great deal of knowledge and experience has been gained which will be invaluable in future studies.[1]

B. *Pressure Transducer Sterilization,*

Gordon A. Crawford

Pressure transducers are used to monitor engineering measurements (those concerned with the performance of the spacecraft) and scientific measurements (those concerned with the environment external to the spacecraft). In view of sterilization requirements, a program was initiated to define and solve the problems associated with the sterilization of engineering pressure transducers.

This program was divided into the following three parts:

1. An investigation of the numerous commercially available pressure transducers to find those suitable for spacecraft needs, i.e., insofar as range, size, weight, power, and output are concerned. (Transducer types are listed in Tables 1 and 2; their relative advantages or disadvantages are presented in Ref. 1.)

Table 1. Types of pressure transducers

1. Resistive
   - Potentiometric
     - Wire-wound
     - Carbon film
     - Conductive plastics
2. Ionization
   - Philips
   - Hot cathode
   - Cold cathode magnetron
     - Red head
   - Radiological
     - Alphatron
     - Betatron
3. Thermoelectric
   - Thermocouple
   - Thermister
   - Pirani
   - Pyroelectric
4. Force balance
5. Digital output
   - Binary code
   - Grey code
6. Oscillating
   - Vibrating cylinder
   - Osciducer
   - Vibration
7. Photosensitive
   - Photovoltaic
   - Photoresistive
   - Photoelectric
8. Electrokinetic
9. Piezo-electric
10. Capacitive
11. Field gradient
    - Hall effect
12. Inductive
    - Variable reluctance
    - Differential transformer
13. Ionization
    - Variable capacitance
14. Magnetostrictive
15. Piezo-resistive
    - Stram gage
      - Metallic
        - Semi conductor
          - Bonded
          - Unbonded
          - Weldable
      - Variable pitch spring
      - Ohm-strictive
        - Organic polymers
        - Carbon pile
16. Vacuum tube

Table 2. Types of force collectors

1. Flat diaphragm
2. Corrugated diaphragm
3. Aneroid capsule
4. Bellows
5. Circular bourdon tube
6. Twisted bourdon tube
7. Straight tube
8. Concentric tubes
9. Hexagonal tube
10. Piston

[1] A comprehensive report of the latest developments in magnetic tape recorder sterilization appears in JPL's Space Programs Summary No. 37-37, Vol. IV. An abstract of that report is presented at the end of this section.

2. A study of various transducer mechanisms and the materials used in their manufacture to evaluate their ability to withstand sterilization and space environments.

3. Heat sterilization and subsequent testing of some typical transducers to prove their ability to withstand sterilization temperatures.

1. Transducer Selection

Past experience with pressure transducers from two different manufacturers indicated that potential problems existed in meeting the temperature requirements for dry heat sterilization. As a first step, other manufacturers were investigated to determine whether new transducer types had recently been developed.

The Instrument Society of America has published a Transducer Compendium (Ref. 2) listing numerous companies and various types of transducers. This book was used to compile a list of companies with potential capabilities of supplying transducers for the sterilization program. Three companies (Statham Instruments,[1] Micro Systems,[2] and Giannini Controls[3]) were visited, and the manufacturing processes of several different types of transducers were observed.

Each of the three companies expressed confidence that, with additional care in the selection of materials, their present transducers could withstand the sterilization environment. Two of these companies (Micro Systems and Giannini Controls) volunteered the loan of a transducer to be subjected to JPL sterilization and type approval environmental tests; one from each company was accepted on a loan basis and tested.

2. Description of Transducers Tested

The Giannini Controls Corporation 461 322-A1V6-80-75 absolute pressure transducers (Fig. 5) is a high-accuracy, hermetically sealed, potentiometric output, Bourdon-tube-type instrument intended for pressure measurements from 0–300 psia minimum range to 0–3500 psia maximum range. Specifications are listed in Giannini Controls Specification S461322.01.

The Micro Systems transducer type PT8-S/N703 (Fig. 6) is designed for industrial and aerospace pressure measurements and control applications where a high unamplified voltage output is extremely useful. Designed to measure pressures in the range of 0–50 through 0–5000 psi, this transducer is constructed of corrosion-resistant materials and consists of an integrally machined diaphragm to which four active semiconductor strain gages are bonded. The pressure cavity is a welded assembly without O-ring sealed or threaded joints.

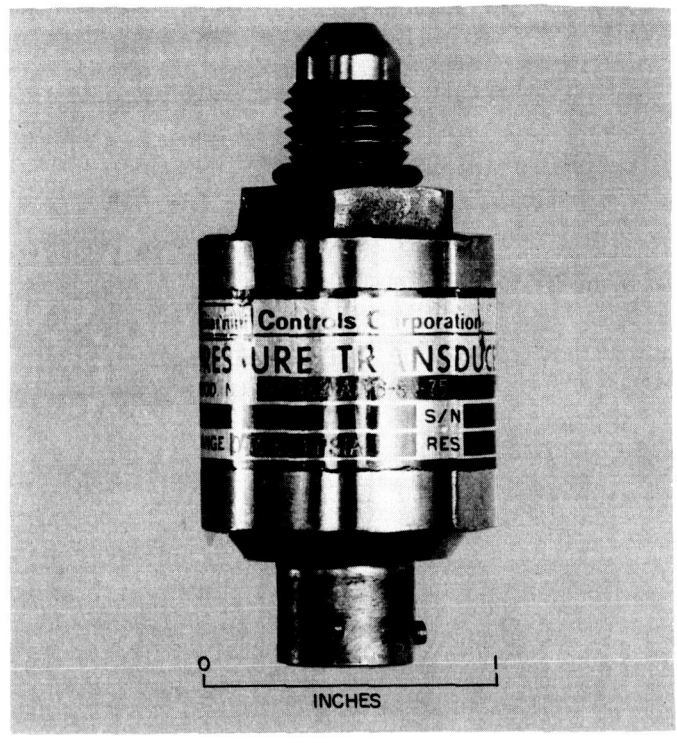


Fig. 5. Giannini Controls transducer

3. Transducer Tests

The two transducers loaned to JPL for testing were calibrated (Fig. 7) before any tests were started. The first test consisted of dry heat sterilization as described in JPL Specification XSO-30275-TST-A. It required subjection to a temperature of 145 ±2°C (293°F) in a dry nitrogen environment for 36 hr. This test was repeated three times with stabilization to room conditions between heating cycles. The transducers were pressurized and monitored between each temperature cycle through the original calibration points (Figs. 8 and 9).

The second test consisted of subjection to vibration per JPL Specification 30257 (Paragraph 4.3.2.1b): 2 min at combined 10-g-rms noise plus 4-g-rms sinusoid, and 6 min at combined 5-g-rms noise plus 4-g-rms sinusoid. This test was performed in all three planes (x, y, and z) and the transducers were then again checked against the original calibration (Figs. 8 and 9).

[1] Los Angeles, Calif.
[2] Pasadena, Calif.
[3] Duarte, Calif.

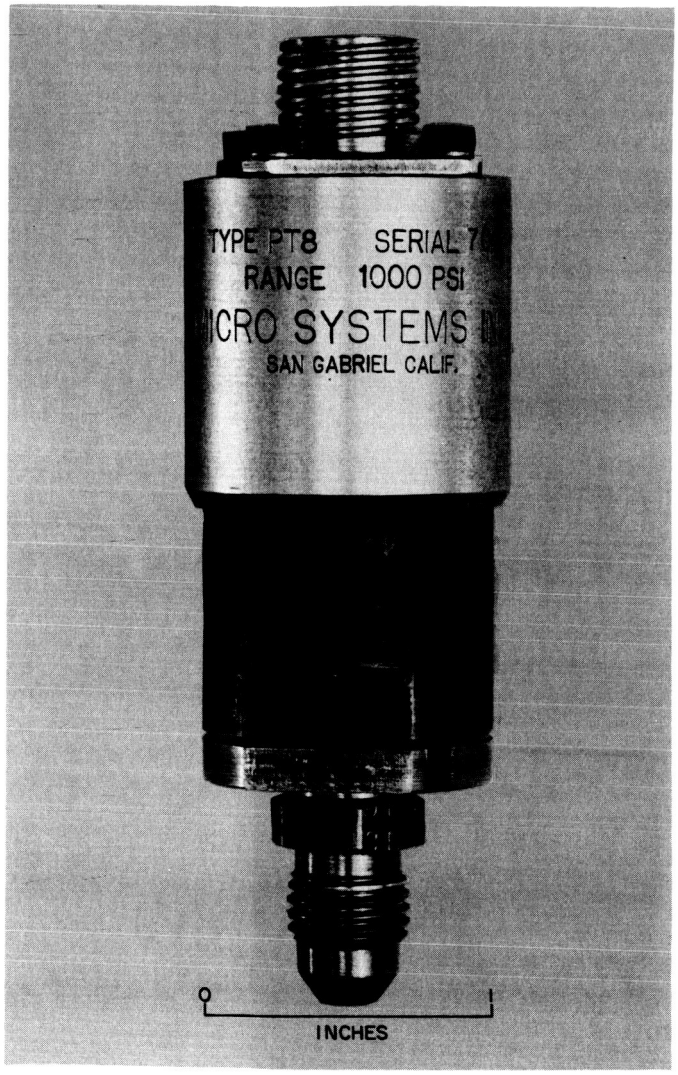


Fig. 6. Micro Systems transducer

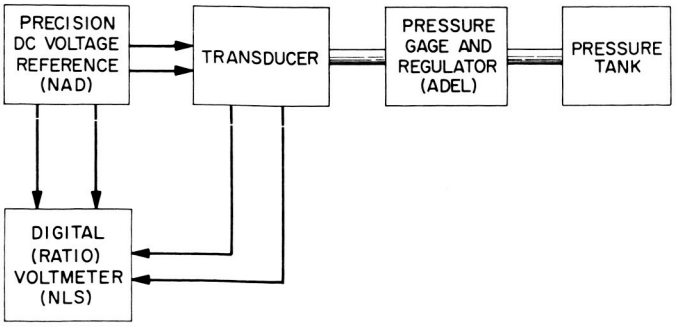


Fig. 7. Pressure transducer calibration setup

The third test performed consisted of subjection to shock per JPL Specification 30257 (Paragraph 4.4.1a): application of shock 5 times in each of 3 axes (200 g), 0.7 millisec. The transducers were then checked against the original calibration (Figs. 8 and 9).

Graphic plots using the figures obtained show the error band which includes all the points at the extremes

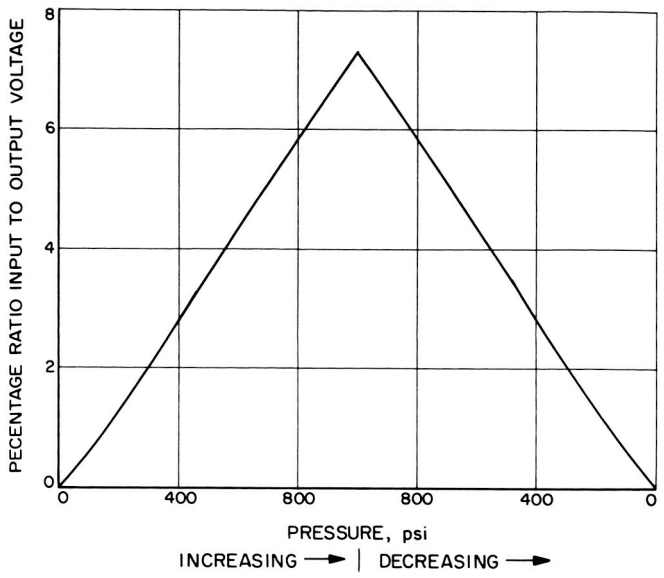


Fig. 8. Resultant error band of combined tests, Micro Systems transducer

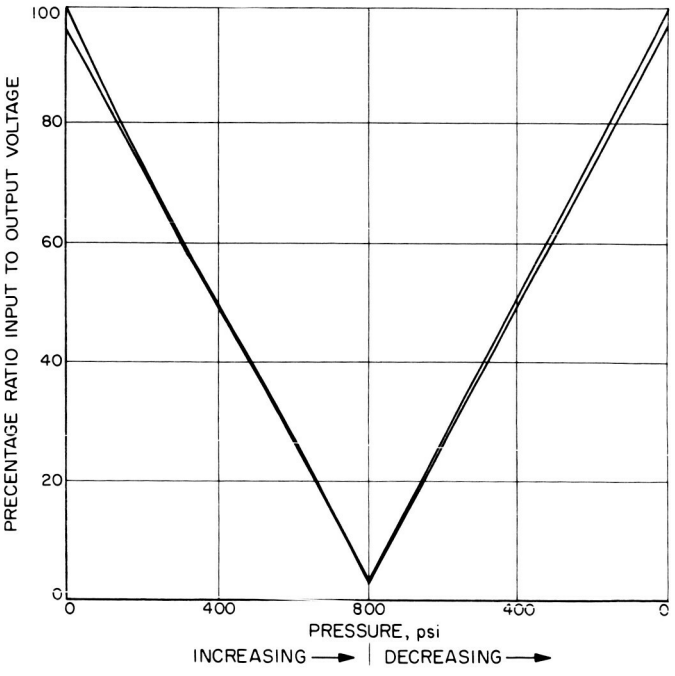


Fig. 9. Resultant error band of combined tests, Giannini Controls transducer

of variation. These extreme points are within ±2% of normal. It must also be noted that these figures would be less than ±2% if the actual figures not included in an error band were used according to the generally accepted methods (Figs. 8 and 9).

4. Test Results

The electrical data obtained after each of the transducer tests do not indicate any gross failure. Mechanical inspection of the Micro System transducer — a commercial unit — revealed that construction improvements could be made. A considerable weight saving could be effected by a different design. Although smaller transducers are manufactured by this company, they were not available for testing at the time. Some silicone potting material was used on the connections in the Micro Systems transducer. The quality of this material is questionable for space use, and the material should be replaced.

Post-testing examinations of the Giannini Controls transducer disclosed some discoloration and creep of the potentiometer insulation material. The adhesive and insulation materials used in mounting and insulating the potentiometer resistance wire have presented great difficulties in the past during heat sterilization. Better insulation materials have been recently developed; organosilicone varnish (GP77, Dow Corning Corp., Midland, Michigan), which combines long thermal life with high bond strength, flexibility, and low, cost should be tested more thoroughly for this application and in vacuum.

5. Conclusions.

Pressure transducers that will qualify for deep space applications are available. These transducers can withstand the rigors of dry heat sterilization and gas (ethylene oxide) decontamination. Prior to commitment of any transducer to a specific program, a four-phase study should be initiated:

1. A list of all materials used in the gage should be compiled by the transducer manufacturer. This list should be thoroughly checked and evaluated.

2. Transducers should be selected and subjected to complete sterilization testing.

3. Mechanical and electrical evaluation should then be performed following vibration, shock, and vacuum tests and any of the other type approval tests prescribed by the mission assignment.

4. A representative sample should be life tested to insure that the sterilization process did not introduce latent unreliability.

REFERENCES

1. *Pressure Transducing and Instrumentation Techniques,* WADD Technical Report 59-743, Contract No. AF 33(616)-6181: Vol. I, Book I, General Introduction and Part A (Transducing Techniques); Vol. I, Book II, Part B (Transmitting Techniques); Vol. I, Book III, Part C (Nuclear Radiation Effects); Vol. II, Part A (Improvement to Current Pressure Transducing and Transmitting Techniques); Part B (Unique Techniques); Part C (References), Giannini Controls Corp., Duarte, California.

2. *Transducer Compendium,* Instrument Society of America, Plenum Press, New York, N.Y.

ABSTRACT

Arens, W. E., "Magnetic Tape Recorder Sterilization," *Space Programs Summary No. 37-37, Vol. IV*, Jet Propulsion Laboratory, Pasadena, California.

A program was undertaken to define and solve all of the problems associated with the complete sterilization of a typical spacecraft magnetic tape recorder. The endless-loop *Mariner C* type magnetic tape recorder was chosen as the basis for the transport design because it was closer to actual flight hardware than any other transport under development at JPL. The program was outlined so that each component to be included in the final transport subsystem was individually designed and/or developed for sterilization compatibility and exposed to complete dry heat sterilization tests.

From these tests, a complete experimental and analytical analysis was performed to adequately define any degradation affecting performance and/or life. Where problems existed, attempts were made to solve these problems so that a reliable sterilizable component would be available for inclusion into a sterilizable subsystem. Since these components were to be assembled into a sealed case, the only components requiring ethylene oxide decontamination survival were the pressure seals. Therefore, the program was defined so that only the seal material was exposed to ethylene oxide testing prior to transport assembly.

The components individually considered for dry heat sterilization were (1) magnetic tape, (2) pressure and drive belts, (3) record/playback motors, (4) rotating modules and lubricants, (5) record/reproduce heads, (6) transport plate, (7) preamplifier electronic modules, (8) adhesives, and (9) covers and seals. During the program, further development work in the areas of the magnetic tape and the reproduce head was required prior to transport assembly.

Following qualification of the individual transport components, a complete transport was assembled, subjected to complete sterilization testing, and subsequently life tested.

VII. GUIDANCE AND CONTROL

The Guidance and Control Division is responsible for spacecraft attitude control, on-board guidance and navigation, on-board timing and sequencing functions, and secondary electrical power supply in support of Laboratory projects. Specifically, the responsibilities of the Division with respect to spacecraft systems encompass:

1. Attitude and velocity increment control subsystems, including sensors (inertial, infrared, and optical), servo amplifiers, servo actuators, and cold gas reaction equipment.
2. On-board closed-loop guidance and navigation subsystems, including sensors, integration, and computing equipment.
3. On-board timing, programming, and sequencing equipment.

Sterilization requirements will apply to all the capsules of the *Voyager* Project. The Division is presently studying the effects of sterilization requirements on several of the critical components for which the Division is responsible. Individual papers are presented giving the status of efforts leading to the development of sterilizable components.

A. *Heat-Sterilizable Battery*, A. A. Uchiyama

The Jet Propulsion Laboratory is currently engaged in a program to develop energy storage devices (batteries) capable of meeting the time–temperature requirements of heat sterilization. The objectives of the program are as follows:

1. To develop a new and basic battery and battery component technology consistent with heat-sterilization temperature requirements.
2. To gain the knowledge which will assist in properly designing and fabricating heat-sterilization batteries.
3. To evaluate and test the batteries and parts of batteries developed.
4. To assess and coordinate the progress and results so as to yield an optimized energy storage device.
5. To integrate the results into the schedules of specific flight programs.

The resultant devices will have the capability to serve as sole power source for spacecraft and capsules or in conjunction with other power sources. The technology to be developed under this task is intended to cover as broad a spectrum of battery performance as necessary so that it may be capable of eventual application in a variety of tasks requiring heat sterilization.

1. Technical Discussions

Sterilization has been a part of the JPL battery requirements since 1960. The present activities are based on JPL Specification No. XSO-30275-TST-A, dated May 24, 1963, which defines the thermal sterilization requirements for flight equipment. In order to describe these requirements in terms of battery technology, the procedure is further defined as follows:

> The cell component, cell, or battery to be sterilized shall be exposed to three cycles of heating, each of which has an interval of 36 hr at 145 ±2°C. The temperature of the test chamber containing the items to be sterilized shall be raised to 145°C in not less than 1 hr and sufficient time shall be allowed during warm-up of the chamber to assure that the test items have been sterilized at 145°C. At the end of the 36 hr, the test chamber temperature shall be reduced to room temperature in not less than 2 hr, and the chamber shall be maintained at room temperature for not less than 2 hr prior to starting chamber warm-up for the next heat cycle.

The above procedure, however, only partially qualifies batteries and battery components. To fully qualify, a battery and its parts must not only survive the above procedure but must also be capable of meeting the specific mission power profile or the battery duty cycle. It is this latter requirement that has in part led to misinterpretation of what could or could not be done by batteries under the present state-of-the-art of heat-sterilizable batteries.

The choice of a battery for initial investigation and the approach to the solution of the problems were based on the following considerations.

1. Fulfilling a *Mariner B* launch.
2. Fulfilling a *Mariner* 1966 launch.
3. Uncertainties in the power profile.
4. Limited weight requirement.

5. Existing data on delivery schedules and development time.

The time element necessitated an immediate qualification of existing battery technology in terms of heat-sterilization requirements. Uncertainties of electrical requirements by the users demanded simultaneous studies and tests to uncover parametric knowledge of battery systems for a wide range of applications. The fourth consideration called for a maximum energy density system and design. The following were the candidate systems:

1. Primary silver-zinc.
2. Secondary silver-zinc.
3. Remotely activated silver-zinc.
4. Silver-cadmium.
5. Nickel-cadmium.
6. Thermal cell systems.

The secondary silver-zinc system was selected because of its higher specific energy and volume density, charge acceptance capability, and stand life. In order to meet delivery schedules, it was necessary to select a battery company experienced in the field of sealed, secondary silver-zinc batteries with facilities to immediately fabricate batteries and battery components.

2. Development of a Heat-Sterilizable Silver-Zinc Battery

This section summarizes the work accomplished by several companies working under JPL from 1962 to the present time. Table 1 presents policy events that affected these contracts.

Table 1. Historical events

Date	Contractual events	Policy events
September 1961	Proposals issued for development of sealed, secondary silver-zinc batteries, capable of heat sterilization at 125°C for 36 hr.	Heat sterilization required for both lunar and planetary programs.
April 1962	Contract 950177 made definite.	*Ranger 4* impacted on dark side of Moon.
July 1962	Temperature requirement changed from 125°C to 145°C, 1 cycle of 36 hr.	Notice to waive heat sterilization on certain components.
December 1962		Heat sterilization requirement deleted from *Ranger* Project. *Ranger 6* impacted Moon January 1963.
June 1963	Contract 950364 issued to investigate and develop materials, processes, and designs which may be utilized in silver-zinc batteries.	*Mariner 66* cancelled April 1964.
January 1964	Requirements for 145°C: 36 hr increased from 1 to 3 cycles.	
December 1964	Termination of Contract 950364 initiated and transfer of life test cells to the Naval Ammunition Depot at Crane, Indiana.	

a. *Delco-Remy Co.*

Tests conducted at 125°C. A total of 71 cells were constructed. The tests were performed to evaluate the following:

1. Separators: Three combinations of separators involving four materials were tried.
   a. Dynel plus fibrous sausage casing (FSC).
   b. Permion 300 (modified polyethylene) plus FSC.
   c. Dynel plus Permion 600 (modified cellulose).

   The separator combination of Dynel and FSC was acceptable for heat sterilization. Permion 300 was unacceptable. Cells with Permion 600 gave good electrical results but caused excessive pressures.
2. Case and Seal: Cells using Dynel plus FSC as separators were able to stand 36 hr at 125°C, provided the nylon case was reinforced with fiberglass cloth and epoxy.
3. Voltage Regulation: Voltage characteristics were not appreciably affected at low discharge rates for temperatures between 50 and 125°F. However, at high rates, 30 amp and a temperature of 30°F, there were significant drops in voltage efficiency.
4. Cycles: Some cells yielded 100% of rated capacity after 100 cycles and gave up to 850 cycles but with less capacity.
5. Stand Life: Cells lost 35% of rated capacity when subjected to a 1-month activated stand life after sterilization.

It was further determined that the wet unformed cell, as compared to the wet fully charged, wet discharged, and dry unformed cells, was most likely to withstand sterilization temperatures. Dry unformed cells were capable of withstanding 125°C but required a remotely activated mechanism prior to charging which would *a priori* lower its energy density and life. No extended stand life is available at 125°C.

Tests conducted at 145°C. Tests at this temperature were conducted on individual components as well as on completed cells. The following conclusions were noted:

1. Capacity: A 50% decrease in capacity was noted and attributed to the positive plate degradation. However, this loss appeared to be due to the reaction of the degradation products from the separators with the positive plate rather than an inherent loss in the plate.

2. Seals and Case: Nylon appeared good but must be life-tested. There was no problem with the terminal-to-nylon cover seal. Penton and Celcon materials failed to give proper cover-to-case seal.

3. Voltage Regulation and Electrode Behavior: The open circuit voltage decayed rapidly and was indicative of the positive plate reacting with separator degradation products. Gas evolved for more than 24 hr after sterilization. Heat-sterilized cells caused excessive pressures on formation charge. Negative plates without such binders as polyvinyl alcohol (PVA) gas less than those using PVA.

4. Separators: Dynel and fibrous sausage casing degrade excessively. The following new materials were evaluated: acrylic acid graft on Teflon, methacrylic acid graft on Teflon, sulfonated styrene graft on Teflon, acrylic acid graft on cross-linked high-density polyethylene. These separators still appear to affect capacity although cycle life has not been affected.

b. *Radiation Applications Inc.* Fifty-one additional materials using cross-linked polyethylene have been prepared under Contract 951015. The cross-links are being effected by the use of an electron beam and divinyl benzene with subsequent grafting of functional groups using cobalt-60 radiation. Two of the 51 materials, designated contractually as 110 and 116, have been selected and 500-sq-ft quantities of each fabricated. The 116 material has been tested by RAI and delivered to JPL for further testing. The 110 material has been fabricated but as yet has not been tested by RAI. The materials, particularly 116, as a result of initial but cursory testing, appear suitable to the JPL requirements for a sterilizable separator material.

c. *Narmco.* In this contract (951091), Narmco is to investigate the field of new thermostable polymers as a source for separator materials. Narmco is to synthesize eight distinct thermostable polymers and test them as polymeric compounds and as films. A 500-sq-ft sample of a suitable separator material will be fabricated from one of the eight polymers.

The preparation of the eight polymeric compounds has been completed. One compound shows excellent film processibility characteristics, but it must be modified chemically to lower its resistivity. This is presently being done.

d. *Electric Storage Battery Co.* A contract (951296) was initiated with ESB in September 1965 for the development of a heat-sterilizable, impact-resistant battery for potential application in the 1971 *Voyager* capsule lander. This contract is primarily for the development of the electrochemistry and the cell case. However, some testing of ESB candidate separator materials is included. It is expected that the Narmco or RAI contracts will provide the main source of separator material. ESB at present has 250 sq ft of the 116 material developed by RAI to be tested. The contract has not been in effect long enough to have produced reportable results.

3. Future Plans

Despite the lack of complete success in the development of the heat-sterilizable silver-zinc battery, this system still offers excellent prospects. Future activities will be directed towards the development of new separator materials. The problem of capacity loss in this system is not as serious when it is considered that the proper design choice of active material ratio overcomes this loss in large measure. The new separators being developed are dimensionally more stable than conventional cellophane. Consequently less material, less electrolyte, and smaller case sizes are required, with a corresponding reduction of weight and volume for an equal amount of energy and a resultant increase in energy density. With the proper material and design, this system could afford up to 50 w-hr/lb after sterilization.

4. Other Systems

Nickel cadmium batteries yield energy densities ranging from a few to at best 15 w-hr/lb. Although they are

capable of many thousands of cycles (although not at 100% of depth of discharge), their stand life is short compared to secondary silver-zinc batteries. The Jet Propulsion Laboratory is presently testing and evaluating 60 nickel-cadmium cells for compatibility with sterilization requirements under Contract 951092 with TRW Systems.

B. Actuator Sterilization, *E. F. Koch*

The immediate purpose of this program is the development of heat-sterilizable actuator components for use in attitude control and various hinge actuations on typical spacecraft. Specifically, this consists of the development of gas components for attitude control, antenna actuators, autopilot actuators, and solar vane actuators. The actuators will be applicable for all types of spacecraft and, where needed, for landing capsules and orbiters.

The state-of-the-art of the actuator field is such that heat-sterilizable actuators may be designed and fabricated without any anticipated research and development problems. However, adequate testing of new designs is mandatory for certification of sterilizability. Close quality control of materials used in fabrication is necessary so that the mechanism can qualify for the heat sterilization environment. A technical development that might benefit the industry at large will be an actuator mechanism that can be used at elevated temperatures.

The JPL Actuator Group is presently working on the following items:

1. A new type of jet-vane actuator is under development contracted to the Aeroflex Laboratory as part of the development of an improved autopilot actuator.

2. A small heat-sterilizable actuator motor has been developed and is now being evaluated for heat-sterilization temperatures.

3. A geared actuator using the aforementioned small actuator motor has been designed at JPL and will be tested to sterilization temperatures.

4. Existing actuator components and assemblies will be evaluated at sterilization temperatures.

C. High-Temperature Photocathode Image Dissector, *Edgar S. Davis*

The electrostatic image dissector has been shown to be a superior detector where high sensitivity, long life, and ruggedness are important parameters. The CBS electrostatic image dissector is shown in Fig. 1. Direct-descent Mars landers will require Canopus sensors and, possibly, approach guidance planet sensors and antenna pointing Earth sensors. For all of these applications, the image dissector looks attractive. A program to develop a flight-worthy electrostatic image dissector capable of being heat- and gas-sterilized was undertaken with CBS laboratories in June of 1964.

Gas sterilization (12% ethylene oxide and 88% Freon 12 by weight) is not considered to be a problem. Heat sterilization (145°C for 36 hr) has adverse effects on the most sensitive photocathodes because they generally employ cesium, which is quite volatile at the sterilization

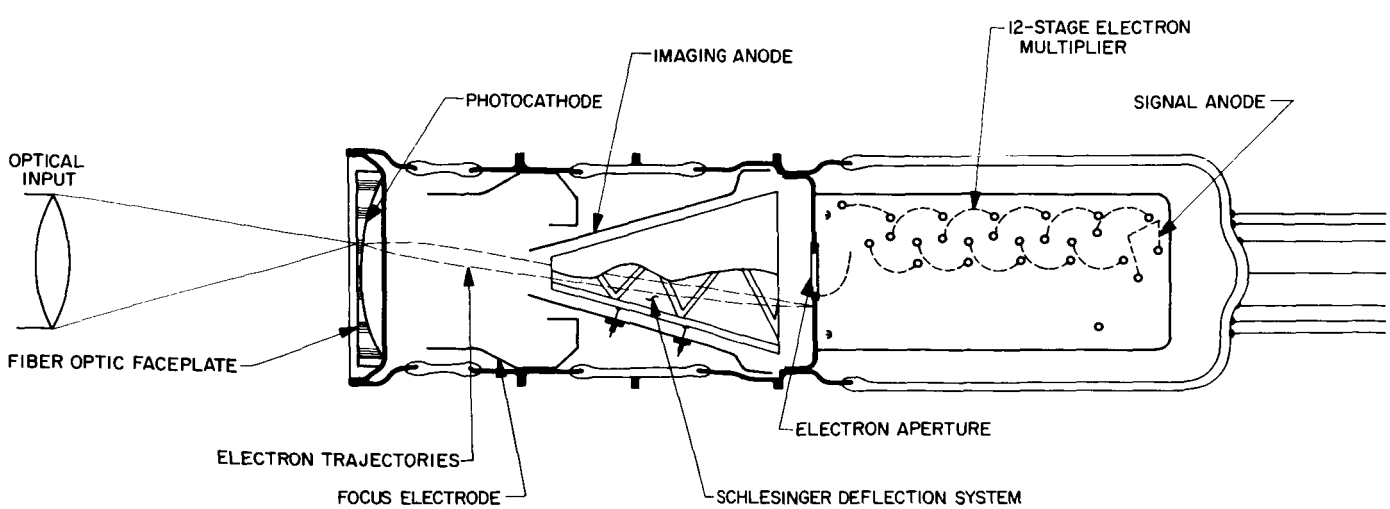


Fig. 1. The electrostatic image dissector

temperature. Because of this inherent weakness in phototubes using cesium, the bi-alkali photocathode has been given primary attention. Other workers in this field have shown that the bi-alkali surface is better able to withstand elevated temperatures if only because the alkali metals sodium and potassium have a lower vapor pressure than cesium and are less likely to migrate. Emphasis was placed on obtaining the highest possible sensitivity. Special attention was paid to the multiplier gain which can be obtained with these tubes, since in the absence of cesium the secondary emission ratio of conventional silver magnesium dynodes is somewhat reduced.

The approach to the problem of achieving useful cathode sensitivity and gain involved the fabrication of over thirty 2-in. photomultiplier tubes, each tube incorporating a processing experiment. Each tube was processed under the closest possible control to study the various effects of the activation schedule on sensitivity, gain, and stability when repeatedly sterilized. Heating and cooling rates, temperature, activation bake times, pressure, photoresponse, gain and leakage were monitored throughout the processing of each tube. After being activated under close control, each tube is subjected to a high-temperature aging cycle. Although this aging cycle reduces the photoresponse of the tube it has a stabilizing effect on the photoresponse and gain for subsequent sterilization cycles.

Each group of tubes has been subjected to six sterilization cycles. General improvements in photoresponse and stability have resulted with each group of tubes. Figure 2 illustrates the effect of sterilization on photoresponse. Two interesting characteristics are worth noting. First, the initial photoresponse is as good as the best S-11 photocathode without the use of cesium. Second, the photoresponse has stabilized by the second or third cycle to a level adequate for sensor applications; 20% stability is considered acceptable for sensor applications. Figure 3 shows that multiplier gain remains quite stable through three cycles of sterilization and does not suffer an initial loss in sensitivity. Although performance is somewhat erratic with subsequent cycles, improvement should come with more experience. Figure 4 shows the spectral characteristics of the bi-alkali tubes before and after sterilization. The factor of 2.0 drop in photoresponse noted in Fig. 2 is primarily caused by a decrease in the "red" sensitivity of the tube. The wave length of the peak sensitivity, 0.45 micron, is not measurably changed by sterilization, and the peak sensitivity is more stable than the photoresponse. Peak sensitivity typically drops by less than a factor of 1.5 when a tube is repeatedly sterilized.

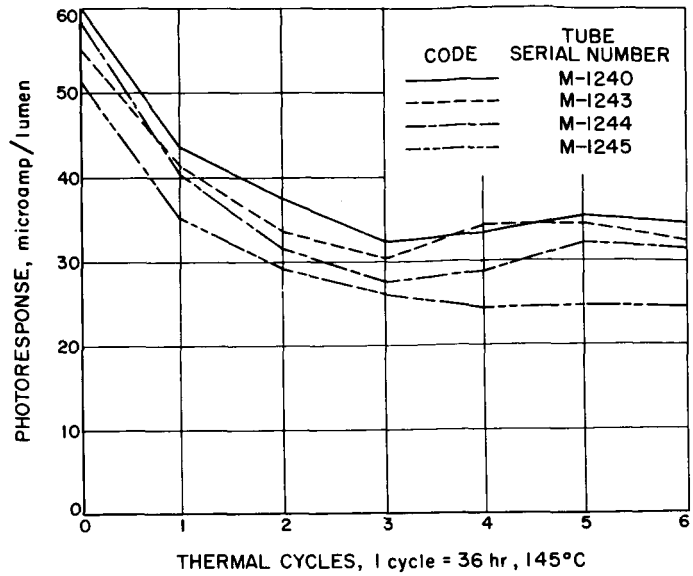


Fig. 2. Effect of thermal cycling on photocathode response

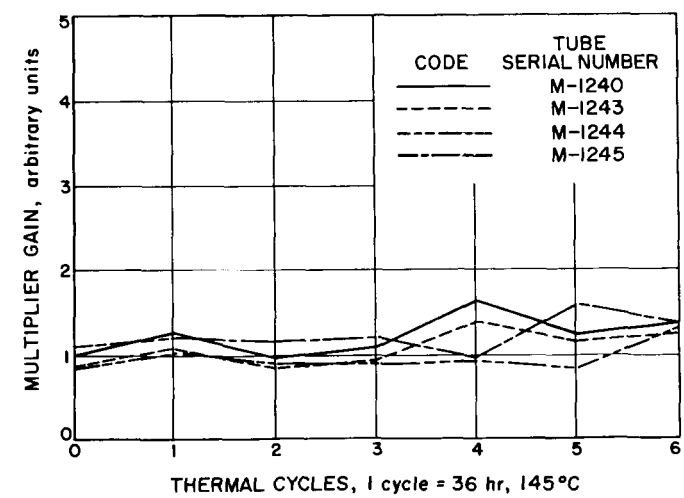


Fig. 3. Effect of thermal cycling on multiplier gain

Transferring the activation process developed in the 2-in. photomultiplier to the image dissector geometry is currently being accomplished. Preliminary results indicate that the performance in the image dissector will be similar to the photomultipliers.

The 2-in. sterilizable photomultiplier tube developed for this study should be useful for many of the scientific experiments being considered for *Voyager* missions. It is recommended that interested scientists should follow up the development of this tube with CBS, since there are no known sensors applications which will require a 2-in. photomultiplier.

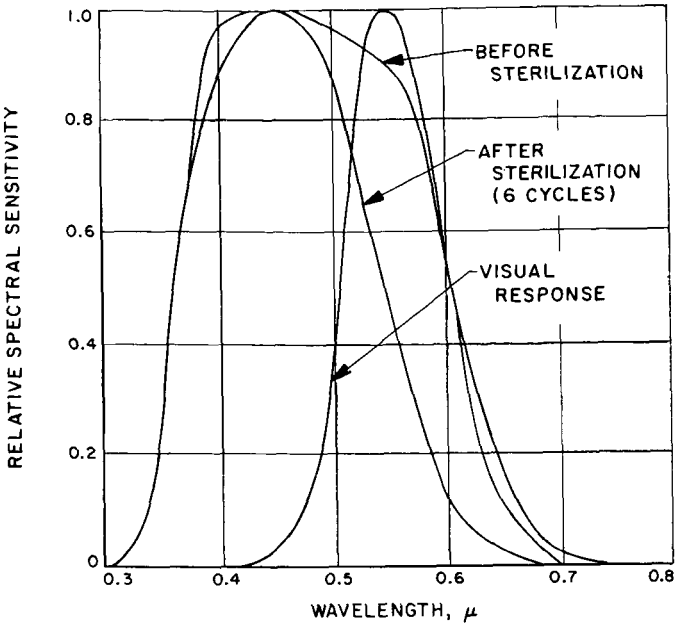


Fig. 4. Spectral response characteristics of the CBS bi-alkali phototube

D. Development of Thermal Sterilization Capability in Inertial Sensors, W. E. Bachman

The requirement for both thermal and gas (ethylene oxide) sterilization of inertial sensors was first initiated in 1961 as part of the *Ranger* Block II program. Thermal sterilization at that time consisted of one 24-hr soak at 125°C. Both gyros and accelerometers were used in the *Ranger* attitude control system and are discussed separately.

1. Honeywell Model GG49 Gyro

Initially, the gyro underwent only minor modifications of sealants and adhesives to make it capable of the 125°C soak. However, in the fall of 1961 a series of catastrophic failures were encountered. These failures were traced to a breakdown of insulation on the wire used in the gyro signal generator and torquer, a combination known as the "Dualsyn." The failed units used a double Formvar-coated wire which was wound and formed on stacked laminations. The gyro manufacturer, Honeywell, conducted a study which determined that Formvar-insulated wire from various manufacturers was of variable quality due to imperfections in the Formvar. They further verified that Formvar was weakened by the 125°C temperature to the point where use after sterilization could result in complete breakdown. After an evaluation of improved insulations, a General Electric product–Nyleze–was found to be the best qualified and was subsequently used in all *Ranger* gyros. No further catastrophic failures occurred after this change. Typical drift performance changes brought on by thermal sterilization showed an average spin axis mass unbalance change of 1.03 deg/hr and an average input axis mass unbalance change of 0.41 deg/hr. These values were considered acceptable for the *Ranger* requirements, but were considered significant because the total mass unbalance drift rate allowable was 4.0 deg/hr.

Gas sterilization was accomplished on all *Ranger* Block II gyros but never caused significant performance degradation. This was undoubtedly because the gyro is a hermetically sealed unit.

Early in 1961 radiation sterilization was attempted on two GG49 gyros using 10^7 R/hr of beta particles from a cobalt source for a period of 36 hr. The first radiated gyro showed a mass shift of approximately 1.0 deg/hr, but after disassembly and inspection the drift rate shift could not be related to the radiation exposure. The second radiated gyro showed no significant performance change and subsequently was successfully life tested for 1000 hr. Further attempts to utilize radiation sterilization were not made because of the limited pile size and operational problems of sterilization of complete gyro module assemblies.

2. Accelerometer Sterilization Experience

As part of the *Ranger* midcourse correction system, an accelerometer was used to initiate motor cutoff. This accelerometer was the Bell Aerosystems Co. Model IIIB. Although no catastrophic failures were induced by the exposure of the instruments to 125°C for 24 hr, bias (null offset) shifts were much larger than could be tolerated without some form of recalibration prior to flight. Table 1 is a list of bias changes for all accelerometers sterilized during the *Ranger* program.

In an effort to correct this deficiency in the accelerometer, a product improvement program was initiated jointly with Bell Aerosystems in September 1960. This program was based on the assumption that the large bias shifts encountered were largely due to warping of the seismic proof mass assembly in high temperatures. The assembly was originally fabricated in several parts that were cemented together with special epoxy adhesives.

JPL felt that a one-piece structure would be more stable at elevated temperatures, and therefore the program was directed toward the development of a method for fabrication and assembly of a one-piece pendulum

Table 2. Sterilization bias shifts: Model IIIB accelerometers (Procedure: hot soak at +125°C for 24 hr; read bias before and after)

System	Accelerometer S/N	Type	Δ bias, μg
RA3 flight	—JR62	B10	−580
RA4 flight	—JR60	B10	−1160
RA5 flight	—JR25	B10	+210
Spare	—JR19	B10	+840
Spare	—JR64	B10	−660
Spare	—JR63	B10	+870
Spare	—JR29	B10	+650
Spare	—JR10	B10	−870
Spare	—JR61	B10	+1650
Vega spare	JR19	B5	+1320
Vega spare	JR20	B5	−350
Block III spare	JR392	B17A	−755
Block III spare	JR393	B17A	−185*
Block III spare	JR393	B17A	−150*
Block III spare	JR394	B17A	+230

*Sterilized twice.

structure. The development effort resulted in a newly designed instrument called the IIIB17; the first production instruments were received in July 1961. The performance improvement obtained from this new design can be seen in the lower part of Table 2. The mean bias shift for the original IIIB5 and IIIB10 types was 833 μg, while the IIIB17 instruments show a mean change of 330 μg. No effort to further improve this performance was undertaken on the IIIB accelerometers, as the thermal sterilization requirements were abandoned on the *Ranger* program.

E. Electrostatic Gyro Sterilization Program, *W. E. Bachman*

The electrostatic gyro (ESG) is an inertial attitude reference sensor which is under development as part of a research program to develop gyros which show potential for significant improvement over conventional sensors in the areas of simplicity, power, and life while providing equivalent or better performance.

The electrostatic gyro (ESG) principle is very different from conventional gyro principles. In an ESG, a spherical rotor is freely suspended in an electrostatic field set up by six diametrically opposed electrodes forming a spherical cavity around the rotor. Optical pickoffs view a pattern on the rotor and provide information to a computer to determine the relative orientation of rotor spin axis and gyro housing (Fig. 5).

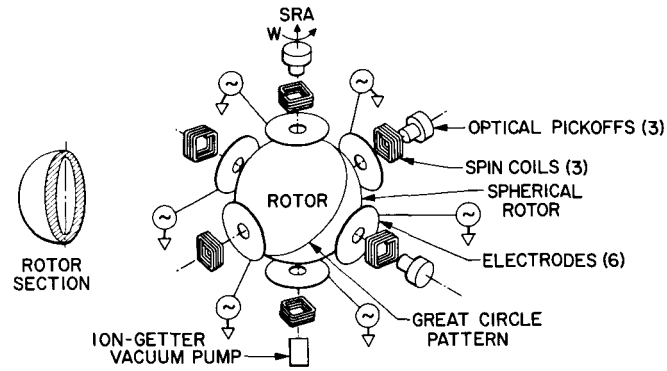


Fig. 5. Electrically suspended gyro

Development of the ESG dates back to 1952, when it was conceptualized at the University of Illinois. Governmental funding was initiated in 1956 and was primarily directed toward development of a gimbaled ESG. The JPL development program is directed specifically toward a strap-down, all-attitude configuration. The JPL development program has proceeded through an initial study program to define the most desirable configuration for spacecraft use under projected environmental conditions; it is now in a prototype evaluation phase. In conjunction with the basic ESG development program, principal components were sterilized to determine what additional development would be necessary to insure that the gyro would ultimately have thermal sterilization capability. The components selected for sterilization are tabulated below:

1. Two rotors.
2. Three envelope halves.
3 One photodiode.
4. One optical pickoff assembly.
5. One pickoff preamplifier.
6. Two suspension system output transformers.

The sterilization results of these components are summarized in Table 3. The components were inspected

Table 3. Summary of results of sterilization tests

Part	Mode of failure	Satisfactory survival of sterilization procedure
Rotor No. 518, unplated beryllium	None	Yes
Rotor No. 213, beryllium, plated with chrome over brass	Blistered, shape changed	No
Envelope half No. 18, pump and electrode surfaces of electroless nickel plated over copper	Blistered	No
Envelope half No. 2, pump and electrode surfaces of electroless nickel plated directly on the ceramic	None	Yes
Envelope half No. 1, pickoff end electrode surfaces same as No. 2	None	Yes
Photo diode, optical sensor	Circuit opened	No
Optical pickoff	None	Yes
Pickoff preamplifier	Output at 20 kc and 100 kc decreased; capacitor in feedback loop changed value	No
Output transformer No. 127	Inductance as indicated by tuning capacitance changed 4%	Questionable
Output transformer No. 128	Inductance changed 4%	Questionable

before and after sterilization to evaluate the following characteristics:

1. Rotor and envelope halves:
   a. Rotor and envelope surface conditions were evaluated by visual inspection for blisters or other flaws.
   b. Rotor and envelope shape were evaluated as defined by Indi-Ron evaluation.
   c. Envelope leakage was assessed as indicated by a Veeco leak detector.
   d. Rotor mass balance was evaluated as indicated by the pendulous period of the rotor when supported in a spherical air bearing.
2. Photodiode, optical pickoff, and preamplifier:
   a. Photodiode was evaluated by continuity tests.
   b. Optical pickoff was evaluated for mechanical shifts and general performance.
   c. Preamplifier performance was assessed by gain measurements at various frequencies.
3. Suspension system transformers:
   a. The suspension system output transformers were checked for changes in turns ratio, inductance, insulation resistance phase shift, and core loss.

The test results indicate that nonplated rotors and electroless nickel plated envelopes can survive sterilization. The optical pickoff assembly also suffered no problems from sterilization. The electronic components, photodiode, and preamplifier did have failure problems. These problems resulted from solid-state components which were not necessarily expected to pass sterilization. It is expected that these problems will be solved by proper selection of sterilizable electronic components. There was also a slight inductance change in the suspension system output transformers the implications of which will require further investigation.

F. Gas-Bearing Gyro Sterilization Development,

W. E. Bachman

The basic gyro being used in this development is a single-axis, rate-integrating gyro of miniature size and guidance performance capability. Gyros of this general type with ball-bearing spinmotors were used on both *Ranger* and *Mariner* spacecraft as rate sensors for spacecraft stabilization and as position sensors for spacecraft maneuvering. The specific gyro on which sterilization development is being conducted has a hydrodynamic gas-bearing spin motor and a hydrostatically fluid-supported gimbal. Such a gyro may find use on spacecraft or capsule missions requiring precision gyro performance exceeding 5000 hr. Principal features of the gyro under development are tabulated below:

1. Single-axis, rate-integrating gyro.
2. Floated gimbal with hydrostatic centering system instead of the conventional pivot and jewel system.
3. Hydrodynamic gas-bearing spin motor.
4. Ceramic motor and gimbal construction.
5. Approximately 3 in. long, 2 in. diameter, 1 lb weight.
6. Approximate performance, 0.02 deg/hr random drift.

Prior to the initiation of a sterilization development program, an early version of the same gyro was loaned to JPL for evaluation. The results of these evaluations indicated that the gyro performance was sufficiently high

and the parameters sufficiently close to desired to allow particular parameter sizing and environmental capability development in order to qualify this instrument for future spacecraft missions requiring a high-performance, long-life sterilizable gyro. In addition to sterilization development, there are other areas of development being supported by JPL to tailor this gyro for future spacecraft system use. These areas are briefly mentioned below in order to show the total development effort.

1. High-g shock and vibration capability is being developed to satisfy launch and preacquisition g-level requirements.
2. Increased torquer scale factor with improved time and temperature stability is being developed to simplify system applications. Increasing torquer scale factor will reduce the attitude-control package weight for existing systems by approximately 2 lb. Improved temperature stability can reduce calibration requirements.
3. A high-frequency suspension pump is being developed to operate with the spin motor power source and thus reduce the types of power sources required for gyro operation. Suspension system bias torques are also being reduced to eliminate the need for electrical bias compensation.

The sterilization development plan was organized into three phases as follows:

1. Review the existing gas-bearing gyro design to uncover obvious weak areas. Conduct material studies and necessary component redesign to achieve a sterilizable gyro.
2. Build and evaluate a dummy gyro test vehicle without a spin motor to guide final gyro design and build.
3. Design, build, and evaluate a final sterilizable gyro.

1. Results of Phase 1

a. Bonding agent evaluations. An evaluation program was conducted to determine structural epoxy strengths before and after sterilization. Test specimens were prepared by joining samples of actual gyro materials with various epoxies. The specimens were strength-tested, sterilized, and then strength-tested again. The test results indicated the best epoxies for particular applications. In general, the epoxies were stronger after sterilization than before.

b. Gyro case evaluation. The original gyro design utilized a case made of 7075 aluminum. Sterilization tests of gyro case test specimens made from 7075 and 6061 aluminum indicated that the latter is about one order of magnitude more stable than the former. Thus, 6061 aluminum was selected as the case material.

c. Differential thermal expansion investigation. Thermal expansion difference between dissimilar materials was investigated to uncover possible areas of overstressing during sterilization. The results of this investigation indicated that the spin motor hysteresis ring and hydrostatic pump stator assembly presented thermal mismatch problems. These components were redesigned to tolerate differential expansion.

d. Flotation fluid evaluation. Before the sterilization development program began, it was known that the existing Bromolube flotation fluid could not withstand 145°C without chemical breakdown. Another flotation fluid, Fluorolube, was selected for evaluation with the component parts. Test samples representing actual gyro materials were immersed in Fluorolube and soaked at 145°C for 1000 hr. Periodic tests were made to evaluate the chemical activity by measuring specimen weight changes and observing specimen surface appearance changes. In most cases, slight material changes did occur but were not of importance. In other cases, significant deterioration took place and appropriate material changes were made.

e. Gimbal redesign. Fluorolube has a lower density than Bromolube, and therefore the gimbal density was reduced to maintain flotation. This was accomplished by reducing the gimbal cross section and increasing its length. The hydrostatic pump and pickoff and torquer assembly had to be moved and modified to allow for the gimbal changes. No problems developed in connection with this design change.

2. Results of Phase 2: Dummy Gyro Test Vehicle

Upon completion of the subcomponent and material studies, a dummy gyro test vehicle was constructed without a spin motor but with all other design changes included. This test vehicle was then evaluated for gimbal torques before and after each of five sterilization cycles. The torque test results are tabulated in Table 4 as an equivalent drift rate in deg/hr. Stability from cycle to cycle is the most important consideration rather than absolute drift rate.

Table 4. Torque test results

Run	300°F soak time, hr	Flex lead torque, deg/hr	Flex lead torque change, deg/hr	Fluid torque, deg/hr	Fluid torque change, deg/hr
Ref.	0	−5.03		−0.51	
Cycle 1	20	−3.54	+1.49	−0.38	+0.13
Cycle 2	39	−0.99	+4.04	−0.48	+0.03
Cycle 3	21	−0.28	+4.75	−0.50	+0.01
Cycle 4	23	+0.47	+5.50	−0.43	+0.08
Cycle 5	21	+0.13	+5.16	−0.47	+0.04

As indicated in Table 4, fluid torques are reasonably stable. This is an important result as it indicates relative stability of the case and gimbal near the fluid flow gaps. However, flex lead torque was very unstable and showed large changes from one cycle to the next. This problem was investigated and corrected by forming subsequent flex leads from a more stable material and cycling at a temperature well above 145°C before installation into the gyro. Additional evaluation of the improved flex leads indicated drift rate shifts from one sterilization cycle to the next on the order of 0.04 deg/hr.

3. Results of Phase 3: Final Gyro Design, Construction, and Evaluation

Following the dummy gyro evaluation, the final gyro design and construction were completed, incorporating all improvements resulting from the investigation program. As part of the development program the contractor was required to establish the gyro presterilization performance coefficients and then subject the gyro to five sterilization cycles with a standard performance evaluation after each cycle (one sterilization cycle consists of a 36-hr soak at 145°C). The contractor progressed through the second sterilization cycle and performance was only slightly outside design goals. The goals are tabulated below as follows:

Performance coefficient	Maximum change from sterilization, deg/hr
Mass unbalance	±0.14
Reaction torque	±0.1

After the third sterilization cycle, however, the spin-motor failed to start. The motor was released by snapping this gyro about its spin axis. It is believed that, in addition to damage caused from sterilization, this snapping could easily have caused reaction torque and mass balance changes. After the motor was released, the drift rates exceeded the design goal. All of the drift test results are given in Table 5.

Table 5. Drift test results

Drift coefficient	Mass unbalance, deg/hr		Reaction torque, deg/hr
	Input axis	Spin axis	
Reference run	+0.53	−0.58	+0.09
First sterilization, change	+0.08	+0.14	+0.04
Second sterilization, change	−0.07	−0.15	+0.01
Third sterilization, change	+0.53	−0.74	+0.09

After a complete pre-teardown evaluation to locate the cause of failure, it was concluded that physical contact between the rotor and some nonrotating motor part was the probable cause. The gyro was then disassembled, and it was discovered that there was physical contact between the motor hysteresis ring and the stator cover. This contact resulted from stator cover distortion caused from thermal expansion differences between the stator cover and dissimilar materials in contact with it. This is one area that was overlooked in the design evaluation. Having discovered the problem area, additional material evaluation and design changes were undertaken. Subsequent extensive evaluation of the redesigned spin motor assembly demonstrated that the redesigned stator cover could survive sterilization without failure. However, a stator winding shorted out after a number of sterilization cycles. This problem was solved by increasing the epoxy back fill to improve insulation. Following this correction the gyro was built up and a reference performance evaluation was conducted. The performance was erratic, and a subsequent gyro teardown exposed a dielectric failure in the gimbal suspension pump. A new pump was built, with special care being exercised to prevent a recurrent failure. Again the gyro was rebuilt, and this time it went through the complete test sequence, including five sterilization cycles, without catastrophic failure.

The test results are summarized in Fig. 6 and Table 6. Referring to Fig. 6, one can see that the mass unbalance and reaction torque have a definite trend with each sterilization cycle. The mass unbalance trend may be caused from flotation fluid absorption; the reaction torque shifts are probably due to flex lead null shifts. Fluid torque is reasonably stable, indicating case stability. The overall performance shift with sterilization cycles did not

Table 6. Torque shifts and random drift

Run	Torque shifts, deg/hr			Random drift (4 hr, 1σ), deg/hr		Drift trend, deg/hr	
	Reaction torque	Mass unbalance, input axis	Mass unbalance, spin reference axis	Output axis vertical	Input axis vertical	Output axis vertical	Input axis vertical
Ref.	—	—	—	0.003	0.002	0.0002	0.0007
Cycle 1	−0.26	0.26	−0.34	0.003	0.002	−0.0007	−0.0004
Cycle 2	−0.07	−0.02	−0.00	0.001	0.001	0.0002	−0.0002
Cycle 3	−0.07	−0.08	−0.07	0.001	0.002	−0.0005	−0.0010
Cycle 4	−0.09	−0.08	−0.10	0.001	0.002	−0.0005	−0.0009
Cycle 5	−0.04	−0.08	−0.13	0.002	0.003	0.0008	−0.0013

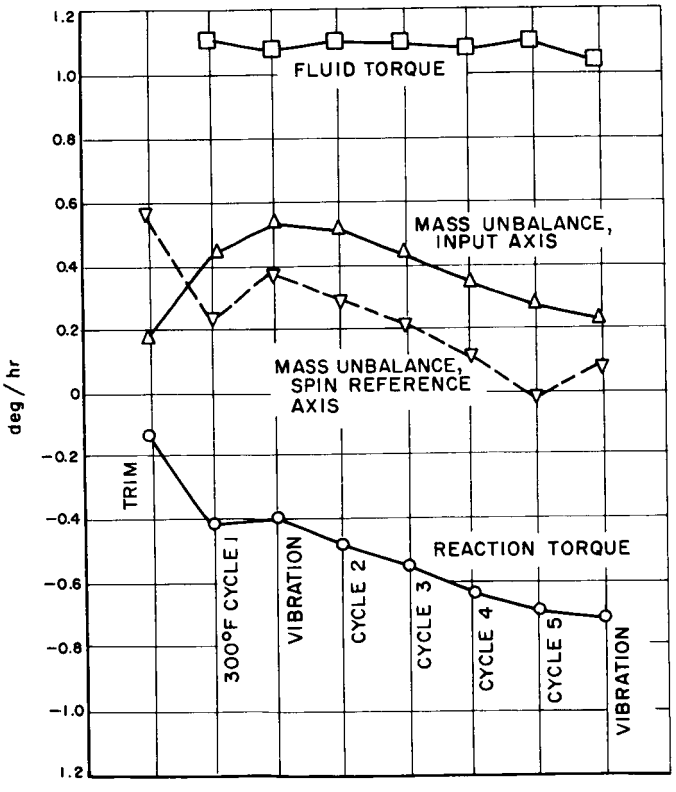


Fig. 6. Gyro balance torque history

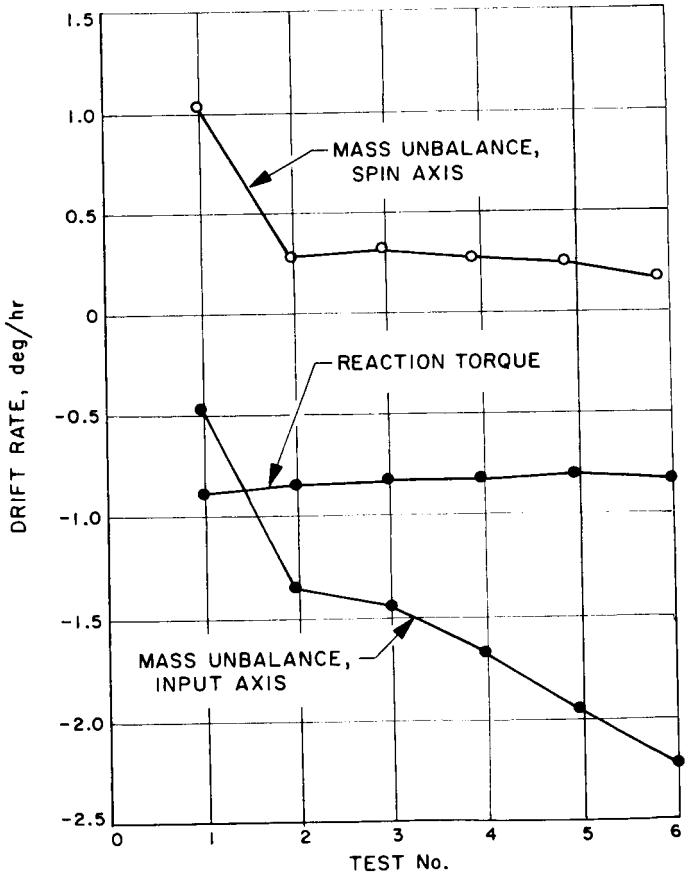


Fig. 7. Gyro data plot

meet performance goals. However, passing sterilization without catastrophic failure is considered a significant accomplishment in itself.

Testing at JPL indicates that g-sensitive drift terms on this gyro are continuing to change while the g-sensitive drift rate appears to have stabilized. These trends can be seen on the data plot shown in Fig. 7. In addition to the data shown, the gyro is also exhibiting a spin motor anomaly wherein the synchronous speed cannot be reached at operating temperatures above approximately 130°F. Normal operating temperature is 180°F. It is presently thought, however, that this malfunction was caused by rotor damage during one of the buildup cycles and is

therefore not an effect of thermal sterilization. The problems encountered during the proof sterilization cycles and in subsequent testing will be further studied on a second sterilization model presently being fabricated and planned for testing near the end of the year.

G. Bell Model VII Accelerometer, *W. E. Bachman*

Early in 1964, Bell Aerosystems announced production of a miniaturized version of the Model IIIB accelerometer. This new design, designated the Model VII, was about one-half the weight and one-third the volume of the Model IIIB. JPL purchased two of these instruments for performance testing and preliminary sterilization evaluation. These instruments were not rated for sterilization temperatures, but were to be used to explore problems associated with the higher (145°C) sterilization temperatures.

Both of the units have been under extensive test since early 1965 at JPL. The first unit was rejected within 30 days for an abnormally high change of bias due to temperature cycling. This unit has now been returned and is being reevaluated at JPL. Early tests show a significant improvement in bias stability. The second accelerometer performed somewhat better on bias shifts but failed under vibration testing in a catastrophic manner. This unit has also been returned to the vendor.

ABSTRACTS

- Uchiyama, A. A., "Energy Storage," *Space Programs Summary No. 37-15, Vol. IV*, p. 78, Jet Propulsion Laboratory, Pasadena, California, June 30, 1962. (This article was erroneously attributed to R. F. Landel and B. G. Moser.)

  A program was initiated to develop an energy storage device which can withstand temperatures up to 145°C for periods up to 36 hours without serious degradation of the electrochemical design characteristics. A contract was awarded to the Delco Remy Division of General Motors Corporation to study the feasibility of existing secondary silver-zinc cells for such purposes. The following parameters were to be studied:

  1. Plate and separator stability.
  2. Case design.
  3. Seal design.
  4. Material suitability.

  The contract for this phase was to be concluded by June 15, 1962, and a final report issued. Preliminary data indicated that presently available cells are deficient in their ability to withstand the sterilization environment.

- Sweetnam, G. E., "Energy Storage," *Space Programs Summary No. 37-16*, Vol. IV, pp. 102–103, Jet Propulsion Laboratory, Pasadena, California, April 31, 1962.

  A contract was awarded to the Delco Remy Division of General Motors Corporation to investigate the feasibility of using existing secondary silver-zinc cells in a sterilizable spacecraft. Original tests were based on a 125°C heat sterilization temperature requirement. At this temperature, many problems were encountered. Particularly, high internal pressures in cells during the sterilization cycle caused mechanical failure of the cell. The contract was extended to allow evaluation of the cells at 145°C.

- Sweetnam, G. E., "Heat Sterilizable Batteries in Electrical Conversion," *Space Programs Summary No. 37-18, Vol. IV*, pp. 62–63, Jet Propulsion Laboratory, Pasadena, California, December 31, 1962.

  Tests were conducted to determine the effect of heat sterilization on an existing silver-zinc battery and to identify problem areas. Results of the test are summarized. It is concluded that heat sterilization of sealed secondary batteries is feasible; however, development work will have to be performed on case design and separator combinations, and the effect on mechanical and electrical characteristics of the sterilization procedure will have to be evaluated.

- Uchiyama, A. A., and Arcand, G. M., "Battery Sterilization Studies," *Space Programs Summary No. 37-30, Vol. IV*, p. 29–32, Jet Propulsion Laboratory, Pasadena, California, December 31, 1964.

  Present battery separator materials do not perform in a satisfactory manner. A contract has been issued to Radiation Applications, Inc., for the purpose of fabrication and testing of battery separator materials resistant to thermal sterilization. An unsterilized cell yields a power output of about 30 w-hr/lb. A similar cell with structural reinforcements yields approximately 10 w-hr/lb after sterilization. The oxides of silver used in the battery plates are somewhat unstable at sterilization temperatures, and decompose to yield metallic silver.

- Lutwack, R., "Development of Separators for Heat-Sterilizable Batteries," *Space Programs Summary No. 37-31, Vol. IV*, p. 65, Jet Propulsion Laboratory, Pasadena, California, February 28, 1965.

  Presently available battery separators for silver-zinc batteries frequently fail catastrophically during the type-approval sterilization cycle of 145°C for 36 hr. A contract has been awarded to Radiation Applications, Inc., to investigate the performance of polyethylene grafted with acrylic acid. Preliminary results are reported.

VIII. ENGINEERING MECHANICS

JPL's Engineering Mechanics Division has the responsibility for spacecraft materials, electronic packaging, structures, dynamics, mechanisms, and environmental survival. The spacecraft sterilization requirement is of particular significance in the areas of materials and electronic packaging. Programs have been completed or are presently underway to determine the effects of sterilization on decelerator parachute materials, polymeric products and formulations, temperature control surfaces, electrical soldered and welded connections, and electronic packaging processes. A parachute sterilization study has been completed and concludes that fabric parachute decelerators are feasible with proper design and material selection. Future programs are planned to investigate impact limiter materials, electrical cables and connectors, and typical electronic assemblies.

Two programs are reported here: polymeric products and electrical connections. Work on the polymer task is about half completed and indicates a sufficient number of materials can endure sterilization satisfactorily. The evaluation of electrical connections is in too early a stage to draw any conclusions but is reported here to illustrate some of the problems caused by sterilization of electronic packaging.

A. Sterilization of Polymeric Materials, H. Harvey

When the requirement to sterilize planetary spacecraft was established in 1963, there was anticipation of problems ahead with the polymeric products[1] then in use. There was good reason for this concern: only two years earlier, when the *Ranger* lunar spacecraft was to be sterilized, considerable difficulty was encountered. Encapsulating materials shrank, causing electronic equipment failures; elastomeric seals leaked, and wire insulation cracked. If similar problems are to be avoided on the *Voyager* Project, polymers compatible with sterilization must be identified. To accomplish this, a test program is underway to evaluate these products after exposure to the sterilization environments. Some of the program highlights are reviewed here.

The procedure developed for sterilizing spacecraft may be accomplished in two steps. The first is a decontamination procedure to lower the microbiological population; the second is a thermal sterilization cycle that kills the remaining organisms. Decontamination is done with a gaseous mixture of ethylene oxide and Freon-12, which is applied at the subassembly level and to the entire spacecraft after it is completely assembled. Thermal sterilization is performed by subjecting the completed spacecraft to 135°C for a period of 24 hr.

To insure that flight hardware will survive sterilization, it is necessary to test and evaluate identical hardware by subjecting it to a "type approval" treatment which is a little more severe than the actual flight sterilization treatments used. These "type approval" requirements are stated in two JPL Specifications: GMO-50198-ETS, covering decontamination, and XSO-30275-TST-A, covering thermal sterilization. The first specification requires exposure to a mixture of 12% ethylene oxide and 88% Freon-12 at a relative humidity of 30–50% during two 24-hr cycles, one at 24°C and the other at 40°C. The second specification requires a three-cycle exposure at a temperature of 145°C in dry nitrogen, the duration of each cycle being 36 hr. Basically, these specifications have been used to evaluate polymers in this test program.

The use of polymeric products on spacecraft is extensive. A typical example is the *Mariner IV* electronic subassembly shown in Fig. 1. Some of the products in this unit are epoxy adhesives, Teflon-insulated wire, epoxy and polyurethane encapsulants, Teflon and polyolefin sleeving, and polyurethane conformal coating. The largest use of polymers is in the area of electronic packaging, with electrical cabling and associated hardware a close second. Other uses (in addition to electrical) are typically for thermal insulation, seals, bearings, structural adhesives, battery cases, and potting. The success of future space missions depends on reliable performance of these products.

This program is aimed at evaluating polymers for the effects of decontamination and sterilization only. It must be understood that many other factors such as vacuum stability and radiation resistance must be evaluated before material is qualified for spacecraft use.

1. Test Program

The selection of polymeric products to be tested was given careful consideration. It was decided that products already used in spacecraft applications should be evaluated first instead of looking for new products that might

[1]Products are defined as compounded polymeric formulations sold under a trade name.

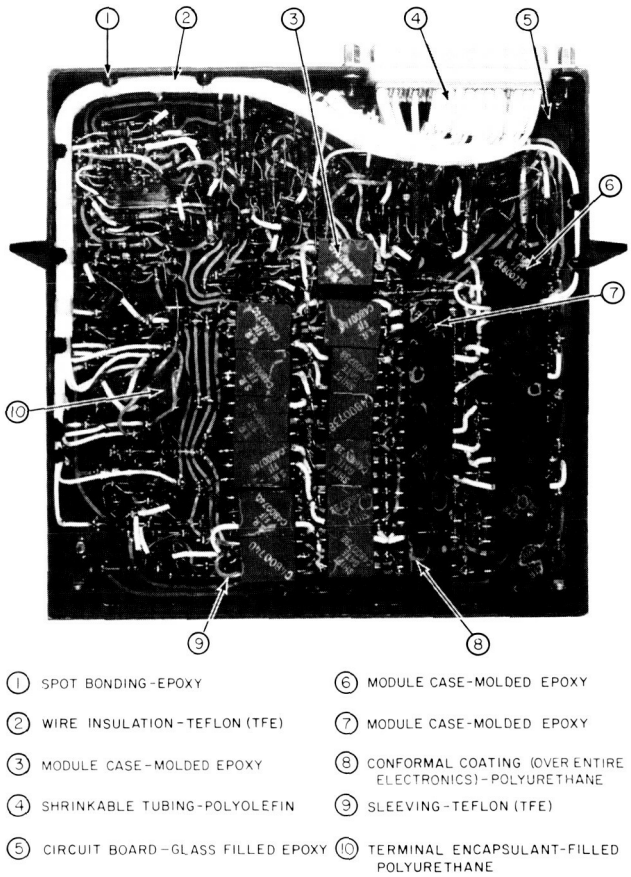


Fig. 1. *Mariner IV* electronic subassembly

be more amenable to sterilization. This approach has several advantages. For example, these polymers have already been proved capable of meeting the demands of spacecraft service, and some of them have been the subject of considerable effort expended over the years in the development of processing techniques, specifications, and approved product sources. The program was started by testing the products used on *Ranger* and *Mariner* spacecraft. The products presently under test are shown in Table 1 together with product types and suppliers.

The test plan used for evaluating materials is shown in Fig. 2. It is accomplished in four steps, (1) preliminary screening, (2) ethylene oxide evaluation, (3) thermal evaluation, and (4) a combination of ethylene oxide and thermal evaluation. Products that pass preliminary screening are continued on through the remaining steps of the program. Preliminary screening is carried out so as to discover the very poor products early, before much effort is expended on them. This screening is accomplished by exposing each product to a single 36-hr cycle at 145°C and evaluating it for visual effects, volume change, weight change and, in some cases, tensile strength. If the screening tests show any gross changes in properties, the product is rated incompatible with sterilization and is not tested further.

Products that pass preliminary screening are subjected to further exposures and testing. The next step for products that pass screening is exposure to the three-cycle heat environment. The products that fail this exposure are dropped from the program. The products that pass are carefully considered, and a selected number for each spacecraft application are continued on through ethylene oxide exposure and the combination ethylene oxide and heat exposure.

Exposure to the ethylene oxide and thermal environment is done in accordance with the type approval treatments previously described, except that the thermal treatment is increased in temperature to 149°C and the time for each cycle is increased to 40 hr to provide a little margin over the type approval treatments previously described.

After a product is exposed to heat, ethylene oxide, or the combined environment, it is tested to determine if it has been degraded. To completely evaluate each polymer for its actual spacecraft application would require a very large and complex test program. Because funds and time are not available for such a program, a simpler approach is taken. Products in the program are divided by function into seven categories: adhesives, coatings, encapsulants, films, reinforced and unreinforced plastics, elastomers, and tapes. Standard test methods are selected for evaluating products in each category (see Table 2). The disadvantage in this approach is that it only screens products and does not actually qualify them for use on the spacecraft; many other tests must be performed before a product can be truly qualified for spacecraft use. But the method does provide a good basis for selecting products which will later be evaluated more fully for specific application.

Reviewing the technical literature available on temperature and ethylene oxide effects on polymers, we hoped to find some help in making selections of products compatible with sterilization. Much information was available relative to thermal effects on pure polymers, but it was surprising to find how little there was on compounded products. We did not expect to find much on compatibility with ethylene oxide, although a small amount of such work was done in the early part of the *Surveyor* Project.

Table 1. Polymeric products presently being evaluated for the effects of ethylene-oxide decontamination and thermal sterilization

Tradename	Manufacturer	Material type
Adhesives		
A-4000	Dow Corning	Silicone
R-823	Carl H. Biggs	Epoxy
206 Cement	Caram	Polychloroprene
3022	Epoxy Products	Epoxy
EC-1614 A/B	3-M	Epoxy polyamide
EC-2216 A/B	3-M	Epoxy
Eccobond 55/9	Emerson Cuming	Epoxy
Eccobond 55/11	Emerson Cuming	Epoxy
Eccobond 56C/9	Emerson Cuming	Epoxy
Eccobond 57C-A/B	Emerson Cuming	Epoxy
Epon 8/A	Shell Chemical	Epoxy
Epon 422	Shell Chemical	Epoxy phenolic
Epon 828/A	Shell Chemical	Epoxy
Epon 828/Z	Shell Chemical	Epoxy
Epon 901/B-1	Shell Chemical	Epoxy
Epon 901/B-3	Shell Chemical	Epoxy
Epon 924A/B	Shell Chemical	Epoxy
FM 96	Bloomingdale Rubber	Epoxy
FM 1044	Bloomingdale Rubber	Epoxy polyamide
GT 200	Schjedahl	Polyester
HT 424	Bloomingdale Rubber	Epoxy phenolic
A2/A	Armstrong Products	Epoxy filled
PC 12-007A/B	Hysol	Epoxy
Pro-seal 501	Coast Pro-seal	Polysulfide
RTV 102	General Electric	Silicone
RTV 108	General Electric	Silicone
RTV 140	Dow Corning	Silicone
Eccobond 26A/B	Dow Corning	Epoxy
EC 1103	3-M	Epoxy
RTV 891	Dow Corning	Silicone
E-3022	Epoxy Products	Epoxy
Hysol 5150/3690	Hysol	Epoxy polyamide
Encapsulants		
Polycel 440R	Polytron	Polyurethane
PR1527A/B	Products Research	Polyurethane
PR1930-½	Products Research	Silicone
Pro-seal 777	Coast Pro-seal	Polyurethane
Scotchcast 3	3-M	Epoxy
Sylgard 182	Dow Corning	Silicone
Stycast 1090/9	Emerson Cuming	Epoxy
Stycast 1095/11	Emerson Cuming	Epoxy
RTV 60/T12	General Electric	Silicone
Hapex 1200/1210	Hastings Plastics	Epoxy
RTV 11/T12	General Electric	Silicone
Stycast 2850 GT/9	Emerson Cuming	Epoxy
PR1930-2	Products Research	Silicone
Scotchcast 241A/B	3-M	Epoxy
Stycast 3050/9	Emerson Cuming	Epoxy
Stycast 1095/9	Emerson Cuming	Epoxy
Stycast 1264 A/B	Emerson Cuming	Epoxy
Stycast 2741/15	Emerson Cuming	Epoxy
Eccocoat IC-2	Emerson Cuming	Polyurethane
Eccosil 5000	Emerson Cuming	Silicone

Table 1 (Cont'd)

Encapsulants (Cont'd)		
Tradename	**Manufacturer**	**Material type**
Stycast 2651/11	Emerson Cuming	Epoxy
Solithane 113/300	Thiokol Chemical	Polyurethane
Epocast 212/951	Furane Plastics	Epoxy
Selectron 5119	Pittsburgh Plate Glass	Polyester
Hysol 4251 (C7-4251)	Hysol	Epoxy polyester
Epocast 202/9615	Furane Plastics	Epoxy
Eccofoam FP/12-6	Emerson Cuming	Polyurethane
Eccofoam S	Emerson Cuming	Polyurethane
Apco Foam 1414-1.5/EPY	Applied Plastics	Polyurethane
Elastomers		
AMS 3195	Rubatex Mfg.	Silicone sponge
No. 805-70	Plastic and Rubber Products	Butyl
Hadbar 1000/80	Hadbar	Fluorosilicone
Hadbar 5000/50	Hadbar	Fluorosilicone
L-308-8	Parker Seal	Fluorosilicone
L-449-6/60	Parker Seal	Fluorosilicone
N-195-7/70	Parker Seal	Nitrile
PMP-6035	Pacific Molded Products	Silicone
PMP-6100	Pacific Molded Products	Silicone
PMP-42011AE	Pacific Molded Products	Neoprene
RTV 501	Dow Corning	Silicone
RTV 615 A/B	General Electric	Silicone
No. 1814	W. J. Voit	Butyl
S-417-7	Parker Seal	Silicone
No. 1050-70	Plastic and Rubber Products	Silicone
No. 391-5	Rubatex	Silicone
No. 391-7	Rubatex	Silicone
SR-349-70	Stillman Rubber	Buna N
Viton 77-545	Parker Seal	Fluorocarbon
Viton B-60	DuPont	Fluorocarbon
Viton B-95	DuPont	Fluorocarbon
B-318-7/70	Parker Seal	Butyl
Hadbar 4000/80	Hadbar	Silicone
Hadbar XB800-71	Hadbar	Butyl
RC-5 No. 1852	Rubbercraft	Neoprene
RC-5	Rubbercraft	Silicone
Silastic 1410	Dow Corning	Silicone
SR 613-75	Stillman Rubber	Butyl
Coatings		
B-276	Westinghouse	Epoxy
Cat-a-lac 443-1	Finch Paint and Chemical	Epoxy
Cat-a-lac 463-1	Finch Paint and Chemical	Epoxy
Corlar 585/586	DuPont	Epoxy
Eccocoat VE A/B	Emerson Cuming	Epoxy
Eccosil 33	Emerson Cuming	Silicone
Varnish 220F	Westinghouse	Alkyd
Hi-Heat Paint 171-A-28	Fuller Paint	Silicone/aluminum
Insl-X-U86	Insl-X Products	Melamine
Interchemical 12412	Interchemical	Fluorosilicone
Laminar-X500 (white)	Magna Chemical	Polyurethane
W 2374	Fuller Paint	Alkyd/silicone
PR-1902	Product Research	Silicone

Table 1 (Cont'd)

Tradename	Manufacturer	Material type
Coatings (Cont'd)		
TUF-ON 747-8	Brooklyn Paint and Varnish	Phenol aldehyde
UC-11659	Pittsburgh Plate Glass	Silicone/aluminum
Uralane 241/973	Furane Plastics	Polyurethane
Electrofilm 2396	Electrofilm	Sodium silicate/MoS_2
Electrofilm 4306	Electrofilm	Phenolic/MoS_2
Alkanex Varnish 9522	General Electric	Alkyd polyester
B224-2 Varnish	Westinghouse	Alkyd
Cat-a-lac 463-1-8	Finch Paint and Chemical	Epoxy
Eccocoat EC200	Emerson Cuming	Epoxy
No. 445 Silicone	Sinclair Paint	Silicone
Pyre-ML Varnish (RK-692)	DuPont	Polyimide
SR-290	General Electric	Silicone
Reinforced and unreinforced plastics		
Diall 52-20-30	Mesa Plastics	Diallyl phthalate
Diall FS-4	Mesa Plastics	Diallyl phthalate
Diall FS-10	Mesa Plastics	Diallyl phthalate
EG-758-T	Mica	Epoxy/glass
Fiberglass 91LD	American Reinforced Plastics	Phenolic/glass
Micarta H-5834	Westinghouse	Phenolic/glass
Laminate 500J	Budd	Epoxy/glass/Cu
EG 752	Mica	Epoxy/glass
Laminate NS	Plastic Center	Phenolic nylon/glass
Lexan 103-112	General Electric	Polycarbonate
Lexan 133-122	General Electric	Polycarbonate
Micarta 8457G-10	Westinghouse	Epoxy/glass
Micarta Grade 238	Westinghouse	Phenolic/linen fabric
Micarta H17480 (G-10)	Westinghouse	Epoxy/glass
Micarta H-2497 (G-11)	Westinghouse	Phenolic
Micarta HY180 (G-10)	Westinghouse	Phenolic
Micarta LE-221	Westinghouse	Phenolic/linen
XP-206	3-M	Epoxy/glass
Films		
H-Film (Kapton)	DuPont	Polyimide
Mylar Type C (1 mil)	DuPont	Polyester
Mylar Type D (3 mil)	DuPont	Polyester
Mylar Type M22 (1 mil)	DuPont	Polyester
Tedlar 200 AM 30wH	DuPont	Polyvinyl fluoride
Mylar Type A (10 mil)	DuPont	Polyester
Mylar Type HS (0.65 mil)	DuPont	
Mylar Type D (5 mil)		
Oils and greases		
Aeroshell 7A	Shell Chemical	Polyester/lithium soap
Apiezon Grease T	Shell Chemical	Hydrocarbon
DC-5	Dow Corning	Phenylmethyl siloxane
Diallylphthalate	Union Carbide	Phthalate ester
DC-11	Dow Corning	Silicone
DC-200-350CS	Dow Corning	Poly(dimethylsiloxane)
Versilube F50	General Electric	Silicone

Table 1 (Cont'd)

Tradename	Manufacturer	Material type
Tapes		
Mystic 7000	Mystik Tape Products	Glass fabric/silicone Adhes.
Mystic 7351	Mystik Tape Products	Mylar/rubber adhes.
Scotch No. 67	3-M	Glass fabric/epoxy
Tape No. 27	3-M	Glass fabric/epoxy
Tape No. 7455	Mystik Tape Products	Al-glass/silicone adhes.
Impregnated fabrics		
SRD 5905	3-M	Silicone/dacron
Ink		
D25W2	Sherwin Williams	Alkyd
No. 73X	Independent Ink	Proprietary
Perma-dri-ink 177	Acme Marking Device	Proprietary

Table 2. Tests for evaluating compatibility of polymers with decontamination and sterilization treatments

Test	Product category								Test Method
	Adhesives	Coatings	Elastomers	Encapsulants	Films	Oils and greases	Reinforced and unreinforced plastics	Tapes	
Tensile strength			X		X		X	X	ASTM D-638, D-412, D-882
Elongation			X		X				
Tensile shear strength	X								FTMS 175-1033/ASTM D-1002
Adhesion		X							ASTM D-2197
Compression set			X						ASTM D-395
Tear resistance					X				ASTM D-624
Hardness			X	X			X		ASTM D-676/ASTM D-1706
Viscosity/penetration						X			ASTM D-2196, ASTM D-273
Volume change			X	X					
Volume resistivity		X	X	X	X		X	X	ASTM D-257
Surface resistivity		X	X	X	X		X	X	ASTM D-257
Dielectric strength		X	X	X	X		X	X	ASTM D-257
Flexibility		X							FTMS 141-6223

Manufacturers' bulletins and catalogs proved to be a good source of information. They usually contained some clue to the heat compatibility of specific products. In most cases this information was given as a maximum useful temperature. In addition, much information was gained on both the methods for mixing and preparing products properly and on the room temperature properties of these products.

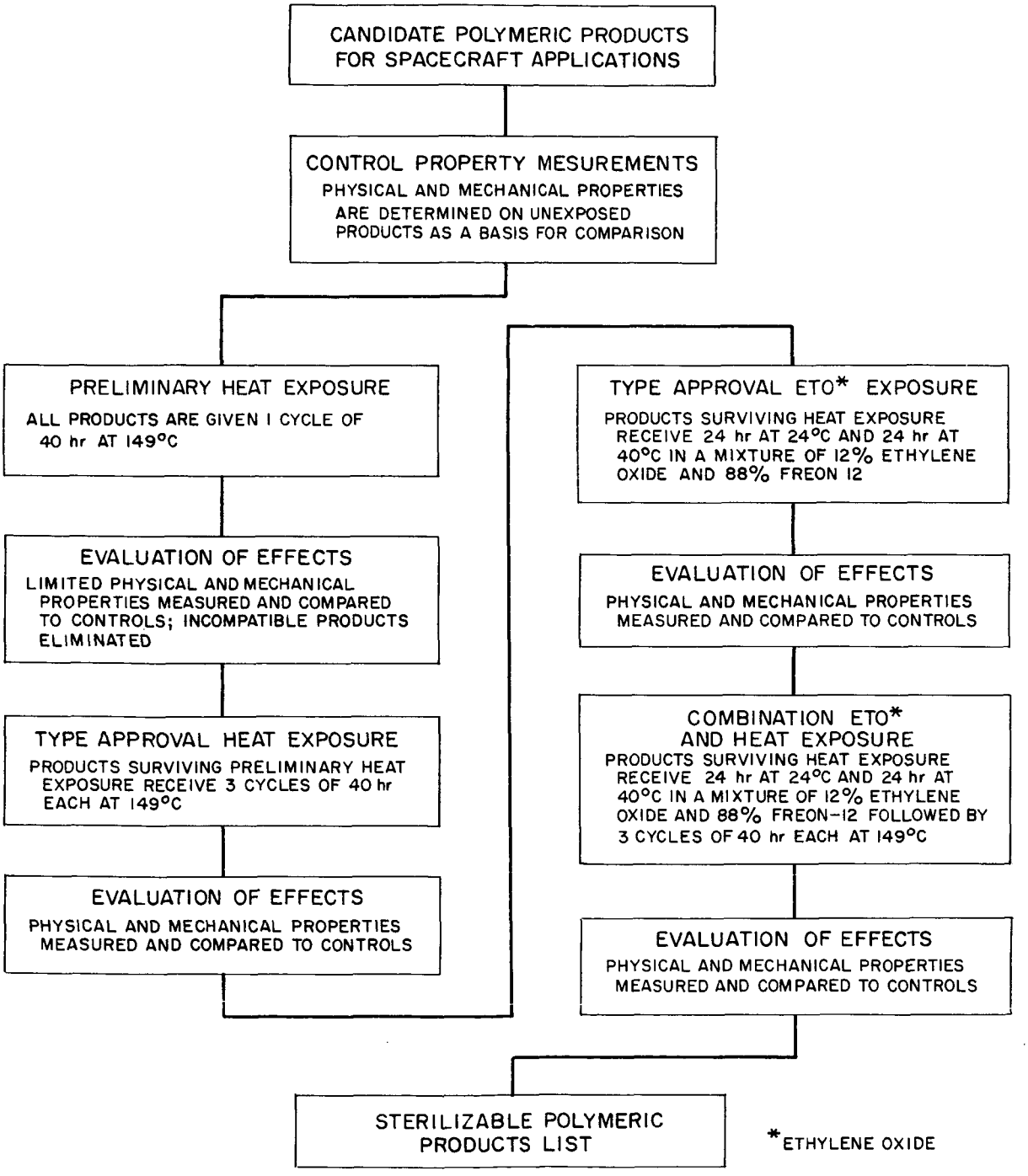


Fig. 2. Plan for evaluating polymeric products for the effects of ethylene oxide decontamination and thermal sterilization

2. Results

Each product evaluated in this program is rated in one of three classes: compatible, marginal, or incompatible. The criteria used are very simple. For a material to be compatible its property data after exposure must show no degradation. A marginal rating is given when some loss in properties occur, and only when a product is degraded so badly that its usefulness is limited is it rated incompatible. Where the line should be drawn between marginal and incompatible is established by judgment. Products rated marginal or incompatible should be used only after careful consideration. However, if their losses in properties can be tolerated, these products still can be used in special situations where compatible materials cannot meet requirements. It is important, however, to carefully study property changes with respect to intended use. A change in any property indicates a lack of stability in the product.

The results obtained in the program show the majority of products to be compatible with sterilization. Data obtained so far show about 20 to 25% of the products tested to be marginal or incompatible. Most products show considerable resistance to heat and ethylene oxide exposure. In fact, some products are actually improved after heat cycling. The few products found incompatible or marginal appear to have compatible substitutes. Although much testing remains to be done, we are confident that no unsurmountable problems exist with the use of polymeric products on sterilized spacecraft.

Of the adhesives evaluated for thermal exposure, only six have thus far been found degraded. These all suffered from a loss in shear strength. Several other adhesives tested had increased shear strength after exposure. This probably is due to post-curing brought on during thermal cycling.

Encapsulants appear to be the only category where a potential problem exists. Of seven products tested, three had excessive volume changes after either heat or ethylene oxide exposure — one as high as 3.7%. Two others experienced a considerable reduction in hardness. A small volume change in these materials is critical to their successful use.

The data on products in most other categories show only a few products in each category to be affected. Reinforced and unreinforced plastics show very good resistance, with only two marginal ratings up to now. Among the elastomers, four products were found to be marginal because of loss in strength and increased hardness. All of the coatings have so far been found compatible, except for three temperature-control coatings that discolored. The only startling results obtained are on two very excellent high-temperature films, H-films and Tedlar 200. Both of these lost significantly in tensile strength and elongation after exposure to ethylene oxide. Subsequent exposure to heat restored some of the strength to the H-films.

The completed results on the list of products now in the test program should be available in the near future. If everything goes according to schedule, the thermal exposure testing should be completed sometime in December 1965; the other two testing exposures should be completed sometime early in 1966.

3. Discussion

The resistance to thermal exposure is difficult to predict for polymeric products that are compounded with other ingredients. Most of the base polymers used in preparing products are thermally stable. Typically, they are silicones, epoxies, phenolics, fluorocarbons, and polyurethanes. However, in compounding products to do a specific job, ingredients are added to alter the natural properties of the base polymer that also can alter the heat resistance of the product. These ingredients are classified as plasticizers, curing agents, flexibilizers, accelerators, stabilizers, flow-control agents, pigments, and fillers. They are used to improve flexibility, resilience, impact resistance, and water vapor transmission, to lower shrinkage, to raise or lower viscosity, to increase pot life, to lower exothermic temperature rise, to reduce cost, to lower density, to improve adhesion, etc. With these modifiers, the properties of the base polymers can be changed to fit many applications (which is what makes polymers so versatile).

If only the pure base polymers were being dealt with, the job would be easy. These polymers have been studied by several investigators and have been found quite stable even after exposure to temperatures above that with which we are concerned. Decomposition of the base polymer is not the problem. What happens to the other ingredients is what causes trouble. Many ingredients are volatile; some are decomposed; some react adversely with other ingredients or with the base polymer. These are the factors that change the properties of products that have been degraded by thermal sterilization.

Unfortunately, the formulations of polymeric products are usually proprietary. Without information on specific

ingredients, the behavior of a product when heated cannot be understood. Because of this, the thermal stability of most products cannot be predicted until tested.

Although ethylene oxide and Freon-12 are not considered to be very reactive with materials, there is some concern for their compatibility with polymers. Chemically, it is possible for ethylene oxide to interact with a polymer by physical absorption or chemical reaction; both could affect the properties of the product. The requirement that a product be compatible with the combined effect of ethylene oxide and Freon exposure followed by heat exposure can cause additional problems. It is apparent that any chemical reaction taking place between the ethylene oxide and the polymer (or any of its modifiying ingredients) would be accelerated by the 145°C temperature of the heat exposure. Freon-12 used as a diluent in the decontamination gas mixture does not react chemically with most materials. However, it does have solvent properties that can cause swelling of some polymers. There is information reported in the literature on swelling of elastomers after prolonged contact with liquid Freon-12. Some evidence has been found in this program to show that Freon-12 has a similar effect on both elastomers and encapsulants.

From test data so far obtained, it appears that exposure to the mixture of ethylene oxide and Freon-12 is not very damaging to most polymeric products. Significant effects have been found in only two materials, Tedlar-200 and H-film, as previously stated. Physical absorption is believed to be responsible for this behavior, since subsequent thermal exposure of the H-film restores some of its lost strength. However, some chemical reaction could have taken place, and a careful investigation of these effects is being conducted. It is possible that thin films may be severely degraded by reactions that would be superficial in a more massive product and therefore could go undetected.

B. *Effects of Sterilization on Electrical Connections*, R. Holtze

Present-day spacecraft electronic assemblies consist of a combination of complex electronic and electromechanical equipment. This equipment, in turn, is comprised of subassemblies, modules, and components of various materials and shapes which must be interconnected for electrical continuity and packaged or fastened together for subsequent assembly. Figure 3 shows a typical electronic package. Current concern in this area is to reduce size, simplify replacement, provide environmental protection, and assure high reliability.

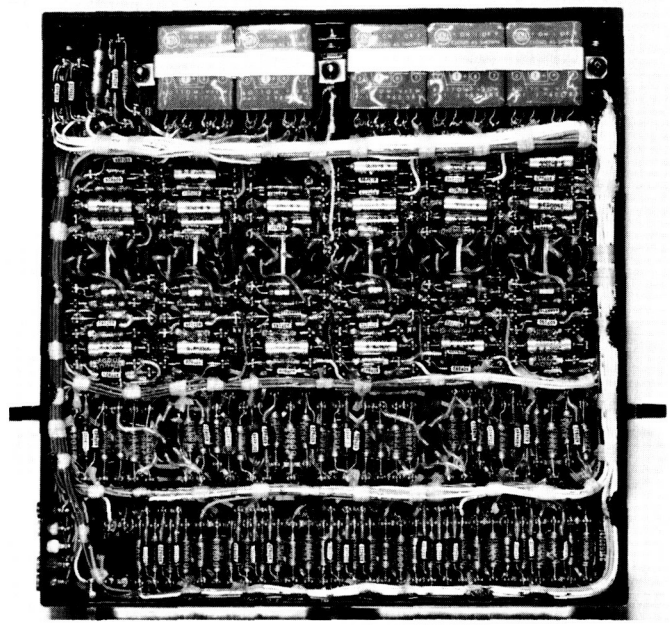


Fig. 3. Typical electronic package

Most of the electrical connections in these packages are made by soft soldering and resistance welding. Figure 3 shows various types of electronic components with leads soldered to terminals. Figure 4 illustrates the resistance-welded joints in a cordwood module.

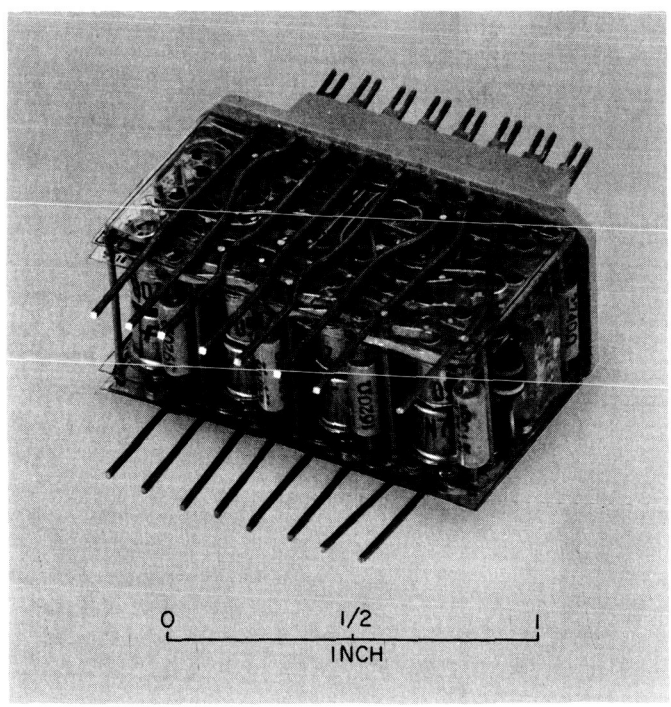


Fig. 4. Cordwood module, resistance-welded joints

1. Problem Discussion

The electrical connections, which are ordinarily required to withstand harness assembly load and environments such as shock and vibration, now additionally are required to withstand heat sterilization treatments. With such complex assemblies of dissimilar materials of various forms and sizes, thermal expansion imposed by heat sterilization can seriously degrade the reliability of electrical connections insofar as their continuity and function are concerned. In addition to determining the external loads on the soldered and welded joints, information is needed on the effects of the heat sterilization treatment on the joints. Although the effects of heat on the welded joints are not expected to be serious, the soldered joints may be appreciably affected.

A literature search revealed that although there is a substantial amount of data on the properties of solder in the temperature region of 300°F, there is little information on the properties and behavior of soldered joints at this temperature and after exposure to this temperature. It is almost impossible to avoid some harness assembly loads on connector cup soldered joints; therefore, the strength of these joints during the heat sterilization compatibility test time and at the required temperature (three 36-hr cycles at 293°F) was ascertained. The mechanical and electrical properties of a variety of soldered joint material combinations after heat sterilization are required in order to determine if changes are necessary for soldered joints in sterilizable electronic equipment.

Metallurgical changes in the solder, if any, are not expected to be of consequence. However, the differences in thermal expansion of the joined dissimilar metals, caused by heat sterilization, could have a substantial effect on the interface characteristics.

Since cable connectors consist of both metallic and polymeric materials, the effects of ethylene oxide decontamination as well as heat sterilization on the electrical properties and dimensional stabilities of some existing connector designs remain to be ascertained.

2. Test Plan

A test program which dealt with eleven different material combinations for soldered joints and seven material combinations for resistance-welded joints was initiated under contract. The program included mechanical strength tests and electrical tests of joints before and after heat sterilization. These electrical tests were conducted before, during, and after vibration. In addition, stress-rupture strength tests at sterilization temperature were conducted on two types of connector cup to stranded conductor soldered joints.

Test work was also performed to determine the effect of ethylene oxide decontamination and heat sterilization treatments on the solderability of the various materials used in the fabrication of solder joints. Metallographic examinations and solderability determinations were made on the joint materials both before and after the ethylene oxide and heat sterilization treatments. This work was performed to determine possible problem areas in making equipment modifications and repairs involving soldering after preliminary decontamination and sterilization treatments.

3. Results

An analysis of the test results showed, with one exception, that no appreciable changes occurred in the physical or electrical properties of the soldered or welded joints due to heat sterilization. The most significant degradation observed in the tests on soldered joints occurred during the stress–rupture tests of the stranded conductor to connector cup joint. It was determined that the stress–rupture strength for 108 hr at room temperature was about 70% of the short-time ultimate strength but that the stress–rupture strength for 108 hr at 145°C was only about 5% of the short-time ultimate strength at room temperature.

Solderability tests of the materials specified for solder joints indicated no appreciable effect resulting from the ethylene oxide decontamination treatment. The heat sterilization treatment did result in poorer solderability of two materials: the solder-coated bifurcated terminals and the gold-plated Dumet leads with nickel undercoat.

The investigation of sterilization effects on connectors has not been completed at this time.

4. Future Work

Additional work will be done to evaluate high-temperature solders for soldering of stranded wires in connector cups. The Sn-63 solder has such low strength at sterilization temperatures that various presently used joint design configurations should be tested to determine if high-temperature solders or other joining methods are necessary.

The results of the tests on connectors will be evaluated to determine if improvements in connector materials or design are needed in order to produce sterilizable connectors with satisfactory performance characteristics.

IX. PROPULSION AND PYROTECHNICS

The Propulsion Division is responsible for research, advanced development, and flight program support in the fields of chemical, nuclear, and electric propulsion systems and related devices. The Division also carries out propulsion system studies and assists in the formulation, monitoring, and direction of industrial contracts in the propulsion area.

To implement the sterilization requirements, the Division has demonstrated that a polyurethane solid propellant can be decontaminated by using ethylene oxide during propellant mixing. Also, the Division is conducting studies and experiments which will yield propellants and propulsion-related devices capable of withstanding sterilization environments.

ABSTRACTS

- Montgomery, L. C., "Sterilization of Solid Propellants," *Space Programs Summary No. 37-17, Vol. IV*, pp. 170–171, Jet Propulsion Laboratory, Pasadena, California, October 30, 1962.

- Montgomery, L. C., "Sterilization of Composite Solid Propellant," *Space Programs Summary No. 37-23, Vol. IV*, pp. 76–77, Jet Propulsion Laboratory, Pasadena, California, October 31, 1963.

  Early exploratory studies showed that polyurethane propellant is not sporicidal *per se*; therefore, it became necessary to determine the feasibility of introducing a chemical sterilant into the propellant. Two approachs, both of which use an ethylene oxide gas mixture as sterilant, are possible. They are (1) to diffuse the gas into the bulk of previously cast and cured propellant, and (2) to introduce ethylene oxide into the propellant during mixing and before curing.

  Both techniques are capable of producing satisfactory decontaminated propellant; however, the diffusion technique is quite slow, because the penetration rate of ethylene oxide into the polyurethane binder is approximately 0.0156 in./hr, and the diffusion rate into the propellant is slightly slower. Using the technique incorporating liquid ethylene oxide in the propellant mixture, one dummy and two live motors of the *Syncom* design have been cast using heat-sterilized motor cases and casting the motors under sterile conditions. The live motors have shown satisfactory operation.

- Montgomery, L. C., "Sterilization of Solid Propellants," *Space Programs Summary No. 37-20, Vol. IV*, pp. 58–60, Jet Propulsion Laboratory, Pasadena, California, April 30, 1963.

  Recently obtained results show that complete sterilization of a propulsion module having state-of-the-art performance can be accomplished. The major breakthrough that allows chemical sterilization of propellant is the demonstration that the introduction of liquid ethylene oxide in percentages up to 16% by weight of the binder does not degrade the propellant and also destroys an inoculum of 1.3×10^6 spores per cc of *Bacillus subtilis* var. *niger*. A concentration

of 6% ethylene oxide in the propellant has been found completely satisfactory from the standpoints of a bacteriology as well as propellant performance in tensile bars and burning rate strands. The remaining work on the study is to demonstrate the casting and performance of a complete self-sterilized motor.

- "Sterilizable Propulsion Systems," *Space Programs Summary No. 37-22, Vol. II, Part IV-B*, Jet Propulsion Laboratory, Pasadena, California (Confidential)
  (Classified material not abstracted)

- Montgomery, L. C., "Heat-Sterilizable Propellants," *Space Programs Summary No. 37-31, Vol. IV*, p. 207, Jet Propulsion Laboratory, Pasadena, California, February 20, 1965.

  Final results of this investigation indicated that none of the commercially available solid propellants satisfied all JPL acceptance criteria after heat sterilization. However, three of these propellants nearly attained the survival limits, and changes in the geometric configuration may eliminate the internal stresses causing failure.

- Benedict, A. G., "Development of Sterilizable Pyrotechnic Devices," *Space Programs Summary No. 37-32, Vol. IV*, pp. 112–114, Jet Propulsion Laboratory, Pasadena, California, April 30, 1965.

  Explosive devices in common use in the aerospace field are usually actuated by squib initiators. The design of such explosive devices to withstand thermal sterilization is generally routine, but designing squib initiators to withstand thermal sterilization is more complex because of the limited range of materials suitable for squib construction; the relatively small size of the squibs precludes the use of sophisticated expansion joints dependent on precise machining.

  Although several available squib initiators can withstand thermal sterilization, none incorporates the range of features desirable for JPL spacecraft application, and it was consequently decided to evolve a new basic squib initiator adaptable to various purposes. The approach to the design consisted of selection and optimization of the insulating insert and the bridge configuration, selection of the matchhead, and optimization of the body details and the mating connector.

  The squib seal offered the major problems; epoxies and thermosetting plastics were abandoned as seal materials after preliminary consideration because of unattractive thermal characteristics and strengths. Glass seals can withstand pressures of about 30,000 psi, but present a problem in meeting a need for an insert which is thermally conductive but electrically insulating. Ceramic seals appeared most promising, and development work by JPL and by two contractors has since led to evolution of a satisfactory basic design for ceramic seals.

  Prototype deposited-film bridges produced at JPL have performed well in the face of severe temperature shock (−300 to +300°F), and have demonstrated excellent no-fire characteristics. Proposals for development of production techniques are now being requested.

  Work remains to be done on anti-static-discharge features; responses to requests for proposals in this area are currently under review.

Sterilizable primers would be needed for spacecraft explosive devices that are mechanically rather than electrically initiated. Two 400°F primer mixes (G-11 and G-16) developed for Frankford Arsenal by Remington Arms will withstand thermal sterilization, but tests have shown these primers to be very sensitive to mishandling; it will be necessary to examine this problem carefully if high reliability is to be achieved. Preliminary tests will be made at JPL.

- Curtis, H. D., and Harper, A. D., *Optimization of System Operating Parameters for Heat-Sterilizable Liquid Propulsion Systems,* Technical Memorandum No. 33-211, Jet Propulsion Laboratory, Pasadena, California, June 1, 1965.

  As a means of attaining a sterile spacecraft, design information is required for liquid propulsion systems which can be heat-sterilized in the loaded condition without venting. An analysis was performed to determine the values of the system operating parameters which minimize the system mass. Results are presented for both internally and externally pressurized tankage systems. The dry weight of sterilizable systems is approximately twice that of comparable non-sterilizable systems. The system mass is minimized when the tank is 60 to 70% filled with propellants (prior to heating) in the externally pressurized case and approximately 40% filled in the internally pressurized case.

- Stanford, H. B., "Hydrazine Sterilization Tests," *Space Programs Summary No. 37-35, Vol. IV,* Jet Propulsion Laboratory, Pasadena, California, September 30, 1965.

  The feasibility of sterilizing a liquid fuel, hydrazine, was demonstrated. A small, 6.5-in.-diameter 6-Al-4V titanium tank was filled with hydrazine to about 60% of capacity, sealed, and subjected to three heat cycles of 293°F for 35½, 37, and 46½ hr, respectively. After the third cycle and cool-down to room temperature, an increase in pressure (as a result of decomposition) of 16 psi over the initial tank pressure (7 psig) was recorded. Since some concern has been previously expressed as to the ability of this fuel to withstand this type of temperature cycling, it was felt that these results, although limited in scope, showed evidence of the practicality of employing the fuel, either as a monopropellant or bipropellant, for capsule applications.

- Marsh, H. E., and Montgomery, L. C., *Sterilized Solid-Propellant Rocket Motors for Mars Landing Missions,* Technical Report No. 32-725, Jet Propulsion Laboratory, Pasadena, California, March 30, 1965 (Confidential).

  (Classified material not abstracted)

- Marsh, H. E., and Montgomery, L. C., *Sterilized Solid-Propellant Rocket Motors for Mars Landing Missions, Revision 1,* Technical Report No. 32-725, Jet Propulsion Laboratory, Pasadena, California, June 30, 1965.

  In the initial phase of the program to develop a sterile solid-propellant motor for planetary and lunar landings, the chemical sterilization approach was taken. Also, the chemical sterilization of a state-of-the-art propellant has been demonstrated. Current objectives, however, require the heat-sterilization approach. The ultimate test criterion for the heat sterilizability of a spacecraft component is the ability of the component to survive three cycles of exposure to 145°C for

36 hr. "Off-the-shelf" propellants from seven U.S. manufacturers were included in the initial tests. Three candidates were eliminated in initial sterilization cycle slump tests which were performed in both air and nitrogen atmospheres. Having retained some integrity in the slump test, the remaining four propellants are evaluated for the physical and ballistic changes that occur because of the sterilization treatment. The significant crusting of the propellants and its related physical effects are evaluated. The development of a silicone propellant for heat-sterilization application is also discussed.