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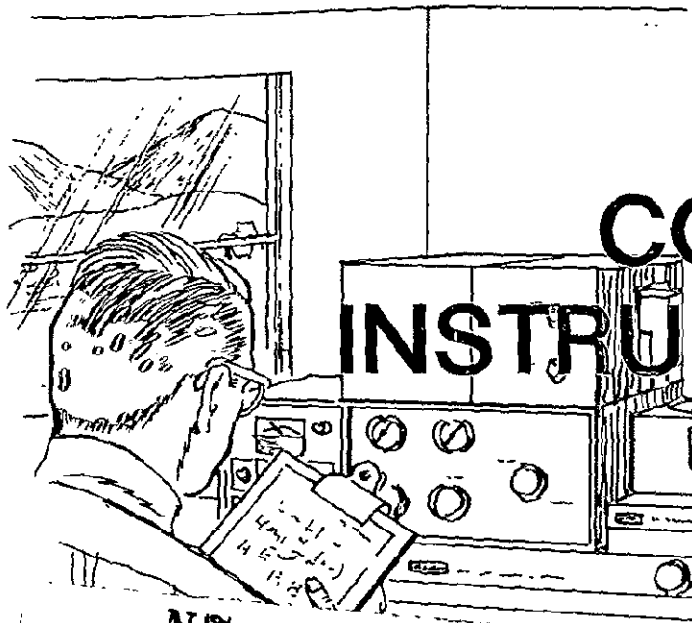


FINAL REPORT

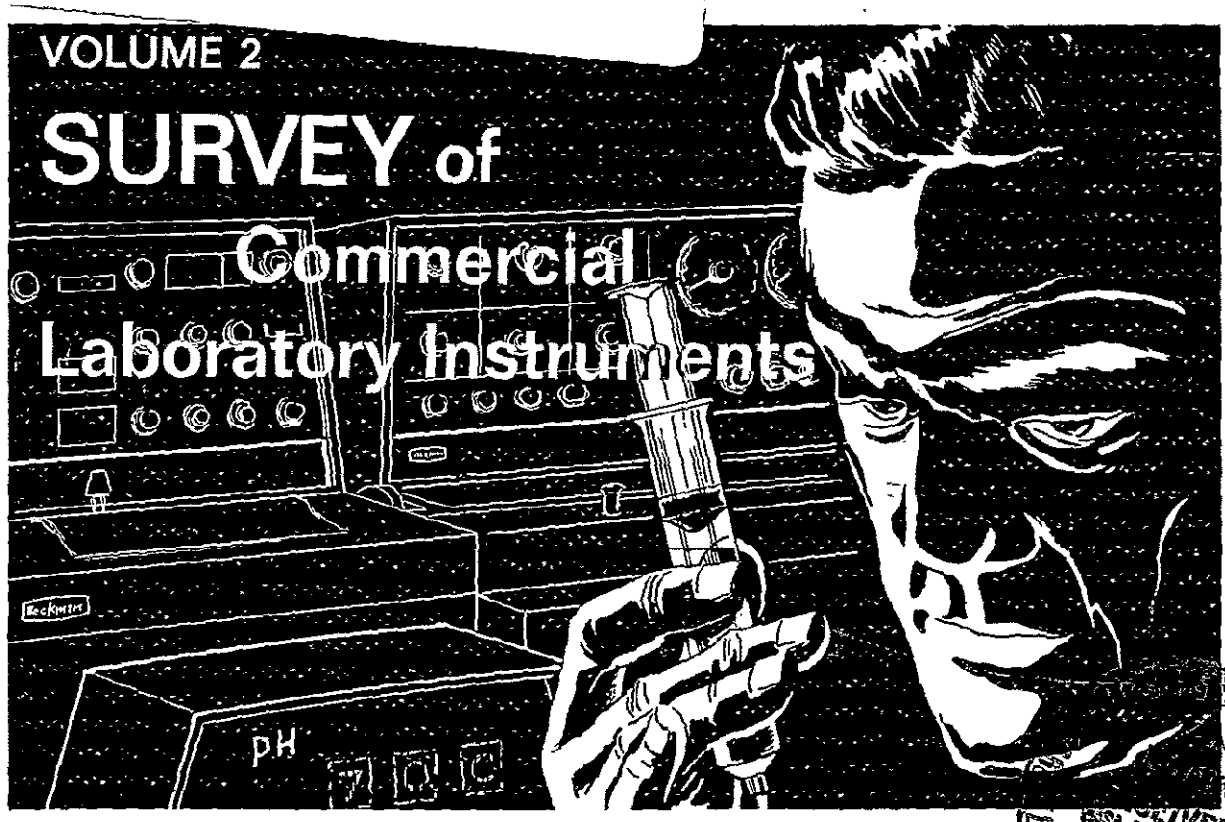
COMMERCIAL INSTRUMENTATION for Space Station Application

October 31, 1970

Contract No. NAS8-26119

VOLUME 2

SURVEY of Commercial Laboratory Instruments



Prepared for:
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
GEORGE C. MARSHALL SPACE FLIGHT CENTER

Beckman® INSTRUMENTS, INC.
ADVANCED TECHNOLOGY OPERATIONS
FULLERTON, CALIFORNIA • 92634

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FR-1065-101

VOLUME 2

SURVEY of Commercial Laboratory Instruments

ALLEN C. NORTON, Ph.D.

October 31, 1970

Contract No. NAS8-26119

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PREFACE

This second volume of the Final Report presents the results of an intensive survey of commercial instrumentation applicable to the Space Station. The Blue Book* was used to determine types of instruments needed for support of the laboratories and experiment program. Twenty-four instrument categories were selected to provide a representative, not necessarily complete, selection of types of instruments needed. The report on each category of instruments is written in approximately the same format: principles of operation, applications, logistics, operation, interface, safety, modifications needed, and available instruments. The survey was written on the basis of actual instruments available at the time of writing (mid-1970). Instruments under development and trends in instrument development are noted where applicable. The final section in this volume deals with the problems of sample handling in a zero-g laboratory, and a few sample-handling devices appropriate for Space Station application.

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*Candidate Experiment Program for Manned Space Stations, "The Blue Book".

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Section 1
AUDIOMETERS

1.1 PRINCIPLES OF OPERATION

The audiometer is a psychosensory testing device for determination of auditory thresholds on cooperative human subjects. A relatively pure tone is produced from an electronic oscillator and is presented to the subject through a set of earphones. The earphones allow presentation of the tone independently to either ear. As the sound intensity is increased and decreased at each frequency, the subject indicates when he can hear the tone. After a threshold is established at one frequency, another sample frequency is tested. An audiogram is thus generated, showing auditory sensitivity as a function of frequency.

Changing of frequency and intensity was usually done manually--the operator recording the response to each stimulus. There are presently some completely automatic instruments in which the intensity and frequency are changed automatically. With the automatic instruments, the subject presses a switch when he hears the tone and releases it when he does not. The responses are recorded and plotted automatically.

1.2 APPLICATIONS

Audiometers have only one application: determination of auditory thresholds. Although the result is generally always threshold measurement, audiometers can be used in a few different ways. For example, audiometers might be used as

auditory stimulation for neurological or behavioral research. Audiometers are applicable to the following functional program elements (FPE's):

5.13 Biomedical and Behavioral Research
5.14 Man/System Interaction

1.3 LOGISTICS

1.3.1 Packing and Launch

Audiometers are generally quite rugged and require no special packaging other than that normally provided for railroad shipment.

1.3.2 Installation

Audiometers need no special installation beyond unpacking, connection to electric power, and suitable tie-down to prevent floating around the laboratory in a zero-g environment.

1.3.3 Consumable Supplies

Manual audiometers use no consumable supplies. Automatic audiometers need recording paper and ink or ribbon for the recorder.

1.3.4 Accessories and Spare Parts

Audiometers require earphones for presentation of the stimuli to the subject's ear. These must be covered with sufficient padding to prevent air coupling of sound to the opposite ear. On-board headsets would not be adequate for this application.

A backup set of earphones and spare electronic parts for the audiometer should be provided.

1.3.5 Maintenance and Repair

A replacement approach is recommended for repair and maintenance of audiometers in the Space Station environment. Occasional calibration of the instrument with a calibrated microphone may be needed.

1.4 OPERATION

1.4.1 Warm-up and Speed-of-Operation

Currently available audiometers are of entirely solid-state circuitry and are available for use immediately upon turning on. Determination of a complete audiogram usually takes approximately 10 to 20 minutes per ear.

1.4.2 Operation Skills

Completely automatic audiometers can be operated with relatively little previous experience. Only slight training specific to the instrument is required for routine operation. For manual instruments, some experience with psychophysical testing techniques is needed. For interpretation of the audiograms and nonroutine use of audiometers, professional experience in audiometry is needed.

1.4.3 Operating Procedure

The manual audiometer requires a subject and an operator. The operator changes the frequency and intensity of the tone and records the responses. The actual sequence of presentation is determined by the operator and his experience in audiometry. A completely automatic audiometer requires only the subject. The record of his responses is made automatically.

1.4.4 Sample Preparation and Handling

Human subjects can be handled according to normal clinical procedures.

1.5 INTERFACE

1.5.1 Interface with Other Laboratory Instruments

In some behavioral or neurological research applications, an audiometer could be used to present an auditory stimulus to a human subject. In this case, audiometers might be required to interface with electrophysiological recording equipment.

1.5.2 Interface with Vehicle Systems

If used in direct off-the-shelf configuration, audiometers require only operating power from the vehicle systems.

Audiometry could be automated by using the on-board data management system to control the stimuli and record the responses. If the latter option is selected, modification of existing instruments might prove more costly than development of an oscillator and attenuator for this application.

1.6 SAFETY

1.6.1 Flame Hazards

The audiometers do not require the use of a flame and are not inflammable.

1.6.2 Microbiological Hazards

Microbial growth is not a problem usually associated with audiometers.

1.6.3 Electromagnetic Interference

No appreciable amount of EMI is generated.

1.6.4 Ionizing Radiation

Ionizing radiation is neither produced by nor interferes with the operation of the audiometer.

1.6.5 Physical Hazards to Personnel

Personnel must be protected from sharp knobs and protruding corners on audiometers.

1.7 MODIFICATION

No major modification, beyond rigid mounting and protection from knobs and corners, is needed for the use of audiometers in a zero-g environment.

Although not recommended, automation of the audiometers by use of the on-board data management system would require a modification to allow interface between computer and audiometer.

1.8 AVAILABLE INSTRUMENTS

Manual audiometers are manufactured by:

Grason Stadler
Lafayette Instrument Company
Tracor, Inc.

Automatic audiometers are manufactured by:

Grason Statler
Tracor, Inc.

Section 2
ATOMIC ABSORPTION SPECTROPHOTOMETER

2.1 PRINCIPLES OF OPERATION

The atomic absorption spectrophotometer measures the absorption of specific wavelengths of light as a light beam passes through a flame into which a sample has been introduced. The absorption of specific wavelengths by the unknown sample is proportional to the concentration of a specific element in the sample. There is a strict specificity between the wavelengths produced by the lamp and the wavelengths absorbed by the unknown sample; a special lamp is used for detection of each element. These lamps require high voltage regulated power supplies. The function of the flame is to identify the sample by raising the atoms to an excitation level at which they will absorb light at characteristic wavelengths. Acetylene is the common fuel used, while air and nitrous oxide are the common oxidant. The hazards of using an open flame in a zero-gravity environment is discussed in Paragraph 2.6.1.

Some atomic absorption spectrophotometers provide for increasing the in-flame length of the optical path by passing it three times through the flame with a mirror system (Figure 2-1). Some models provide a double-beam optical system in which a reference beam does not pass through the flame (Figure 2-2). Analysis of the light beam after it has passed through the flame is achieved by setting the wavelength and slit of a monochromator and reading the output of a photomultiplier. Some models have used standard UV-spectrophotometers as sensors, but current versions employ a monochromator and photomultiplier designed for

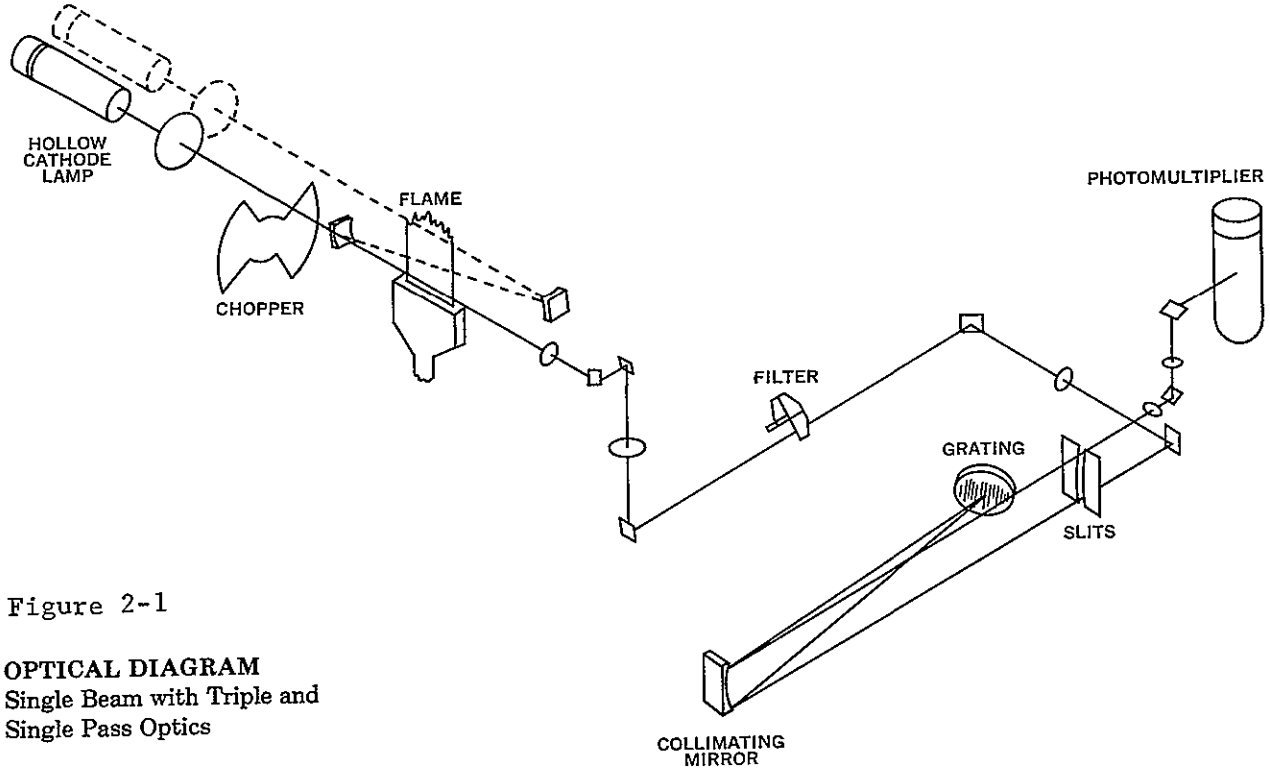

Figure 2-1

OPTICAL DIAGRAM
Single Beam with Triple and Single Pass Optics

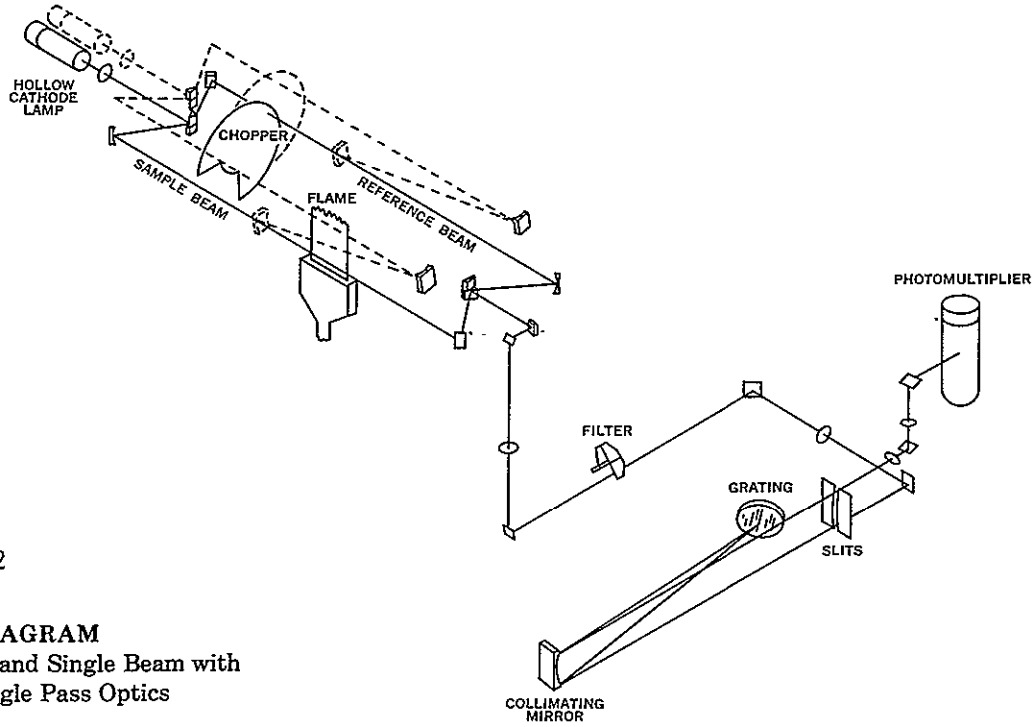

Figure 2-2

OPTICAL DIAGRAM
Double Beam and Single Beam with Triple and Single Pass Optics

this application. The output of the photomultiplier is an analog voltage signal which is read on a meter or chart. On other models the output is digitized.

2.2 APPLICATIONS

Atomic absorption spectrophotometers are applicable to the following functional program elements (FPE's):

- 5.9 Small Vertebrate (Bio D)
- 5.10 Plant Specimens (Bio E)
- 5.13 Biomedical and Behavioral Research
- 5.15 Life Support and Protective Systems
- 5.16 Materials Science and Processing
- 5.18 Exposure Experiments
- 5.23 Primates (Bio A)
- 5.25 Microbiology (Bio C)
- 5.26 Invertebrates (Bio F)
- 5.27 Physics and Chemistry Lab

These instruments could also be used in Bioscience, Biomedical, or Physics and Chemistry Laboratories.

In biomedical research, atomic absorption spectrophotometry can be used for determination of major ionic species as well as trace metals in blood, serum, tissue, urine, and bone ash. For contamination studies, these instruments can detect trace metals in air and water, and also find use in materials science and processing experiments.

Elements detectable at less than 1 ppb (1 ppb = 10^{-3} ppm) include Ca, Cr, Cu, K, Mg, Mn, Na, and Zn. Other elements detectable below 1 ppm include Li, Be,

Al, Sc, Ti, V, Fe, Co, Ni, Ga, Ge, As, Se, Rb, Sr, Y, Mo, Ru, Rh, Pd, Ag, Cd, In, Sn, Sb, Te, Cs, Ba, W, Re, Os, Pt, Au, Hg, Tl, Pb, Bi, Nd, Sm, Eu, Gd, Dy, Ho, Er, Tm, and Yb. Indirect methods are available for nonmetallic elements such as phosphorous, sulfur, and the halogens.

2.3 LOGISTICS

2.3.1 Packing and Launch

Atomic absorption spectrophotometers require no particularly different packing procedures than other precision photo-optical instruments. The hollow cathode lamps, although glass, are rugged and should withstand launch stresses with little precaution other than careful packing in their normal shipping boxes. The lamps are low pressure and would withstand reduced pressure during transport or even rapid decompression.

2.3.2 Installation

Unpacking and installation of atomic absorption spectrophotometers is routine. Mounts are needed to keep the instrument stationary during space operations. During instrument operation the slight thrust of the burner would, if anything, merely tend to press the instrument against the surface of the working table or mount. (Burner thrust, however, is negligible with respect to instrument mass.) The installation requirements are complete with attachment of electric power, operating gases, and flame venting.

2.3.3 Consumable Supplies

The main consumable supplies for atomic absorption spectrophotometers are the flame support gases. Acetylene is the most frequently used fuel; hydrogen is occasionally used. The flame is supported by compressed air and/or nitrous oxide.

2.3.4 Accessories and Spare Parts

The major accessories for atomic absorption spectrophotometers are burner modules and lamps. Different burners are available for different gas combinations and different flame configurations. For zero-gravity operation, a one slit burner giving a relatively "stiff" flame is preferred.

The specific wavelengths of light needed for atomic absorption spectrophotometry are provided by hollow cathode and osram vapor discharge lamps. The wavelengths produced are specific to the material with which the lamp is constructed and, also, specific to the nebulized atomic species being absorbed. A different lamp is needed for each element. Hollow cathode lamps are available for most of the metallic elements. Some of the available single and multiple element hollow cathode lamps are listed in Tables 2-1 and 2-2. Osram vapor discharge lamps are available for cesium, potassium, rubidium, sodium, and thallium. Electrodeless discharge lamps are not recommended for space stations because of the EMI produced by their microwave power supply.

Standard solutions are needed for each element to be analyzed. Three standard concentrations are needed for each concentration range; the standards should encompass the concentration range expected, since extrapolation is not desirable.

Many models of atomic absorption spectrophotometers are adaptable for use as atomic emission spectrophotometers and atomic fluorescence spectrophotometers. Modification kits for these purposes would allow some extension of the flexibility of atomic absorption spectrophotometers.

Symbol	Cathode Material	Window[1]	Gas Fill[2]	Maximum Current mA**	Wave-lengths
Al	Aluminum	P	Ne	20	309.2
Sb	Antimony	Q	Ne	25	217.6
As	Arsenic	Q	Ne	20	193.7
Ba	Barium	P	Ne	25	553.6
Bi	Bismuth	Q	Ne	12	223.1
B	Boron	Q	Ar	20	249.7
Cd	Cadmium	Q	Ne	12	228.8
Ca	Calcium	P	Ne	20	422.7
Ce	Cerium	Q	Ne	20	286.2
Cs	Cesium	P	Ne	25	852.1
Cr	Chromium	Q	Ne	25	357.9
Co	Cobalt	Q	Ne	25	240.7
Cu	Copper	P	Ne	20	324.7
Dy	Dysprosium	Q	Ne	15	421.2
Er	Erbium	Q	Ne	15	400.8
Eu	Europium	Q	Ne	20	459.4
Gd	Gadolinium	Q	Ne	20	368.4
Ga	Gallium	Q	Ne	15	287.4
Ge	Germanium	Q	Ar	20	265.1
Au	Gold	Q	Ne	15	242.8
Hf	Hafnium	Q	Ne	18	307.2
Ho	Holmium	Q	Ne	15	410.4
In	Indium	Q	Ne	20	304.0
Ir	Iridium	Q	Ne	25	285.0
Fe	Iron	Q	Ne	20	248.3
La	Lanthanum	Q	Ne	15	392.8
Pb	Lead	Q	Ne	15	283.3
Li	Lithium	P	Ne	30	670.8
Lu	Lutetium	Q	Ne	20	336.0
Mg	Magnesium	Q	Ne	20	285.2

Note 1: P = Pyrex (>70% transmission at 305 mμ)
Q = Quartz (>75% transmission at 220 mμ)

Note 2: Ar = Argon gas fill
Ne = Neon gas fill

Table 2-1 (1 of 2). Atomic Absorption Supplies--Single-Element Types

Symbol	Cathode Material	Window[1]	Gas Fill[2]	Maximum Current mA**	Wave-lengths
Mn	Manganese	Q	Ne	25	279.5
Hg	Mercury	Q	Ar	15	253.7
Mo	Molybdenum	Q	Ne	30	313.3
Nd	Neodymium	Q	Ne	20	462.4
Ni	Nickel	Q	Ne	20	232.0
Nb	Niobium (Columbium)	Q	Ne	25	334.9
Os	*Osmium	Q	Ar	20	
Pd	Palladium	Q	Ne	25	247.6
P	*Phosphorus	Q	Ne	20	
Pt	Platinum	Q	Ar	25	265.9
Pr	Praseodymium	Q	Ne	20	295.1
Re	Rhenium	Q	Ne	25	346.1
Rh	Rhodium	Q	Ne	25	343.5
Rb	Rubidium	P	Ne	30	780.0
Ru	Ruthenium	Q	Ar	25	334.9
Sm	Samarium	Q	Ne	20	429.7
Sc	Scandium	Q	Ne	20	391.2
Se	Selenium	Q	Ne	15	196.0
Si	Silicon	Q	Ne	20	251.6
Ag	Silver	Q	Ar	20	328.1
Na	Sodium	P	Ar	25	589.0
Sr	Strontium	P	Ne	25	460.7
S	*Sulfur	Q	Ne	20	
Ta	Tantalum	Q	Ne	30	271.4
Te	Tellurium	Q	Ne	15	214.3
Tb	Terbium	Q	Ne	25	432.7
Th	Thorium	Q	Ne	20	324.5
Tm	Thulium	Q	Ne	20	409.4
Sn	Tin	Q	Ne	15	286.3
Ti	Titanium	Q	Ne	25	364.3

*At the time of publication, there are no known procedures for determination of this element by atomic absorption.

**The current rating shown is the maximum permissible figure and does not represent typical operation. For optimum performance and life, a tube should be operated at the lowest current consistent with the desired output characteristics.

Table 2-1 (2 of 2). Atomic Absorption Supplies--Single-Element Types

Cathode Material	Window[1]	Gas Fill[2]	Maximum Current mA**
As-Ni	Q	Ne	20
As-Sb-Bi	Q	Ne	15
As-Se-Te	Q	Ne	15
Au-Cu-Fe-Ni	Q	Ne	20
Au-Ni	Q	Ne	15
Ba-Ca-Sr	P	Ne	25
Ba-Ca-Sr-Mg	Q	Ne	25
Ca-Mg-Al	Q	Ne	20
Ca-Mg-Al-Li	Q	Ne	18
Ca-Zn	Q	Ne	15
Cr-Co-Cu-Fe-Mn-Ni	Q	Ne	30
Cr-Co-Ni	Q	Ne	25
Cr-Cu	Q	Ne	20
Cr-Fe-Mn-Ni	Q	Ne	30
Cu-Co	Q	Ne	20
Cu-Ga	Q	Ne	15
Cu-Mn	Q	Ne	20
Cu-Pb-Zn-Ag	Q	Ne	15
Cu-Zn-Fe-Mn	Q	Ne	15
Cu-Zn-Mo	Q	Ne	15
Cu-Zn-Mo-Co	Q	Ne	15
Cu-Zn-Pb-Cd	Q	Ne	12
Cu-Zn-Pb-Sn	Q	Ne	12
Fe-Cu-Mn	Q	Ne	20
Fe-Cu-Ni-Pb-Zn	Q	Ne	15
In-P	Q	Ne	20
Mo-Cu-Fe	Q	Ne	25
Pb-Zn-Ag	Q	Ne	12
Se-Ni	Q	Ne	15
Zn-Ag-Pb-Cd	Q	Ne	12

Table 2-2 (1 of 2). Atomic Absorption Supplies--Multiple-Element Types

Symbol	Cathode Material	Window[1]	Gas Fill[2]	Maximum Current mA**	Wave-lengths
W	Tungsten	Q	Ne	30	294.4
U	Uranium	Q	Ne	25	424.4
V	Vanadium	Q	Ne	30	318.4
Yb	Ytterbium	Q	Ar	20	398.8
Yt	Yttrium	Q	Ne	15	410.2
Zn	Zinc	Q	Ne	18	213.9
Zr	Zirconium	Q	Ne	25	360.1

**The current rating shown is the maximum permissible figure and does not represent typical operation. For optimum performance and life, a tube should be operated at the lowest current consistent with the desired output characteristics.

Note 1: P = Pyrex (>70% transmission at 305 mμ)
Q = Quartz (>75% transmission at 220 mμ)

Note 2: Ar = Argon gas fill
Ne = Neon gas fill

Table 2-2 (2 of 2). Atomic Absorption Supplies--Multiple-Element Types

2.3.5 Maintenance and Repair

The electronic and optical components of atomic absorption spectrophotometers do not present any particular problems beyond the normal maintenance of electronic and optical equipment. The electronics portions include the photomultiplier with its amplifier and readout and the power supply for the lamps. The optical portions of the instruments include the monochromator and the light path through the flame.

The gas flow and flame systems are unique to atomic absorption spectrophotometers. They are, however, clean burning and relatively maintenance free.

2.4 OPERATION

2.4.1 Warm-up and Speed-of-Operation

The hollow cathode lamps require about 20 minutes warm-up before operation. The multiple power supplies of some instruments provide for stand-by lamps (for analysis of different elements) to be warming up while one lamp is in use. Adjustment of gas pressure and flame position as well as selection of wavelength and slit width can be made while the lamp is warming up. The warm-up of the photomultiplier and amplifier is easily accomplished within the warm-up time for the lamps. The flame assumes its proper characteristics almost immediately upon lighting; it would be lit and extinguished for each sample analyzed.

When operating, the flame can be lit and the sample analyzed within seconds. Changing samples, even within the constraints of the wet chemistry packets, should not take more than one or two minutes. Changing the lamps to measure different elements would take no more than a few minutes if the lamps must be

separately mounted and unmounted, or a few seconds if the lamps are on a turret. Measurement of the same element in different samples could be made at the rate of several tens of samples per hour, while the rate for different elements would be several per hour.

2.4.2 Operation Skills

Space Station operation of the atomic absorption spectrophotometer is possible for either professional or technical level personnel in any scientific discipline requiring use of the instrument. Anyone with Earth-based experience with an atomic absorption spectrophotometer can operate one in a space station; retraining would be negligible. Pre-flight training of inexperienced personnel can be accomplished in a few hours for routine measurement; even in-flight training is feasible. For other than routine measurements, preparation of samples would require specialized training and experience.

2.4.3 Operating Procedure

Typical operating procedure for an atomic absorption spectrophotometer would be as follows:

Preparation:	Lamp warm-up. Flame position adjustment. Monochromator adjustment.
Calibration:	Light flame (automatic lighter). Introduce standard. Record. Flush with blank. Repeat for one or two other standard concentrations.
Measure:	Light flame. Introduce sample. Record. Flush with blank.

This procedure gives concentration of one element. To test for other elements the lamp must be changed (not necessary if multiple element lamp is used) and the monochromator readjusted (wavelength and slit). The calibration and measurement procedures must then be repeated.

The flame should be extinguished except when measurements are being made to avoid unnecessary stress on the EC/LS (Environmental Control/Life Support Systems).

2.4.4 Sample Preparation and Handling

Samples are introduced into the atomic absorption spectrophotometer by aspiration from a liquid sample. The negative pressure for aspiration is provided by the velocity of the gases in the flame. Liquid samples will be subject to the constraints of other wet chemistry operations and must be introduced through a closed system.

Although several versions of automated sample handling devices are available for atomic absorption spectrophotometers, automated sample handling is not recommended for space station instrumentation. Automated sample changing devices require gravity for their operation, and need for repetitive routine measurements is not expected in the space station experiments.

2.5 INTERFACE

2.5.1 Interface with Other Laboratory Instruments

The atomic absorption spectrophotometer requires input of the sample to be analyzed. The sample, normally in a liquid medium, is introduced into the burner by aspiration from an appropriate wet chemistry container. The output

of the instrument is a photomultiplier amplifier output voltage signal which is proportional to absorption, or concentration of the element being analyzed. The output may be read directly from the meter or applied to the input of a recorder.

2.5.2 Interface with Vehicle Systems

The atomic absorption spectrophotometer will require fuel, oxidants, and electric power from the vehicle systems. The commonly used fuels are acetylene and nitrous oxide. The gases must be supplied under pressure; they are mixed within the instrument. The output of the instrument can be available for input into the vehicle data management system.

Atomic absorption spectrophotometers are generally designed to operate on 115 volts, 60 Hz electric power. Changes from 60 to 400 Hz operation would alter the performance of the chopper motor in the optical system, the gas solenoids in the burner, the spark-type flame lighter, and the motor speed of the strip-chart recorder. Modifications would be needed to counteract each of these changes. In some cases, 400 Hz transformers would be needed in the power supplies. With other types of modifications (Paragraph 2.7), these instruments could be operated from 28-volt dc power sources by use of dc-to-dc converters and other internal modifications.

Venting must be provided for the flame of an atomic absorption spectrophotometer. Maximum safety would be provided by complete separation of the flame and its vent from the cabin environment. Some instrument modification is needed to achieve this. A vacuum line attached to the vaporization chamber of the burner is required to allow evacuation of sample between determinations (Paragraph 2.7).

2.6 SAFETY

2.6.1 Flame Hazards

The presence of an open flame is the major safety hazard of the atomic absorption spectrophotometer. Flameless methods for sample atomization using a plasma torch or plasma flame present even greater safety hazards. With proper venting of the flame and isolation of the flame from the cabin environment, adequate safety can be maintained to allow use of the atomic absorption spectrophotometer in the Space Station. Some care is needed to maintain gas pressures if a nitrous oxide flame is used.

2.6.2 Microbiological Hazards

The atomic absorption spectrophotometer presents no microbiological hazards. Any microorganisms present in the sample would be destroyed in the flame.

2.6.3 Electromagnetic Interference

The atomic absorption spectrophotometer represents the following possible sources of interference:

- Dc-to-dc converter to convert to 28-V dc operation (if required)
- Flame igniter
- Solenoid valves for gases
- Microwave light tubes
- Logic in control or readout circuitry

Sufficient shielding and line filtering will limit radiated and conducted interference to acceptable levels. The flame igniter, typically a spark generator, may be difficult to control even with a moderate amount of shielding

and filtering. However, this is only a single event transient occurring infrequently and, therefore, should present no serious interference problem.

The standard atomic absorption spectrophotometer uses light tubes which are excited by a microwave source. For this reason, atomic absorption spectrophotometers using microwave light tubes are not recommended for space applications.

The detectors represent high-impedance amplifiers and signal conditioners. These circuits may exhibit susceptibility to RF and transient energy without shielding and filtering. The filtering and shielding provided for interference control should suffice.

2.6.4 Ionizing Radiation

Ionizing radiation would be produced by an atomic absorption spectrophotometer only to the extent that radioisotopes are present in the samples analyzed. Proper venting of the flame would remove these radioactive particles from the cabin environment, leaving their ultimate disposal to the venting system.

2.6.5 Physical Hazards to Personnel

Sharp corners and protruding knobs present on most models of atomic absorption spectrophotometers present some physical hazards to personnel. These hazards can be reduced by techniques for mounting the instrument and modification of the front panel controls. Separation of the flame from the cabin should include the thermal insulation necessary to avoid burns.

2.7 MODIFICATIONS

The following modifications are needed for adapting an atomic absorption spectrophotometer for Space Station use.

1. The flame should be vented and separated from the space station cabin environment.

2. The instrument should be mounted to be stable on the bench and to avoid personnel injury from accidental encounter with edges, corners, or protrusions of the instrument.

3. In Earth-based use, the sample vaporization chamber of the burner drains to prevent accumulation of fluids in the chamber. In a zero-gravity environment this draining would not occur. The more efficient drawing of the sample into the flame in the absence of gravity would probably make this unnecessary. However, until this point is demonstrated in flight, this drain should be connected to a vacuum source to allow evacuation of the vaporization chamber if necessary.

4. On some models, clamps will be needed to hold the active, but unused, lamps in place.

2.8 AVAILABLE INSTRUMENTS

There are several manufacturers of atomic absorption spectrophotometers, and each makes several different models. The instrument within each manufacturer's line differs in sophistication of design, versatility, and degree of automation. The major instruments following were selected as those of greatest versatility

and sophistication of design, but the least automated. Automation of atomic absorption spectrophotometers typically involves automated handling of multiple samples and facilitation of analysis of some preselected elements. The automated sample handling techniques are not applicable in a zero-gravity environment, and automated operation decreases versatility. The major instruments available for use in a space station environment are the following:

Company	Model No.
Aztec Instruments, Inc.	AAA-3
Bausch & Lomb, Inc.	AC2-20
Beckman Instruments, Inc.	444
Instrumentation Lab, Inc.	153
Jarrell-Ash	82-500
Perkin-Elmer Corp.	303
Varian Techtron	AA5
Carl Zeiss, Inc.	PMQ II w/FA2-2AA

Specifications of these instruments are listed in Table 2-3. Atomic absorption spectrophotometers are also manufactured by:

Bendix Corporation
Coleman Instruments
Engris Equipment Company
Heath Company
National Instrument Labs
Phillips Electronic Instruments
Process & Instrument Corporation
Pye Instruments Limited

COMPANY	MODEL	PRICE	OPERATION MODE	OPTICS	WAVELENGTH RANGE (mμ)	RESOLUTION (Angstrom)	LAMP	BURNER SYSTEM	SCALE EXPANSION	READOUT
Aztec Instruments, Inc.	AAA-3	$7,450	AA,FE,AF	SB	185 to 900	0.2	HC, mod, VD	P, LF	1,5,10,20X	Meter, digital & recorder opt
Bausch & Lomb, Inc.	AC2-20	$6,200	AA,FE	SB	190 to 800	NG	NG	VA,P,LF	NG	Meter
Beckman Instruments, Inc.	444	$5,600	AA,FE	SB,DB	190 to 852	To 0.2 nm	HC	LF,TF,P	NA	Digital
Instrumentation Lab, Inc.	153	$8,650	AA	Dual DB	180 to 1,000	0.6	HC, Double	VA,AF,P, TC	50X	Digital
Jarrell-Ash	82-500	$6,450 & up	AA,FE	SB	190 to 870	0.2	HC, mod	TC,LF	1 to 10X	Meter
Perkin-Elmer Corp.	303	$6,560	AA	DB	170 to 900	0.3	HC,VD unmod	P	100X	Meter
Varian Techtron	AA-5	$7,190	AA,FE,AF	SB	186 to 1,000	.33A	HC	VA,P	10X	Meter, digital & recorder opt
Carl Zeiss, Inc.	PMQ II w/FA2-2AA	$7,620	AA,FE	SB or DB	185 to 2,500	UV, 0.1 Vis, 0.3	HC,W dc	P	Numerous Sensitivity Ranges	Direct

NOTES:
AA - Atomic Absorption
AF - Atomic Fluorescence
FE - Flame Emission
HC - Hollow Cathode
VD - Vapor Discharge
W - Tungsten
SB - Single Beam
DB - Double Beam
P - Premix
LF - Laminar Flow
TF - Turbulent Flow
VA - Variable Aspirator
TC - Total Consumption

Table 2-3. Atomic Absorption Spectrophotometers

Section 3
BLOOD GAS ANALYZERS

3.1 PRINCIPLES OF OPERATION

3.1.1 Oxygen Analyzers

The measurement of the partial pressure of oxygen is of great value in the study of pulmonary diffusion problems, arteriovenous oxygen content differences, and in other physiologic studies.

The basic operation of oxygen sensors for blood is identical to those used for gas-phase analyses. Essentially all contemporary sensors are based upon the basic membrane-covered polarographic sensor* developed by Dr. L. A. Clark. As described in Section 18, the electrode causes a current to flow by reducing oxygen at the cathode. A gas-permeable membrane (typically a thin sheet of Teflon) shields the electrodes from contamination by the sample, and retains a gel which serves as the conducting medium between the anode and the cathode.

Although the theory of operation is identical for gaseous and dissolved oxygen sensors, their practical aspects of sample presentation differ considerably. Since oxygen is consumed during the measurement process, a fresh sample must be continuously supplied. When measuring oxygen in a gas mixture, diffusion processes are sufficiently rapid to avoid localized depletion. With liquids,

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*U.S. Patent No. 2,913,386 held by Beckman Instruments, Inc.

however, the sample should be flowing or the cathode area reduced to preclude measurable oxygen depletion during the measurement process.

The typical current flow in an electrode designed for use in blood is approximately $1x10^{-11}$ amperes. Because of this small current, there is a requirement for a sensitive and stable high-impedance and high-gain amplifier.

Oxygen measurements must be made at a constant temperature, preferably at 37°C. Control of this temperature is extremely important because of temperature effects on the solubility of oxygen at any given P_{O_2}* and the diffusion rate of the gas across the semipermeable membrane.

The electrode is typically calibrated with two gases, one of which contains no oxygen and the second of which contains an elevated value. The percentage value obtained from the calibrating gases are translated into partial pressure dimensions on the basis of atmospheric pressure and temperature.

The major problem associated with the measurement of P_{O_2} in space is that of weightlessness. Because currently available systems rely upon gravity to preclude entrapment of air bubbles in the sample, they are unappropriate for use in zero-gravity. A further discussion of fluid handling for blood gas measurements will be found in Section 25.

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* Partial Pressure of Oxygen (P_{O_2})

3.1.2 Dissolved Carbon Dioxide Analyzers

Two methods are commonly employed for the analysis of the partial pressure of carbon dioxide in the blood. The first of these is a direct method, utilizing a carbon dioxide sensing electrode, and the second is an indirect Astrup technique.

3.1.2.1 Direct Measurement

The P_{CO_2}* electrode is a modified pH electrode. A pH-sensitive bulb is combined with a reference electrode in the same electrode body. A thin film of a buffer or gel containing bicarbonate is applied to the sensing surface of the electrode and held there with a very thin semipermeable membrane. The membrane generally is silicon rubber, which is permeable to carbon dioxide gas but impermeable to hydrogen ions.

When this assembly is exposed to blood, carbon dioxide from the blood diffuses across the membrane and reacts with the bicarbonate film on the other side. The pH of the bicarbonate solution will change with changes in carbonic acid concentration and thus with changes in the P_{CO_2} of the sample. This pH is measured with a stable glass electrode and the pH is connected to P_{CO_2} through the use of a linear calibration curve relating log P_{CO_2} to pH.

Calibration is achieved through the use of gases of known carbon dioxide concentration. The gases can be combined with those used for the calibration of the oxygen electrode so that two gas reservoirs would suffice for both systems.

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* Partial Pressure of carbon dioxide (P_{CO_2})

As with oxygen, temperature control of the measurement system and the specimen being analyzed is extremely important. The temperature coefficient of the measurement is approximately 3%/1°C, and long temperature equilibration time would be manifested in inaccurate or drifting readings.

3.1.2.2 Indirect Measurement

The indirect measurement of P_{CO_2} is performed by measuring the pH of three aliquots of the specimen of interest. Two of the aliquots must be equilibrated with known concentrations of carbon dioxide gas. The three values are related by the use of a nomogram. Because of the technical difficulties involved with the use of tonometers in zero-gravity, it is not felt that the indirect method would be practical in space.

3.1.2.3 Containment of Liquid Sample

The major functional problem in the measurement of blood gases in space will be the containment of liquid samples throughout the measurement and disposal process, coupled with a requirement that no air bubbles can be entrained in the specimen stream. A configuration to permit this can be designed and a substantial amount of effort has already been expended in this direction.

An additional goal would be the simplification of the maintenance ordinarily associated with the measuring instrumentation, e.g., membrane replacement, cleaning, and calibration.

3.2 APPLICATIONS

Blood gas analyzers are used for making dissolved oxygen and carbon dioxide determinations in blood and other biological fluids, and are directly applicable to the following functional program elements (FPE's):

5.9 Small Vertebrates (Bio D)
5.13 Biomedical and Behavioral Research
5.25 Microbiology (Bio C)
5.23 Primates (Bio A)
5.26 Invertebrates (Bio F)

3.3 LOGISTICS

3.3.1 Packing and Launch

Reasonable care should be taken in providing a sturdy support for the electrodes. They can be transported either in individual packing or mounted in a flow-cell assembly.

3.3.2 Installation

The electrodes should be mounted in a flow cell equipped with a suitable reservoir for waste liquids. The calibration gas bottles should be firmly mounted.

3.3.3 Consumable Supplies

Membranes used in conjunction with the electrodes must be replaced periodically. This time will vary between one week and one month, depending on use. Approximately 2 ml of saline should be used to rinse and soak each electrode after use. Approximately 5 ml buffered solution and 20 ml calibration gas will be required for both electrodes during a calibration and analysis cycle.

3.3.4 Accessories and Spare Parts

Both electrodes should be duplicated with spare sets for backup and replacement.

3.3.5 Maintenance and Repair

After a month's use, both electrodes should be refurbished. This requires membrane replacement and electrolyte gel replacement. Complete electrode replacement might be preferable. Procedures must be developed to make these repairs in a zero-g environment.

3.4 OPERATION

3.4.1 Warm-up and Speed-of-Operation

Before use, the electrode flow chamber must be brought to the proper operating temperature. The temperature must be closely controlled (±0.1°C) at 37°C. This warm-up is expected to require 30 minutes. Calibration will require 30 minutes for both electrodes. Samples can then be analyzed at the rate of one per 3 to 5 minutes.

3.4.2 Operating Skills

Although operation is possible by technical-level personnel, considerable experience with electrochemical methods is required for calibration and maintenance of these devices.

3.4.3 Operating Procedure

Typical operation for electrodes enclosed in a block would be as follows:

Preparation:	Oven warm-up
Calibration:	Open gas bottle No. 1
	Adjust gain to correct setting
	Close gas bottle No. 1
	Open gas bottle No. 2
	Adjust zero offset
	Close gas bottle No. 2
	Open gas bottle No. 1
	Readjust gain setting if required
	Close gas bottle No. 1
Measurement:	Inject sample
	Record after 2 minutes
	Flush with saline

3.4.4 Sample Preparation and Handling

The addition of anti-coagulants is all that is required for sample preparation. The analysis should be performed within several minutes after the sample is collected, or a means must be provided for storage at a low temperature until analysis.

Calibration is normally achieved by bubbling a standard gas through the calibrating liquid to establish an equilibrium at a known partial pressure. Since bubbling is inconsistent with zero-g sample handling, an alternate procedure must be established. This might be solved by direct-gas calibration; however, moving gases and liquids alternately through a chamber is itself a difficult problem for zero-g operation. Calibration with a gas standard requires longer for equilibrium to be established than with liquid calibration.

3.5 INTERFACE

No critical interface problems will be experienced. Calibration procedures will be simplified if an on-board computer is available. Practically any high-input impedance ($>10^{11}$ Ω) amplifier system can be used to monitor these electrodes. These can be operated from a low-voltage dc supply or from a power supply using 110 V ac.

3.6 SAFETY

3.6.1 Flame Hazards

An internal flame is not required for operation of these sensors, and the sensors are not inflammable.

3.6.2 Microbiological Hazards

Possible microbial growth in the waste disposal system used in conjunction with the blood gas electrodes can be effectively controlled with bactericidal agents.

3.6.3 Electromagnetic Interference

The only probable source of interference is from the power supplies, especially from dc to dc converter types operated from 28 V dc. LC filtering and feed-through bypass filtering and a small amount of shielding will effectively limit interference conditions or radiation to within acceptable limits. The extremely high impedance of most electrodes results in circuitry which can be very susceptible to radiated RF energy. The electrodes should be as close as physically possible to the electronics, and cables should be double-shielded to avoid

undesirable pickup. Design of the ground system is also critical. Extra shielding of the electronics may be necessary to provide complete and sufficient protection from radiated energy. The filtering used to control conducted interference will be equally effective in limiting circuit susceptibility to conducted RF and transient energy to within acceptable limits.

3.6.4 Ionizing Radiation

Ionizing radiation is neither produced by nor interferes with the operation of these analyzers.

3.6.5 Physical Hazards to Personnel

Reasonable precautions should be taken to prevent breakage of the P_{CO_2} electrode when modification or service work is performed.

3.7 MODIFICATIONS

1. The simplicity and low cost of the electronic components make it advisable to replace them with space-qualified components.
2. Fluid-filled electrodes must be equipped with an absorbant material to retain the fluids at the sensing surface.
3. A flow-through system for zero gravity must be designed.
4. The glass pH electrode used in the carbon-dioxide sensor should be shielded.
5. A zero-gravity calibration system must be developed.

3.8 AVAILABLE INSTRUMENTS

The high-impedance amplifier may be obtainable from any reputable electronic supplier. Flow-through cells such as required for zero-gravity applications are not available. The major manufacturers are listed in Table 3-1. Although no suitable commercial instruments are available in 1970, development in this field is actively continuing for both clinical and space application, and improved instruments may be available when needed.

Oxygen	Carbon Dioxide
Beckman Instruments, Inc.	Beckman Instruments, Inc.
Instrumentation Lab, Inc.	Instrumentation Lab, Inc.
Yellow Springs Instrument Co.	London Co.
Corning Glass Works	National Welding Co.
London Co.	Electronic Instruments, Ltd.

Table 3-1. Commercially Available Blood Gas Electrodes

Section 4
CELL COUNTERS

4.1 PRINCIPLES OF OPERATION

4.1.1 Blood Cell Counter

Any counter capable of detecting particles larger than 4 microns in diameter may be used to count blood cells. With any counter, it is possible to perform minor modifications to convert a multipurpose instrument into a single-purpose instrument, or vice-versa. In this context, single-purpose instruments are defined as accepting only one sample size, one flow rate, one set of electronic threshold values; and provide a direct count that is equitable to number of blood cells per unit volume. Both types of instruments are commercially available, with the single-purpose types being easier to operate but less versatile. Contemporary instruments use either an impedance or optical principle of operation.

The impedance type instrument was developed by the Coulter Co. The sample is pulled through a small (typically 100 μ) orifice by means of the suction created by a mercury column. Platinum electrodes are placed in the sample beaker and the inner tube. Since the small glass orifice creates over 99 percent of the electrical resistance between the two electrodes, any impedance change in the orifice will create a comparable change in the total impedance. In practice, samples are prepared in physiological saline solutions and the voltage drop across the electrodes is monitored while a constant current is permitted to flow. By using an ac-coupled amplifier, steady-state conditions will result in

zero signal. As a particle moves through the orifice, a momentary increase in impedance occurs, thereby creating a pulse. The height of this pulse is proportional to the cubic volume of the cell. Pulse height also is affected by current flow, sample velocity, and orifice diameter, but these parameters are maintained as constants. Higher current flows provide a better signal-to-noise ratio, but excess current will literally "fry" the cells, resulting in orifice blockage. Small diameter orifices also yield a better signal-to-noise ratio, but are more prone to clogging. An electronic threshold device usually is used to prevent signals from small debris particles from interfering with the larger signals provided by blood cells. Total counts may be registered on a digital counter or printer.

Optical instruments usually view a glass chamber through which the sample flows. An incident light source creates a light pulse each time a blood cell passes the viewing area. A photomultiplier tube is used to monitor these light pulses. The signal amplitude for each cell is proportional to the area of the cell. The counting, amplification, and thresholding circuitry is essentially identical to that used for impedance counters. Best signal-to-noise ratio is obtained with the greatest light intensity. During operation, light intensity, flow velocity, detector sensitivity, and focus must be precisely maintained. Since these parameters are relatively difficult to maintain, various elaborate referencing techniques using secondary photosensors have been devised. These systems tend to be bothered by dirt and clogging of the flow cell more so than impedance systems.

The Fisher Autocytometer II (Figure 4-1) is a typical example of a light-scattering type blood-cell counter. The lamp SL is focused on the sample cell as well as a reference photo cell. The reference photo cell, working in conjunction with the lamp source control, provides constant illumination. A dark field optical system provides the PM tube with scattered light signals as the sample is pulled through the sample cell by pump P. A light-emitting diode standardizes the PM tube. The signal is amplified by the Photomultiplier Amplifier, and signals exceeding a preset value as determined by the pulse discriminator are transformed to square-wave pulses and registered on the digital counter.

In both the impedance and optical techniques, blood cells are diluted approximately 200 to 1, with physiological saline solution (Dilution 1). A small aliquot of Dilution 1 is then diluted 200 to 1 again to provide a 40,000 to one dilution factor (Dilution 2). A known volume of Dilution 2 is then counted by the instrument and suitable airthmetic factors are applied to obtain the total red blood cell count. This high degree of dilution is necessary to prevent excessive coincidence For white blood cells, Dilution 1 is treated with one drop of hemolyzing solution such as saponin to hemolyze the red blood cells. A known volume of this sample is then counted to obtain a white blood cell count.

4.1.2 Millipore and Quantimet--Vidicon Computer Systems

The Millipore and Quantimet are Vidicon computer systems that automatically count cells in a microscopic field. These instruments are very large and costly. All the slide preparation steps and microscopic focusing must be

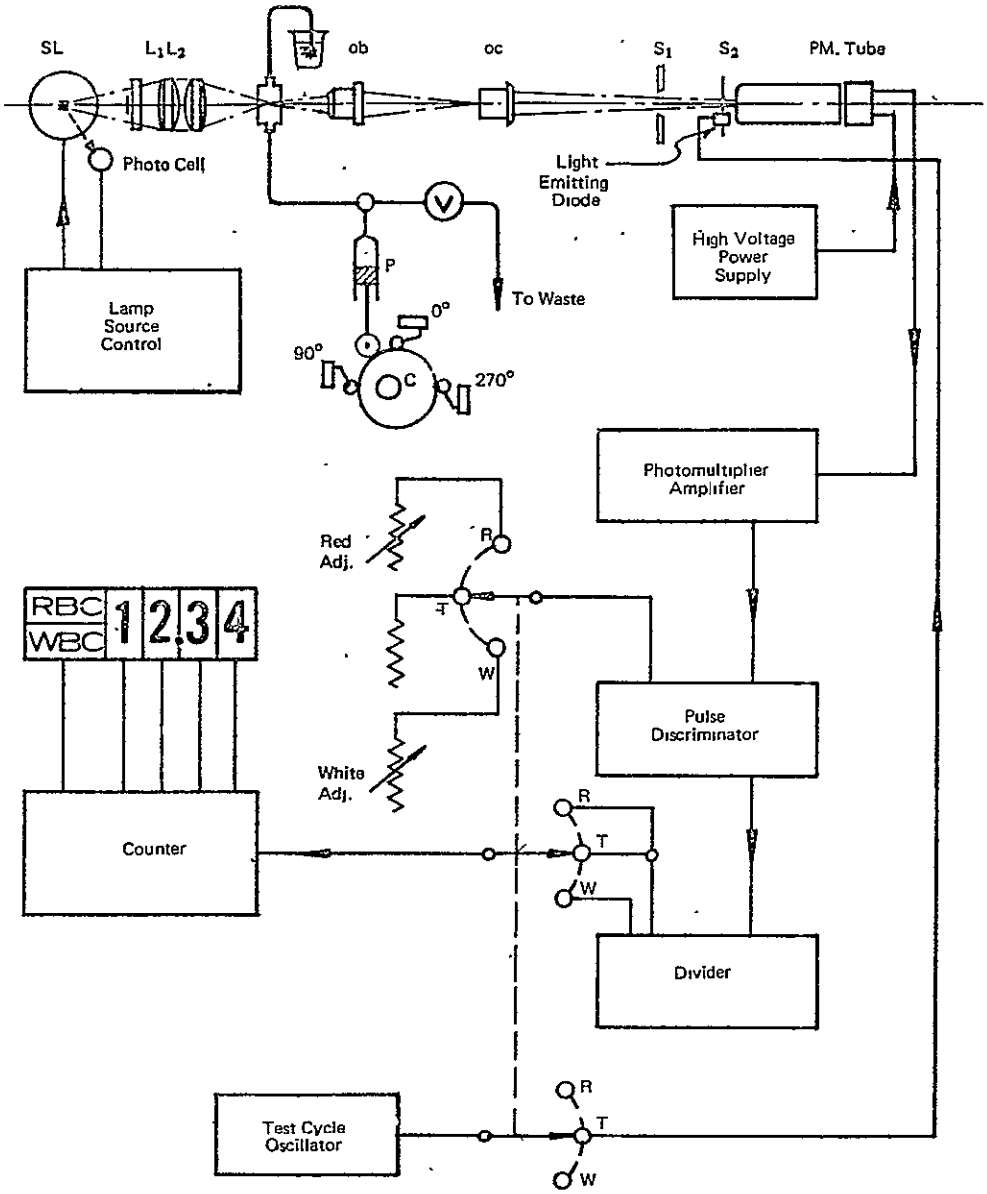


Figure 4-1. Functional Diagram

performed. Since microscopic examination of slides is not an accurate counting technique, the addition of complex counting equipment is not recommended for this application.

4.1.3 Other Counters

Other types of counters for biological purposes are primarily for microbial work. Although blood-cell counting principles can and are being applied to bacterial counting, these methods are nonspecific and also would count inorganic particles. More important, bacteria provide less than 1 percent as much signal as do blood cells. Consequently, modifications such as smaller orifices must be used to improve the signal-to-noise ratio. This leads to excessive orifice clogging. In practice, bacteria counting by these systems is very marginal and can only be performed by highly skilled personnel.

DuPont has a novel bacteria monitor that operates by measuring the light produced when a sample is mixed with reagents. The reagents consist of luciferin-luciferase and buffer. Bacteria supply the necessary adenosine tri-phosphate for the reaction. Like most other tests, the sample-handling procedures must be modified for weightlessness. The reagents for this technique must be frozen until used. It should be noted that this technique can be used with a liquid scintillation counter

The time-honored technique of agar plate culturing is reliable and simple. The resultant colonies are much easier to count when back-illumination is used. A new marking pen counter simplifies counting by electrically registering each

time the pen is touched to the plate. An ink mark on the plates aids in preventing redundant counts.

A more automatic agar colony counter is marketed by American Instrument Co. In this system, the number of imperfections in a tube containing agar and sample is counted automatically. After incubation, another count is made and the resulting difference is attributed to the formation of bacterial colonies.

4.2 through 4.8

Because of the great diversity of types and applications, these instruments have been compared in tabular form in Table 4-1.

MANUFACTURER	BASIC OPERATIONAL DESCRIPTION	MODIFICATION REQUIRED	CONSUMABLE SUPPLIES	OPERATION	INTERFACE	PRICE
Coulter Electronics Model B Counter	Versatile impedance-type blood cell counter.	Vacuum tubes, mercury lines, and gravity-dependent sample feed must be eliminated.	Saponin--1 drop/test Saline--6 ml/test	Elementary procedures but must be redesigned for weightlessness	Severe launch problems, no hazards	$ 5,500.
Particle Data Service, Inc. Blood Cell Counter	Versatile impedance-type blood cell counter.	Sample feed must be modified for weightlessness.	Saponin--1 drop/test Saline--6 ml/test	Elementary procedures but must be redesigned for weightlessness	No problems	2,500.
Fisher Scientific Co. Autocytometer II	Light-scattering blood cell counter.	PMT lamps must be shielded. Sample feed must be modified for weightlessness.	Diluting fluids (2)--15 ml/minute. Reagents (3)--7 ml/minute	Elementary procedures but must be redesigned for weightlessness	PMT and light bulbs must be protected	3,800.
Technicon SMA 4A	Automatic (60 samples/hour) light-scattering blood cell counter, hematocrit, and hemoglobin.	Extensive network of tubing, valves, and pumps required to provide automation--not recommended for this application.	Diluting fluids (2)--15 ml/minute. Reagents (3)--7 ml/minute.	Completely automatic except for sample introduction	PMT and light bulbs must be	Not recommended
Royco Instruments, Inc. Model 341	Light scattering, versatile counter for particles over 5 microns.	PMT lamps must be shielded. Blood dilution technique must be developed.	Saponin--1 drop/test Saline--6 ml/test	Elementary dilution for blood cells	PMT and light bulbs must be protected	8,000 - 12,000
Millipore TT MC	Television-computer system for counting particles per unit area.	None	Depends on sample-handling technique	Manual sample preparation followed by focusing. The instrument then counts the cells in a field	Depends on sample-handling techniques	25,000
Metals Research Quantimet	Television-computer system for counting particles per unit area.	None	Depends on sample-handling technique	Manual sample preparation followed by focusing. The instrument then counts the cells in a field.	Depends on sample-handling techniques	25,000
DuPont	Determines ATP by luminescent firefly reaction. This is related to number of bacteria or blood cells.	Sample handling must be redesigned for weightlessness.	25 assays possible with one enzyme vial, one buffer tablet, and 3 ml water.	Cellular samples are sonicated and then added to mixture of reagents in instrument. Light response is recorded.	Sonicator and freezer must be available.	5,000
American Optical Illuminator	Illuminates and magnifies colonies on petri dishes.	None	None	Place agar plate on illuminator and count colonies	Light bulb should be shielded	115.
American Optical Electronic Register	Tallies colony counts and makes colonies.	None	One membrane per test	Touch pen to colonies on illuminator	No problem	200.
American Instrument Co. Microscan	Counts bacterial colonies in agar tube.	All sample-handling must be modified for weightlessness.	1 ml agar + tube per test	Pour sample and agar into tube. Obtain light scatter counts. Incubate for several hours. Obtain light scatter counts.	Requires an incubator and light-bulb shielding.	

FOLDOUT FRAME

FOLDOUT FRAME

Table 4-1. Instrument Comparison Chart

Section 5
CENTRIFUGES

The category of laboratory instruments, "centrifuges", includes a range of instruments varying in complexity from simple bench-top instruments to complex analytical ultracentrifuges. The simple instruments use an electric motor to rotate a rack of sample tubes. Centrifugal force drives fine particles in the sample to the bottom of the tubes. Beyond this, little need be said of the simple instruments, except that they are a nearly indispensible laboratory instrument for the Space Station. On the other hand, ultracentrifuges, especially analytical ultracentrifuges, may not be necessary for Space Station application. They do, however, require considerable explanation because of the complexity of their design and operation. The simple centrifuges, although not discussed in detail, are most definitely required for Space Station applications The more complex centrifuges are discussed in detail to highlight some of their applications, limitations, and requirements.

5.1 PRINCIPLES OF OPERATION

The general operating principle of all centrifuges is the same. Components of liquid samples are separated by centrifugal force, the centrifugal force being generated by spinning a rotor which contains the sample. The rate of rotation and length of arm determine the forces "g's" developed according to the following relationship:

$$RCF = r N^2 1.118 \times 10^{-6} \quad (1)$$

where

RFC = relative centrifugal force (gravities)

r - radius (cm)

N = rotation speed (rpm).

Two general types of centrifuges can be distinguished on the basis of the g forces developed: general duty centrifuges with g forces below 50,000, and ultracentrifuges above 50,000.

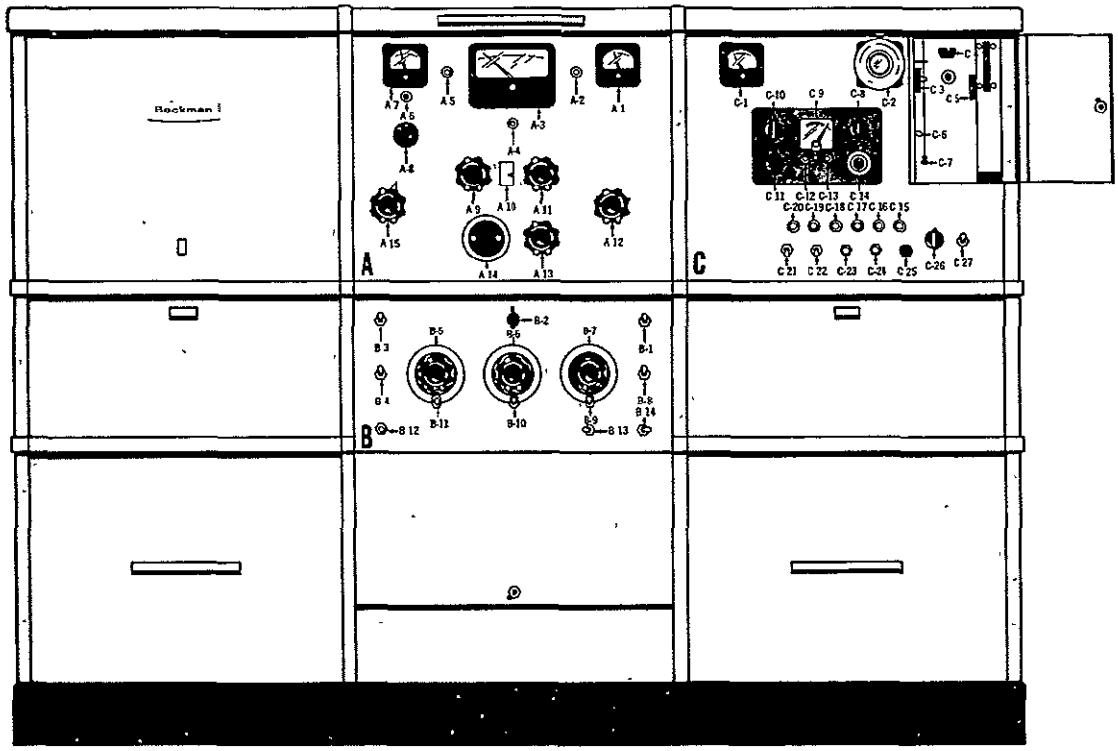
General duty centrifuges come in various sizes and with a variety of modifications and special features. They differ in the size sample permitted and the operating speed. Some centrifuges are special-duty instruments, such as the hematocrit centrifuges which are designed only to spin small capillary tubes. Some general-duty centrifuges are simple bench-top units with only speed controls. Others are relatively elaborate floor-model devices with self-timers and perhaps refrigerated sample compartments. Low-speed centrifuges, which have been specially designed to receive the sample cells of other instruments, may provide a solution to the problem of removal of air bubbles from samples used for optical analysis--the cuvettes of a spectrophotometer, for example.

Although ultracentrifuges extend the rotation speeds and g forces developed by centrifuges, they are, in complexity of design and application, rather different instruments. All ultracentrifuges operate at high vacuum (around 1 micron Hg) for long periods, and usually at reduced temperatures. Two general types of ultracentrifuges may be distinguished: the analytic centrifuge and the

preparative centrifuge. These differ principally in the size of sample handled, built-in optical components, and application. Preparative centrifuges (Figure 5-1) handle larger samples than analytical instruments, they generally have no built-in optical analysis systems, and they are used for preparation of samples for subsequent analysis--perhaps by an analytical ultracentrifuge. Current models of preparative ultracentrifuges operate at speeds up to 75,000 rpm and develop up to 400,000 g forces. Higher-performance instruments will undoubtedly be available within the next few years.

The analytical ultracentrifuge produces high centrifugal forces to measure the movement or redistribution of sedimenting particles. Depending upon the experimental conditions selected, the instrument can be used to determine many molecular parameters, including sedimentation coefficients, molecular weights, diffusion coefficients, particle size and shape, and partial specific volumes. The Analytical Ultracentrifuge is a standard tool for the study of proteins, enzymes, viruses, nucleic acids, and many natural and synthetic polymers. The instrument can also be used to study chemical reactions and the activities of inorganic ions, as well as the compressibility of gels and lattices. Diagrams of an analytical ultracentrifuge are shown in Figures 5-2 and 5-3.

When operating the analytical ultracentrifuge, the sample is placed in the centerpiece, a specially designed container (usually sector-shaped) at the center of a cell assembly. The cell assembly permits light rays to pass through its entire length. After the cell is assembled, it is placed in a rotor hole and an appropriately weighted counterbalance is placed in the opposite rotor hole to balance the rotor. The rotor is then installed in the rotor chamber. The chamber is evacuated, and the rotor is accelerated.



A

A-1. Ammeter
A-2. OVERSPEED Pilot Light
A-3. Tachometer
A-4. BRAKE Pilot Light
A-5. ACCELERATE Pilot Light
A-6. Lower Scale Button
A-7. Voltmeter
A-8. DRIVE SELECTOR Knob
A-9. SPEED SELECTOR Knob (COARSE)
A-10. SPEED SELECTOR Dial
A-11. SPEED SELECTOR Knob (FINE)
A-12. VOLTAGE CONTROL Knob
A-13. EXPOSURE INTERVAL Switch
A-14. EXPOSURE TIME Dial
A-15. TIMER

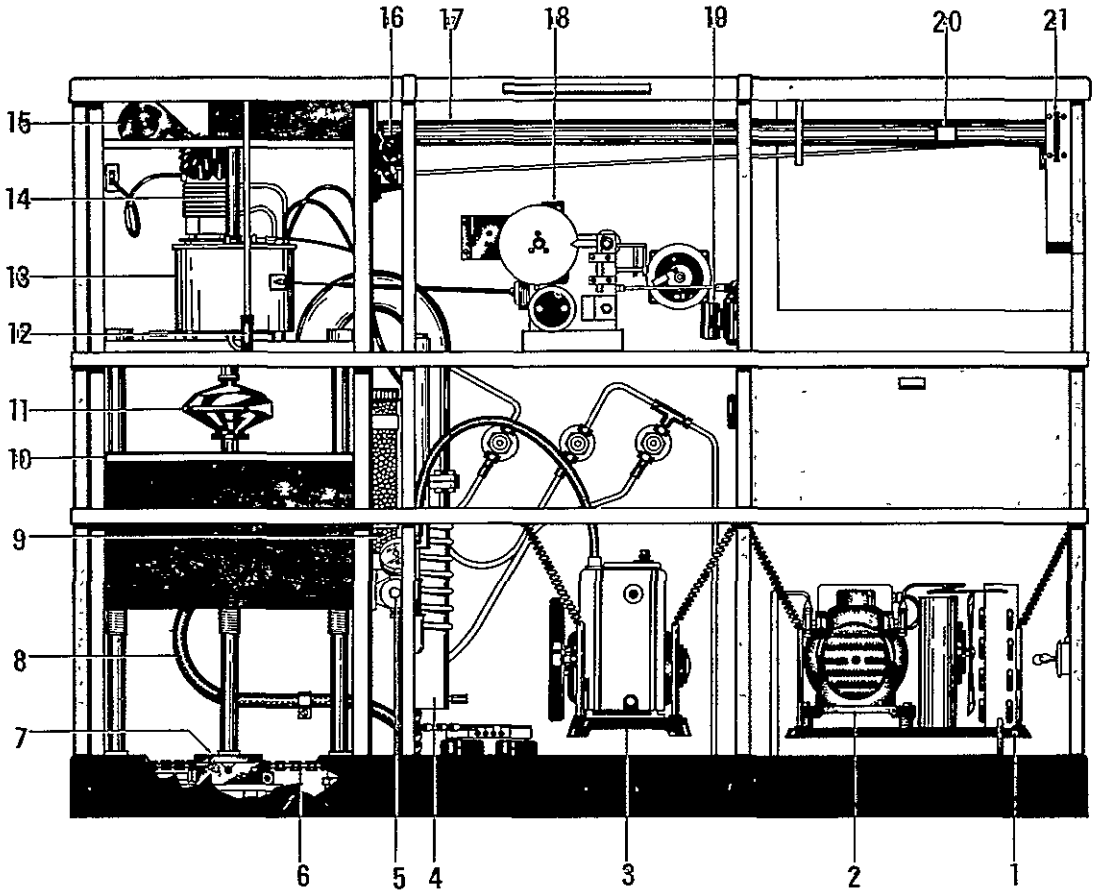
B

B-1. VACUUM GAUGE Switch
B-2. VACUUM GAUGE ADJUSTMENT Knob
B-3. REFRIGERATION Switch
B-4. VACUUM CHAMBER Switch
B-5. VACUUM PUMP AIR VALVE
B-6. DIFFUSION PUMP WATER VALVE
B-7. LIGHT SOURCE WATER VALVE
B-8. BRAKING RATE Switch
B-9. LIGHT SOURCE Switch
B-10. DIFFUSION PUMP Switch
B-11. VACUUM PUMP Switch
B-12. R.T.I.C. CALIBRATION Jack
B-13. Lightsource Intensity (HI LO) Switch
B-14. UV Lightsource Switch

C

C-1. Vacuum Gauge
C-2. Viewer
C-3. Phaseplate Adjustment Knob
C-4. Phaseplate Dial
C-5. Plate Position Dial
C-6. Camera Compartment Light Switch
C-7. Cord for Swinging-gate Assembly
C-8. RANGE Knob
C-9. RTIC Meter
C-10. ZERO ADJUST Knob
C-11. RTIC Function Selector Switch
C-12. RTIC Pilot Light
C-13. Heater Pilot Light
C-14. BALANCE Dial
C-15. CAMERA Pilot Light
C-16. REFRIGERATION Pilot Light
C-17. UV Lightsource Pilot Light
C-18. Schlieren/Interference Lightsource Pilot Light
C-19. DIFFUSION PUMP Pilot Light
C-20. MAIN POWER Pilot Light
C-21. AUTOMATIC PHOTO Switch
C-22. PLATE HOLDER DRIVE Switch
C-23. PLATE HOLDER DRIVE Button
C-24. VIEW SHUTTER Button
C-25. VIEW MIRROR Knob
C-26. CAMERA Selector Switch (selects Schlieren/Interference or UV cameras)
C-27. LONG SHIFT/SHORT SHIFT Switch

Figure 5-2. Analytical Ultracentrifuge (Controls)



1. Refrigeration Condenser
2. Refrigeration Compressor
3. Mechanical Vacuum Pump
4. Diffusion Pump
5. Evapotrol
6. Chamber Lift Mechanism
7. Schlieren/Interference Lightsource
8. Capillary Tube for Refrigeration
9. Drierite
10. Rotor Chamber
11. Rotor
12. Drive Oil Gauge
13. Rotor Drive
14. Drive Motor
15. Blower for Drive Motor
16. Plateshift Mechanism
17. Optical Tube
18. Differential Gearbox
19. Synchronous Motor
20. Viewer
21. Plate Holder Slot

Figure 5-3. Analytical Ultracentrifuge (Components)

As the run progresses, the instrument's component parts function to maintain optimum experimental conditions: the speed-control system minimizes any fluctuations in the selected speed caused by friction or line voltage; the vacuum system keeps chamber pressure at 1 micron; and the temperature control system maintains rotor temperature at the selected value by automatically compensating for any rise in temperature due to friction and heat conduction. Under these conditions, the sample material is subjected to high centrifugal forces that cause the molecular particles to sediment. As the particles are redistributed, light from the optical system light-source is transmitted through the transparent portion of the rotating cell. By means of this light, the optical elements translate particle movement into an optical pattern. Optical patterns can be viewed directly and photographed at preselected intervals, thus recording actual particle activity at various times during the run. Two of the optical systems of an analytical ultracentrifuge are shown in Figure 5-4. The uses of the different optical systems are shown in Table 5-1.

Analytical ultracentrifuges operate at speeds of up to 72,000 rpm and develop forces up to 430,000 g's. Future instruments will develop up to 750,000 g's at 100,000 rpm. Equations and symbols relative to centrifuges are included at the end of this section.

5.2 APPLICATIONS

General-duty centrifuges separate particles from liquid samples. A typical example of this is the separation of red blood cells from plasma during the centrifugation of whole blood.

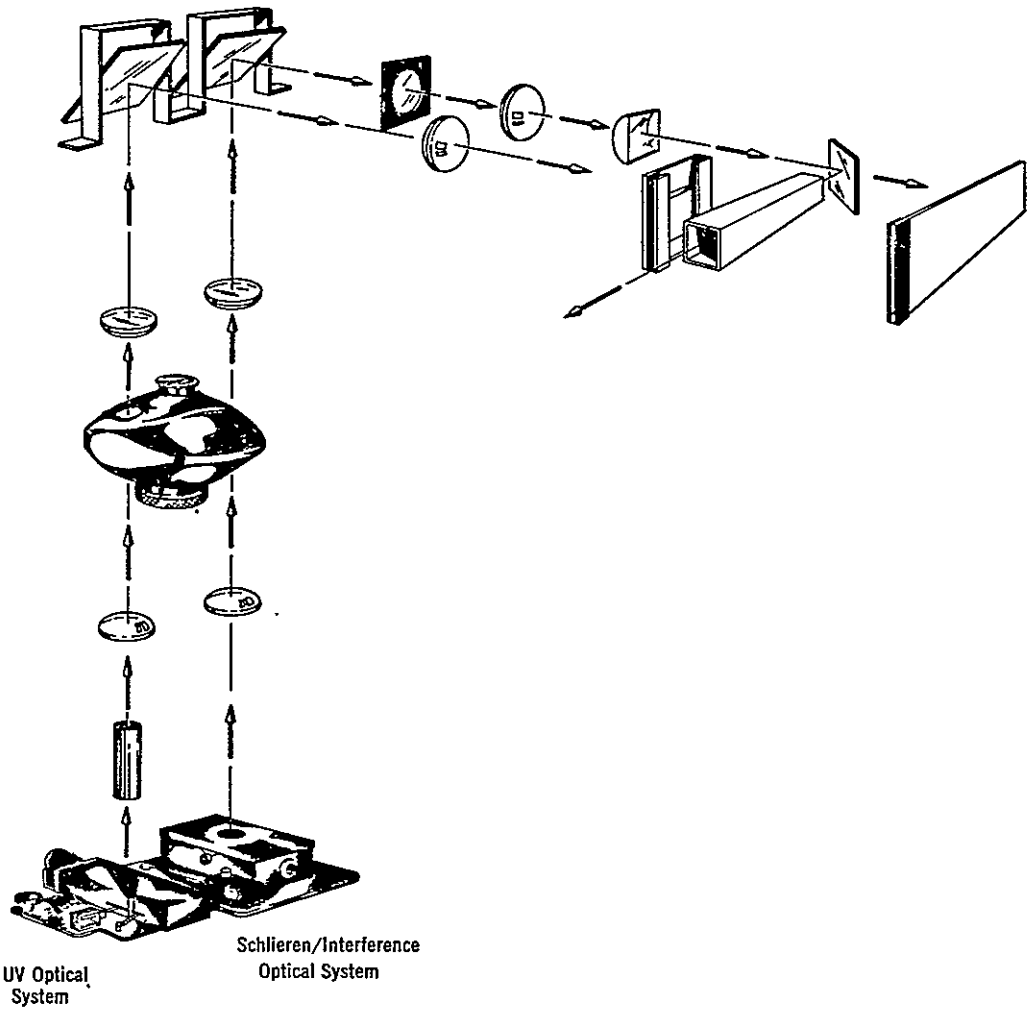


Figure 5-4. Optical Systems

	Schlieren	Interference	Absorption	
			Photographic UV	Photoelectric Scanner
Most important applications	Sedimentation velocity Studies of heterogencity Approach-to-equilibrium	Sedimentation equilibrium Concentration determination	Sedimentation velocity (dilute solutions) Equilibrium banding	Sedimentation equilibrium (dilute solutions) Sedimentation velocity (dilute solutions) Equilibrium banding
Other usual applications	Sedimentation equilibrium Concentration determination	Diffusion studies Sedimentation velocity		Small molecule binding Differential sedimentation
Usual concentration ranges (12 mm cells)	1.0 to 10 mg/ml	0.5 to 5 mg/ml	Proteins-- 0.1 to 1.0 mg/ml Nucleic acids-- 0.01 to 0.1 mg/ml	Proteins-- 0.05 to 1.0 mg/ml Nucleic acids-- 0.01 to 0.1 mg/ml
Special advantages	Direct viewing Direct determination of concentration gradients Heterogeneity visualized Variable sensitivity (phase plate angle)	Direct viewing Direct determination of relative concentrations High accuracy	Discrimination among solutes Applicable to very dilute solutions	Baseline provided Equivalent to direct viewing Discrimination among solutes Applicable to very dilute solutions
Particular disadvantages	Salts may interfere Integration required to obtain concentration Patterns sometimes difficult to read accurately	Differentiation required to obtain concentration gradients Sensitive to cell distortion	No direct viewing Inconvenient Sensitive to oil and dirt on optical components	Sensitive to oil and dirt on optical components

Table 5-1. A Comparison of Optical Systems

Preparative ultracentrifuges can be used for rate and zonal separations, for banding and diffusion experiments, and for establishing equilibrium conditions. Principally in biomedical experiments, preparative centrifuges prepare purified samples or separate components for further analysis.

In analytical ultracentrifuges, optical measurements are taken during the high-speed rotation of the sample. These instruments allow determination of molecular weights, sedimentation coefficients, and diffusion coefficients. The samples analyzed are generally various systems of micromolecules, viruses, or cellular components. Some polymer research uses the analytical ultracentrifuge, usually at higher temperatures.

Centrifuges are applicable to the following functional program elements:

5.9 Small Vertebrates (Bio D)
5.10 Plant Specimens (Bio E)
5.13 Biomedical and Behavioral Research
5.16 Materials Science and Processing
5.25 Microbiology (Bio C)
5.26 Invertebrates (Bio F)
5.27 Physics and Chemistry

5.3 LOGISTICS

5.3.1 Packing and Launch

Normal railroad shipping procedures should be adequate for centrifuges. Rotors and other accessories should be packed separately. The optical systems of analytical ultracentrifuges and some of the subsystems of the other complex types of centrifuges may require blocking during shipment.

5.3.2 Installation

Unpacking, reassembly, connection to electrical power, and tie-down should be adequate for installation of most centrifuges. Ultracentrifuges require water cooling (approximately 2K watts) and vacuum connection, if modified as discussed in Paragraph 5.7. Analytical ultracentrifuges will require realignment of the optical systems.

5.3.3 Consumable Supplies

General-purpose centrifuges require only sample containers; these may require special design (see Paragraph 5.4.4). Preparative ultracentrifuges require a good supply of sample tubes, buffer solutions, density-gradient solutions, lubrication oil, vacuum oil (vacuum pump oil can serve the latter two functions), and some freon for the refrigeration unit. In addition, analytical ultracentrifuges will need 1 or 2 spectrographic camera-plate recordings.

5.3.4 Accessories and Spare Parts

General-purpose centrifuges require no accessories beyond a few different types of rotors and sample holders.

Ultracentrifuges, on the other hand, require several different types of accessories. Several rotors should be taken--titanium rotors are recommended for safety. Spare drive parts and electronic subsystems should also be included. A density-gradient pump is a necessary accessory for preparative ultracentrifuges Current models may require some modification for zero-g application. The density-gradient pump mixes water and a sucrose solution in proportions varying with the

percentage of the sample delivered to produce a buffer solution with a density gradient from top to bottom of the tube. This preparation aids in the separation of components by density. An integrator may be desired for preparatory ultracentrifuges to calculate g's accumulated over time (this accounts for lower g's during acceleration to running speed). Density-gradient beads and reaction recovery systems may also be needed with preparatory ultracentrifuges.

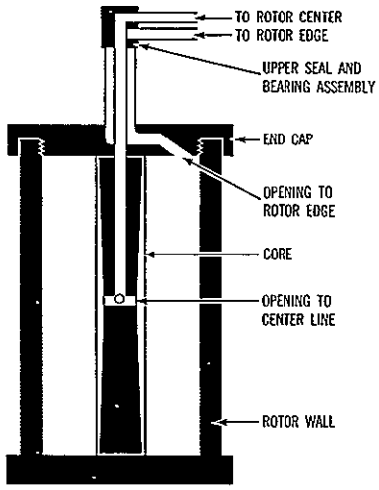
There are four general types of rotors available for preparative ultracentrifuges; swinging bucket rotors, fixed bucket rotors, continuous flow rotors, and zonal rotors. Zonal rotors, modified for small volumes, may be best suited for zero-g sample-handling conditions. A diagram of the operating principle of zonal rotors is shown in Figure 5-5.

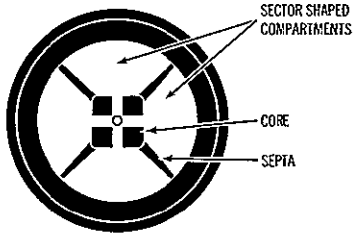
Analytical ultracentrifuges will need extra sample cells and a cell torque wrench for assembly of the cell. A microdensitometer and a microcomparator will be needed for analysis of the plates produced during an analytical centrifuge run.

5.3.5 Maintenance and Repair

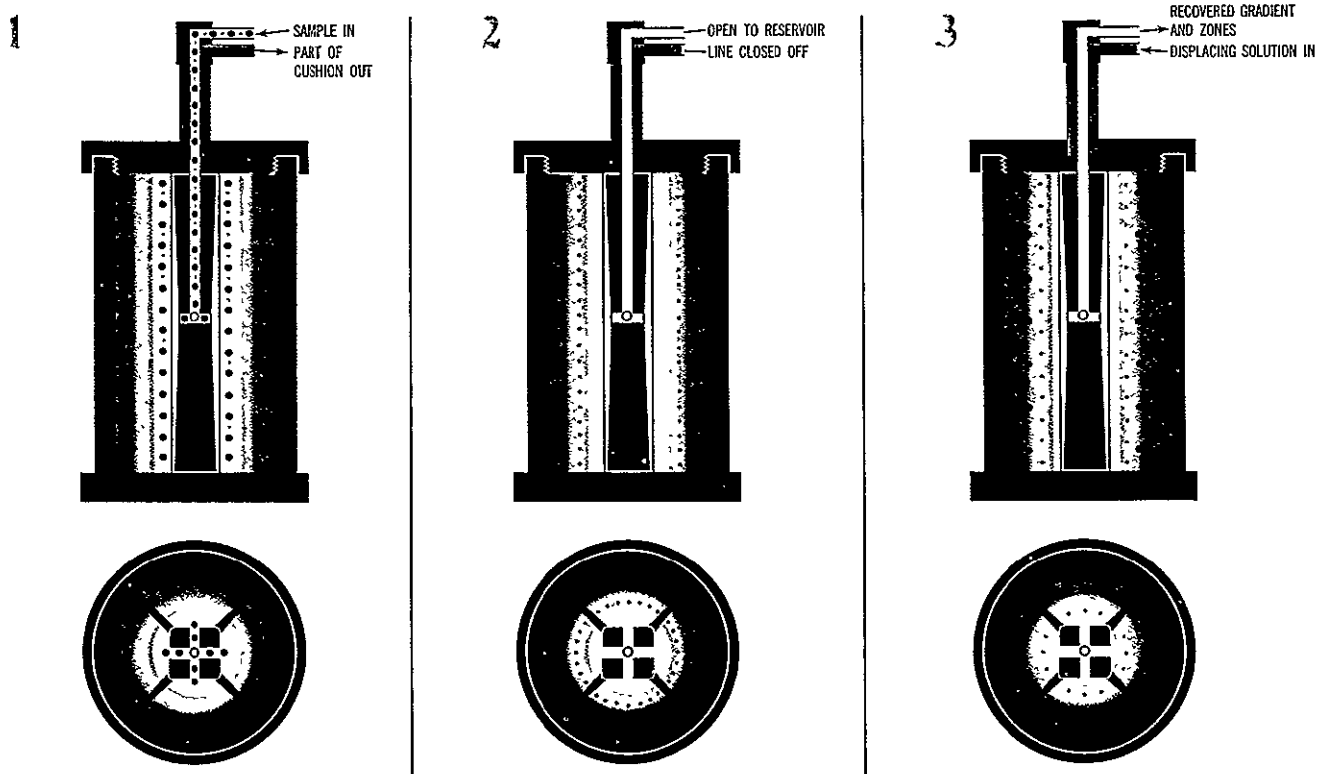
Maintenance should be approached on a replacement basis. Spare modules are recommended for electronic maintenance. A strict replacement schedule of critical drive-chain components should be established to prevent explosive mechanical failures during a run.

Schematic diagram of the B-4 Zonal Rotor· hollow cylinder is driven from the bottom and centered around the upper shaft by the Upper Seal and Bearing Assembly (not shown). The latter terminates ın a special enclosed fluid line seal. Septa of the core dıvide rotor chamber into four sector-shaped compartments to mınimize convection Horizontal cross section is shown below vertical cross section. Rotation is about the core axis.





How the B-4 Rotor Works



At low speed. gradient introduced to rotor edge, light end first; cushion introduced, forcing some light-end gradient out through core. Sample pumped in through core (see figure), dispelling part of cushion from rotor edge.

Overlay pumped in through core to move sample away from core. Rotor accelerated to operating speed to effect separations.

At low speed: dense fluid pumped to rotor edge; overlay displaced through core center line, followed by gradient with zones of particulates.

Figure 5-5. Zonal Rotors

5.4 OPERATION

5.4.1 Warm-up and Speed-of-Operation

General-purpose centrifuges accelerate to their operating speeds within a few seconds (or minutes for high-speed centrifuges). A typical "run" would last from several minutes to an hour or two. Ultracentrifuges take 15 to 20 minutes to establish a vacuum and another 15 to 20 minutes to accelerate the rotor to operating speed and thus require 30 to 40 minutes for "warm-up." Typical running times for ultracentrifuges are from 1/2 to 16 hours for preparative types and 2 to 24 hours for analytical instruments.

5.4.2 Operation Skills

General-purpose centrifuges can be operated by most personnel with only minimal previous experience or training. Preparative ultracentrifuges require technical personnel with some previous training and experience. Analytical ultracentrifuges require professional-level personnel with extensive previous experience.

5.4.3 Operating Procedures

For general-purpose centrifuges, the typical operating procedure is as follows:

- Load sample in tube (special techniques may be needed).
- Load tube in rotor (with counter balance if odd number of tubes used).
- Set time and temperature of run (if available on instrument).
- Start run.
- Terminate run and remove sample.

A typical operating procedure for a preparative ultracentrifuge is as follows:

- Calculate running time and speed.
- Load sample into tube (special techniques may be needed).
- Close tube (torque wrench may be needed).
- Place tube in rotor and seat rotor on drive spud.
- With lid open, check rotation at low speed for undesirable motion or vibration.
- Establish vacuum and accelerate to speed.
- Run for preset time at fixed temperature.
- Slow down, open, and remove sample.
- Prepare centrifuged sample for next analysis (a separated band may be drawn off into a syringe by puncturing the tube wall, for example).

Typical operating procedures for an analytical ultracentrifuge is as follows:

- Calculate time and speed needed for type of sample and analysis technique (see Appendix to this section for relevant relationships).
- Precool rotor.
- Prepare and fill sample cell (extensive experience needed). (The details of the sample cell of an analytic centrifuge are shown in Figure 5-6).
- Establish vacuum.
- Accelerate to running speed.
- Record data during run according to pre-established plan.
- After run, analyze photographic records (sample is usually thrown away but it may be retained for further analysis).

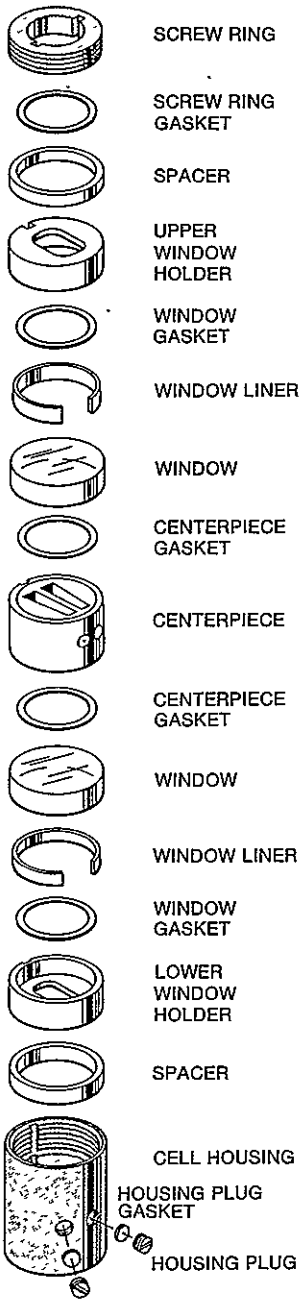


Figure 5-6. Sample Cell

5.4.4 Sample Preparation and Handling

Sample handling techniques needed for preparation of samples to be centrifuged present some unexpected problems. Although the sample will easily fill the bottom of the tube after beginning the centrifugation run, prefilling of the tube in the absence of local gravity is difficult. For general-purpose centrifuges, a syringe barrel which can be filled from closed, wet chemistry packets should be adequate. Some modification of the buckets may be needed. Modified buckets for low-speed centrifuges may find application for filling sample containers for analysis by optical methods which can not tolerate bubbles in the light path--the cuvettes of spectrophotometers, for example.

The sample tubes normally used with preparative ultracentrifuges are critically designed and can not tolerate some of the modifications which would make them compatible for filling from closed, wet chemistry modules. A possible solution to this problem for preparative ultracentrifuges may be the type of rotor. The zonal rotor is designed for spinning large volumes of liquids; nontheless, its basic design solves most of the sample-handling problems of preparative ultracentrifuges. The zonal rotor carries the sample in a torroidal-shaped compartment with a few separators. The inlet and outlet are provided through a face seal, the inlet connecting the (radial) center of the compartment with the outside and the outlet connecting the periphery of the cell with the outside.

The operation of the zonal rotor is shown in Figure 5-5. Samples can be introduced into a zonal rotor from a closed, wet chemistry system and extracted into a similar system. It is possible to fill a zonal rotor with a density gradient while it is operating, and then place a sample on top near the radial

center. After dispersing the sample through the density layers, the separated components are retrieved by flushing the entire cell with a more dense fluid which forces the separated layers out of the cell in sequence. The compatibility of zonal rotor techniques with the problems of sample-handling in zero gravity may make the zonal technique the most appropriate method for centrifugation in the Space Station.

Preparation of the sample cell of the analytical ultracentrifuge also presents the problem of filling the cell while excluding air from it. Some special techniques must be developed; these might include some centrifugation for removing bubbles from the sample.

5.5 INTERFACE

5.5.1 Interface with Other Laboratory Instruments

All centrifuges, except for the analytical ultracentrifuge, are preparatory instruments. They prepare a sample by separating some of its components for further analysis. The separated portion of the sample is then available to other analytical instruments--spectrophotometers or liquid scintillation counters, for example.

5.5.2 Interface with Vehicle Systems

Centrifuges, except for the small hand-operated models, require electric power. During acceleration, ultracentrifuges use power peaks of up to 630 amperes from a 220-V ac line. Ultracentrifuges also use water for cooling. Modification to use recirculating cooled water will be needed for Space Station application. In addition, ultracentrifuges internally provide 1 micron Hg of vacuum, and

cooling of the rotor chamber. With modification to the instruments, both of these requirements could be met from the space vehicle capabilities. If desired, the on-board data management system could control most of the operation of the ultracentrifuge (see Paragraph 5.7).

5.6 SAFETY

5.6.1 Flame Hazards

Centrifuges do not present any flame hazards under normal operating conditions. However, in the case of an explosion of the rotor of an ultracentrifuge, shorting of electronic components and spilling of samples can occur. It should also be pointed out that some types of centrifuge tubes in common use are made of cellulose nitrate. In some cases, these are replaceable with polycarbonate tubes.

5.6.2 Microbiological Hazards

Microbiological hazards could occur, if viable microorganisms are present in the samples, only in the case of spillage while preparing the sample or sample loss in a rotor explosion.

5.6.3 Electromagnetic Interference

The motors (Universal motors used in most) and relays in centrifuges radiate some electromagnetic radiation.

5.6.4 Ionizing Radiation

Radiation hazards could occur only in the case of spillage of samples containing radioactive isotopes.

5.6.5 Physical Hazards to Personnel

The possibility of rotor explosion was mentioned above. Rotors do explode in earth-based laboratories--not frequently, but they do. Explosions occur at high rotating speeds because of accumulated stresses in the rotor. The energy released during a rotor explosion approaches 800,000-foot pounds. The vacuum chamber surrounding the rotor is generally 1-1/2-inch-thick steel providing considerable protection from flying metal objects. However, the concurrent shock wave and forces applied through the instrument frame would make a rotor explosion a near cataclysmic event in a space-station environment. A preparative centrifuge, for example, jumps vigorously around the laboratory following a rotor explosion.

If an ultracentrifuge is used in the space station, safety standards must be set to make rotor explosion impossible. These safety procedures must include extreme care in loading of the sample containers. A leaking sample can unbalance a rotor and lead to an explosion. Careful monitoring of temperature and pressure in the vacuum chamber is necessary. A vacuum leak will produce heating and the rotor will fail. Overspeed protection devices must be used correctly. Careful records of use must be maintained for each rotor so that rotors can be discontinued long before they become candidates for metal fatigue failure.

5.7 MODIFICATIONS

General-purpose centrifuges will need few modifications. Ultracentrifuges, however, are amenable to several modifications. Modified sample containers may be needed. The vacuum pumps could be replaced by venting to the exterior. If this is not done, the output of the built-in vacuum systems must be trapped and scrubbed before venting into the laboratory environment. Cooling of the ultracentrifuges should be done by cooling and recirculating the water. A refrigeration unit is required. Actually, the whole operation of the centrifuge (after loading the sample) could be controlled by the data management system: it could calculate the time and speeds needed for the final conditions desired; it could monitor and control speed, temperature, and pressure; and it could keep the records on the past use of the rotors, their necessary derating, and projected replacement dates.

5.8 AVAILABLE INSTRUMENTS

General-purpose centrifuges are manufactured by:

- Aloe Scientific
- Balder Cryogenic
- Central Scientific
- Chemapec
- M. Christ--Elliott Mercantile
- Clay-Adams
- International Equipment
- Lab-Line Instruments
- Lourdes
- MSE--London
- Phillips-Drucker
- Precision Scientific
- Sargent-Welch
- Ivan Sorvall

Hematocrit centrifuges are manufactured by:

- M. Christ
- Clay-Adams
- Drummond Scientific
- International Equipment
- International Sales
- Lab-Line Instruments
- Lourdes
- MSE--London
- Phillips-Drucker

Preparative ultracentrifuges are manufactured by:

Arden Instruments	International Equip.
Beckman Spinco	International Sales
M. Christ	MSE--London

Analytical ultracentrifuges with optical systems are manufactured by:

Beckman Spinco	International Sales
M. Christ	MSE--London

DEFINITIONS OF PRINCIPAL SYMBOLS

η	Viscosity
λ	Wavelength
ω	Angular velocity in radians per second
π	Constant equal to 3.1416
ρ	Density in grams per cubic centimeter
σ	Standard deviation
θ	Phase plate angle
A	Area, usually in square centimeters
c	Concentration
c_m	Concentration at the meniscus
c_b	Concentration at the bottom of the cell
c_0	Initial concentration
$C.F.$	Conversion factor between concentration units for interference optics and schlieren optics
D	Diffusion coefficient in square centimeters per second
F	Magnification factor for image enlargement due to camera lens
J	Total fringe shift
j	Fringe shift
M	Molecular weight
M_n	Number average molecular weight
M_w	Weight average molecular weight
M_z	z-average molecular weight
n	Refractive index
R	Universal gas constant; equal to 8.315×10^7 ergs per degree per mole
r	Radial distance in centimeters, corrected for camera lens magnification
r_m	Radial distance to the meniscus
r_b	Radial distance to the bottom of the cell
r_p	Radial distance to a point in the plateau region
S	Svedberg; unit of sedimentation rate equal to 10^{-13} seconds
s	Sedimentation coefficient
$s_{20,w}$	Sedimentation coefficient corrected to the value it would have in a solvent with the viscosity and density of water at 20°C.
T	Absolute temperature
t	Time
$\bar{v}$	Partial specific volume in cubic centimeters per gram
x	Radial distance measured on photographic plates before correction for camera lens magnification
y	Vertical distance measured on photographic plates
$(1 - \bar{v}\rho)$	Buoyancy factor
$\frac{dc}{dr}$	Derivative of concentration with respect to radius; the concentration gradient
$\frac{\partial c}{\partial r}$	Partial derivative of concentration with respect to radius; the concentration gradient at time t
$\frac{d\rho}{dr}$	Derivative of density with respect to radius; the density gradient

EQUATIONS	SPECIAL SYMBOLS USED IN EQUATIONS
1	
$\rho_{sol} = \rho_{solv} + c(1 - \rho_{solv}\bar{v})$	ρ_{sol} Density of solution in g/cm^3 ρ_{solv} Density of solvent in g/cm^3 c Concentration in g/ml of solution
2	
$\frac{c_{obs}}{c_s} = \frac{s_f - s_s}{s_f - s_{s(f)}}$	c_{obs} Observed concentration of slow component c_s Actual concentration of slow component s_f Sedimentation coefficient of fast component s_s Sedimentation coefficient of slow component $s_{s(f)}$ Sedimentation coefficient of slow component in the presence of the fast component
3	
$c_t = c_0\left(\frac{r_0}{r_t}\right)^2$	c_t Concentration at time t c_0 Initial concentration (at zero time) r_0 Radial distance to the maximum ordinate of the schlieren peak at zero time r_t Radial distance to the maximum ordinate of the schlieren peak at time t
4	
$s = \frac{1}{\omega^2 r}\frac{dr}{dt} = \frac{2\,303}{60\,\omega^2}\left(\frac{d\log x}{dt'}\right)$	t Time in seconds t' Time in minutes
5	
$s_{20,w} = s_{obs}\left(\frac{\eta_t}{\eta_{20}}\right)\left(\frac{\eta_{sol}}{\eta_w}\right)\left(\frac{1 - \bar{v}\rho_{20,w}}{1 - \bar{v}\rho_{t,sol}}\right)$ $\rho_{t,sol} \approx \left(\frac{\rho_{sol}}{\rho_w}\right)\rho_{t,w}$	s_{obs} Observed sedimentation coefficient η_t Viscosity of water at t degrees (temperature of centrifuge run) η_{20} Viscosity of water at 20 degrees η_{sol} Viscosity of sample solution at known temperature, t' η_w Viscosity of water at t' degrees $\rho_{20,w}$ Density of water at 20 degrees $\rho_{t,sol}$ Density of sample solution at t degrees (temperature of centrifuge run) $\rho_{t,w}$ Density of water at t degrees (temperature of centrifuge run)
6	
$\bar{r}^2 = \frac{\int_{r_m}^{r_b} r^2\,dc}{\int_{r_m}^{r_b} dc} = \frac{\Delta j \sum_{r_m}^{r_b} r^2}{J}$	$\bar{r}$ Average radial distance (position of the second moment of the gradient curve) Δj Increment of fringe shift—usually one fringe

7

$$\bar{r}^2 = \frac{\int_{r_m}^{r_b} r^2\left(\frac{\partial c}{\partial r}\right)dr}{\int_{r_m}^{r_b}\left(\frac{\partial c}{\partial r}\right)dr} = \frac{\sum_{r_m}^{r_b} r^2\left(\frac{\partial c}{\partial r}\right)}{\sum_{r_m}^{r_b}\left(\frac{\partial c}{\partial r}\right)}$$

$\bar{r}$ Average radial distance (position of the second moment of the gradient curve)

8

$$M = \frac{RTs}{D(1 - \bar{v}\rho)}$$

9

$$c_m = c_0 - \frac{r_b^2(c_b - c_m) - \int_{r_m}^{r_b} r^2\, dc}{r_b^2 - r_m^2}$$

$$= c_0 - \frac{r_b^2(c_b - c_m) - \Delta j \sum_{r_m}^{r_b} r^2}{r_b^2 - r_m^2}$$

Δj Increment of fringe shift—usually one fringe

10

$$M = \frac{2RT}{(1 - \bar{v}\rho)\omega^2}\,\frac{2.303(d \log c)}{d(r^2)}$$

11

$$M_z = \frac{M_{wb}c_b - M_{wm}c_m}{c_b - c_m}$$

M_{wb} Weight average molecular weight at the bottom of the cell
M_{wm} Weight average molecular weight at the meniscus

12

$$M = \frac{2RT}{(1 - \bar{v}\rho)\omega^2(r_b^2 - r_m^2)}\,\frac{c_b - c_m}{c_0}$$

13

$$M = \frac{RT}{(1 - \bar{v}\rho)\omega^2}\,\frac{d}{dc}\left(\frac{1}{r}\,\frac{dc}{dr}\right)$$

$$= \frac{RT}{(1 - \bar{v}\rho)\omega^2}\,\frac{slope}{\left(\frac{C.F.}{\tan\theta}\right)}.$$

14

$$M = \frac{2RT}{(1 - \bar{v}\rho)\omega^2} \frac{2.303\, d \log\left(\frac{1}{r}\frac{dc}{dr}\right)}{d(r^2)}$$

15

$$M = \frac{RT}{(1 - \bar{v}\rho)\omega^2} \frac{1}{r_{mid} c_0} \left(\frac{dc}{dr}\right)_{mid}$$

16

$$c_m = c_0 - \frac{1}{r_m^2} \int_{r_m}^{r_p} r^2 \left(\frac{\partial c}{\partial r}\right) dr$$

$$= c_0 - \frac{1}{r_m^2} \left(\frac{\Delta x}{F}\right) \sum_{r_m}^{r_p} r^2 \left(\frac{\partial c}{\partial r}\right)$$

17

$$M = \frac{RT}{(1 - \bar{v}\rho)\omega^2} \frac{1}{r_m c_m} \left(\frac{\partial c}{\partial r}\right)_m$$

18

$$A = \frac{A_{cal} h}{\phi X m_1} (n_{sol} - n_{solv})$$

A_{cal} Area measured on Calibration Cell photo in cm^2
h Effective cell height—equal to the centerpiece thickness
ϕ Angle of deviation of Calibration Cell
X Distance between scribe lines on Calibration Cell
m_1 Magnification due to the camera lens (same value as F)
n_{sol} Refractive index of solution
n_{solv} Refractive index of solvent

19

$$C.F. = \frac{A_{sch} \tan \theta}{J}$$

A_{sch} Area under a schlieren peak in cm^2

20

$$C.F = \frac{A_{cal}\, \lambda \tan \theta}{\phi X m_1}$$

X Distance between scribe lines on Calibration Cell
m_1 Magnification due to camera lens (same value as F)
ϕ Angle of deviation of Calibration Cell
A_{cal} Area measured on Calibration Cell photo in cm^2

21

$$r_e = \sqrt{\frac{r_b^2 + r_m^2}{2}}$$

r_e Radial distance to the isoconcentration point

22

$$\rho_s = \rho_e + \left(\frac{d\rho}{dr}\right)_{r_e}(r_s - r_e)$$

ρ_s Mean buoyant density at the center of a sample band
ρ_e Density of the gradient at the isoconcentration point—equal to ρ_0
r_e Radial distance to the isoconcentration point
r_s Radial distance to the center of a sample band

23

$$\left(\frac{d\rho}{dr}\right)_{r_e} = \frac{\omega^2 r_e}{\beta}$$

r_e Radial distance to the isoconcentration point
β Quantity relating the density gradient formed by a solution to centrifugal force and to the concentration and physical properties of the solute

24

$$M = \frac{RT\rho_0}{\sigma^2\omega^2 r_s\left(\frac{d\rho}{dr}\right)_{r_s}}$$

r_s Radial distance to the center of a sample band
ρ_0 Initial density

25

$$D = \frac{\Delta r^2}{4y^2 t} = \frac{\Delta r^2}{t}\,\frac{1}{3.64}$$

y Argument of the error function

26

$$D_{20,w} = D_{obs}\left(\frac{293.2}{T}\right)\left(\frac{\eta_t}{\eta_{20}}\right)\left(\frac{\eta_{sol}}{\eta_w}\right)$$

D_{obs} Observed diffusion coefficient in cm^2/sec
$D_{20,w}$ Diffusion coefficient corrected to the value it would have in a solvent with the viscosity of water at 20°
η_t Viscosity of water at t degrees (temperature of diffusion run)
η_{20} Viscosity of water at 20 degrees
η_{sol} Viscosity of sample solution at known temperature, t'
η_w Viscosity of water at t' degrees

27

$$D_A = \frac{(A_{sch})^2}{\left(\frac{dc}{dr}\right)^2_{max}}\,\frac{1}{4\pi t}$$

D_A Diffusion coefficient determined by the height-area method
A_{sch} Area under a schlieren peak in cm^2

Section 6
ELECTRONIC HEMATOCRIT

6.1 PRINCIPLES OF OPERATION

The hematocrit value is usually defined in the percent of the total blood volume occupied by the red blood cells. In the standard test, a calibrated glass test tube is filled with blood (to the "100" mark). The blood cells are separated from the plasma by centrifugation. The packed red cell volume then is read off the scale and reported as the hematocrit. Modern techniques use a capillary glass tube that can be centrifuged. Modern techniques use a capillary glass tube that can be centrifuged. The relative volume occupied by the blood cells is then determined by laying the tube on a proportionality scale and reading the hematocrit. Alternately, the hematocrit can be calculated, if the blood cell count and average blood cell volume is known. These measurements can be determined by using cell counters similar to those described in Section 4. As is true of many indirect methods, this technique may have a large error.

With the electronic hematocrit instrument, the blood is drawn into a capillary tube with electrodes at either end. Conductance through the tube is then determined by using a 10,000 Hz oscillator to drive a bridge circuit. Since blood cells can be thought of as insulating spheres, higher conductance values are obtained with lower hematocrit samples. This technique has not been popular in standard clinical laboratories because it cannot be used with oxalated blood. It can be used only with untreated or heparinized blood. In the

latter case, excellent agreement between the electronic and centrifugal hematocrit methods is obtained, but the electronic system is considerably easier to use.

6.2 APPLICATIONS

The electronic hematocrit instrument is applicable to the following functional program elements (FPE's):

5.9 Small Vertebrates (Bio D)
5.13 Biomedical and Behavioral Research
5.23 Primates (Bio A)

6.3 LOGISTICS

No special launch packing procedures and no consumable supplies are needed. Several spare capillary tubes (one for each test) should be taken as insurance against clotting within the tube. Installation does not pose special consideration other than the normal precautions to be taken in a zero-g environment. Special maintenance procedures are not needed.

6.4 OPERATION

The electronic reader will warm up within one or two seconds. After a blood sample is available by lancet or venipuncture, placing a dry capillary tube against a drop of blood will cause it to enter the tube. The tube is then placed in the reader and the hematocrit value is read directly. Flushing with water or saline solution will ready the tube for another sample. The flushing must be into a liquid disposal port. This instrument can be operated with a minimum of training.

6.5 INTERFACE

The system is 5-1/2 inches wide, 4 inches deep, and 6-1/2 inches high, and will pose no installation problems with other laboratory instruments or ventilation systems. The instrument can be operated with either 110 V ac or 6 V dc power.

6.6 SAFETY

Even though the glass capillaries are fabricated from heavy wall tubing, they should be sheathed in plastic to protect from breakage.

6.7 MODIFICATIONS

Modifications are required to permit taking samples from a zero-g wet chemistry container. For example, a syringe, needle, and tubing could be used.

6.8 AVAILABLE INSTRUMENTS

The only available instrument is made by Yellow Springs Instrument Company and sells for $250.

Section 7
ELECTRONIC TEST EQUIPMENT

7.1 PRINCIPLE OF OPERATION

7.1.1 Oscilloscope

The oscilloscope is a universal measuring instrument capable of measuring a very wide variety of rapidly changing electrical phenomena. The oscilloscope, in its usual mode, graphs these electrical changes with respect to time. An alternate mode available on some oscilloscopes allows graphing of one waveform against another waveform without respect to time (X-Y operation).

A block diagram of a typical multifunction oscilloscope is shown in Figure 7-1. The basic parts of an oscilloscope include the following:

- The cathode ray tube (CRT) is the output device.
- The time-base generator produces a linear ramp voltage which drives the CRT in the horizontal plane a calibrated number of centimeters per unit of time. Rates of 1 second per cm to 0.5 μsec per division are usually provided.
- Vertical amplifiers provide amplification for one or more electronic signals. The amplifiers then drive the CRT in the vertical plane. A method is provided to simultaneously display all input signals on the CRT. A wide range of calibrated and variable gains are provided. The calibrations are presented in volts, millivolts, and sometimes microvolts per division of deflection.

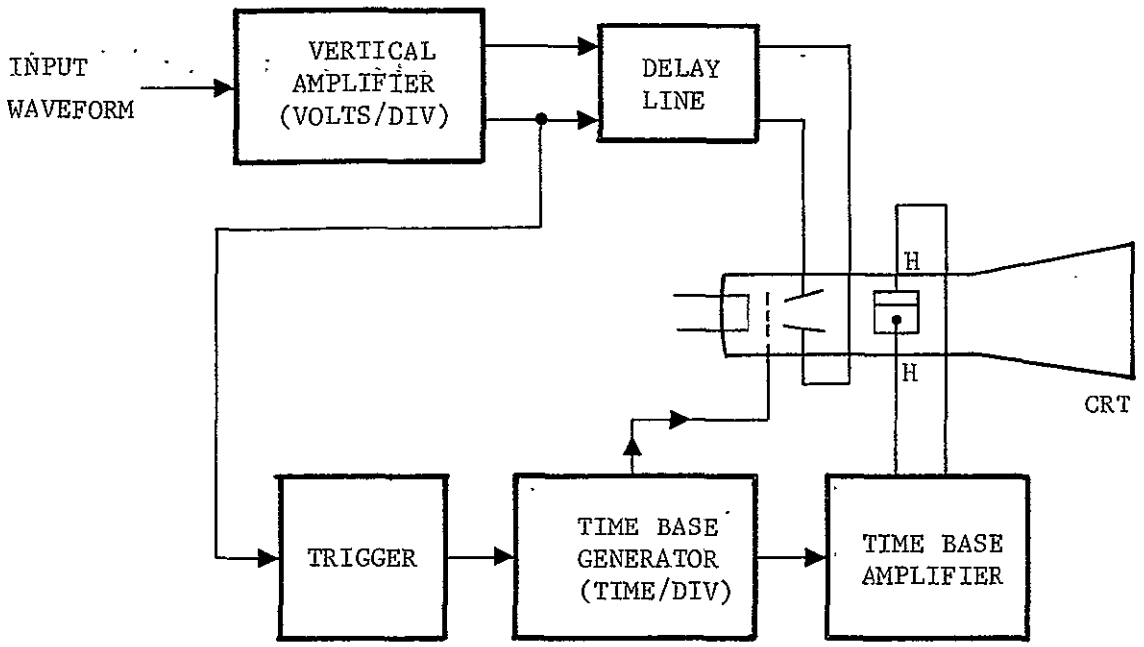


Figure 7-1. Typical Oscilloscope Block Diagram

- Power supplies provide the wide range of dc voltages needed in an oscilloscope. Most power supplies are operated from 120 volt (nominal) or 220 volt (nominal), 50 Hz to 400 Hz power lines. Some oscilloscopes operate with low voltage dc power or from battery packs. This method provides truly portable operation.

7.1.2 Digital Multimeter

The basic measurement for most digital multimeters is dc voltage. Current is read by measurement of the voltage across a precision resistor through which the unknown current flows. Resistance is measured by measuring the voltage across an unknown resistor through which a constant, precision current is

passed. A discussion of the method of conversion of dc voltage to a digital display is beyond the scope of this report.

If the multimeter is capable of measuring the ac voltage and current, these are measured in the same way with the ac voltage first converted to a dc voltage.

Figure 7-2 is a block diagram of a basic digital multimeter. The measurement is read out on a digital display typically composed of neon-filled "Nixie"* tubes. The new light-emitting diode arrays have not yet found much use in digital multimeters, but development in this area is expected before the Space Station is launched.

At this time, the majority of portable digital multimeters feature 3-1/2 digit displays. The fourth digit is a "one" bar to allow over-range to 1200, or in some instruments to 1999. Less portable multimeters are available with the greater accuracy of 4-1/2 digits.

Many digital multimeters include automatic range selection. This reduces the number of manual meter adjustments; however, many of the more portable multimeters do not now provide auto-range as an option.

Battery operation is preferred for troubleshooting. Connections to power buses within the Space Station are not necessary except for battery recharge

*A trademark of the Burroughs Corporation

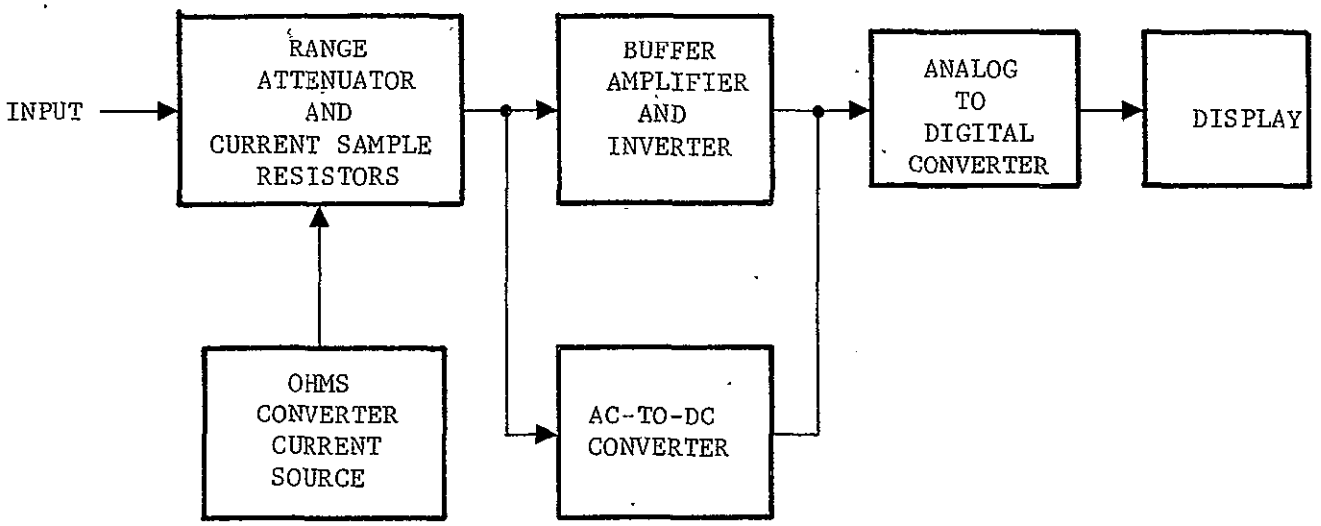


Figure 7-2. Basic Digital Multimeter Block Diagram

or for operation in excess of battery rating time limits. Battery operated 4-1/2 digit instruments will undoubtedly become available in the near future.

7.1.3 Function Generator

A function generator produces square, triangular, and sine waveforms at frequencies ranging from 0.01 Hz to 1 MHz. Other waveforms, including sawtooth and burst patterns, are available in some instruments. For most applications the square, triangular, and sine waveforms are sufficient.

A block diagram of a basic function generator is shown in Figure 7-3. In most designs, the triangular and square waveforms are the basic waveforms generated. The sine waveform is formed from the triangular waveform by nonlinear shaping circuits. Precision shaping is necessary to provide a low harmonic distortion waveform which will not produce excessive sine distortion

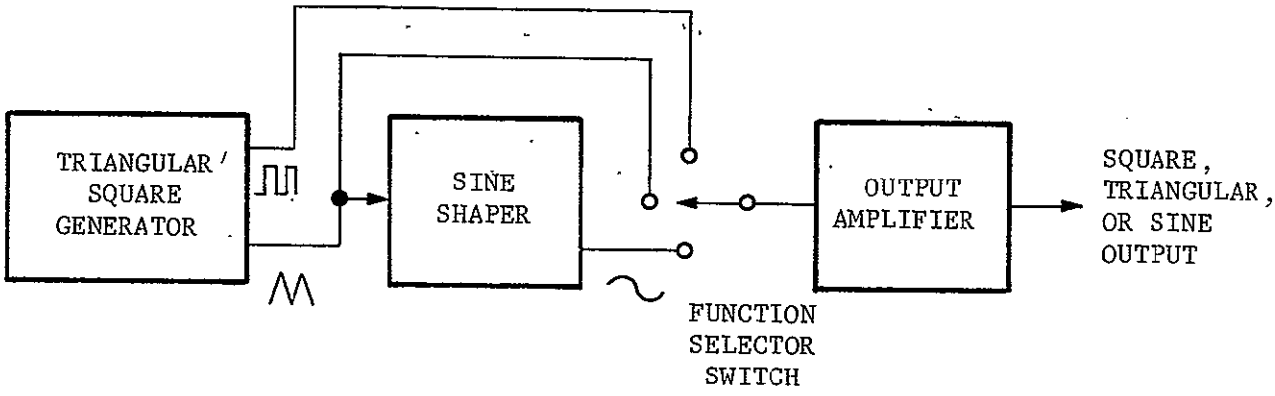


Figure 7-3. Basic Function Generator Block Diagram

when used with some types of filter circuits (differentiators and high-pass filters).

Battery operation is desirable and is available in some function generators as options. Others can be easily converted to fully portable use by addition of a battery pack and charging circuits.

7.2. APPLICATIONS

7.2.1 Oscilloscopes

Oscilloscopes can be used for (but not limited to) the following applications:

- Measurement of low level (5 millivolt and less) to high level (160 volt and more with special probes) ac or dc voltages.
- Measurement of frequencies of periodic waveforms from below 1 Hz to above 5 MHz.
- Measurement of periodic waveforms from 5 seconds to 1 μsec.

- Measurement of rise and fall times of digital waveforms.
- Display of two or more waveforms (on oscilloscopes with multi-trace capability) for purposes of comparison of relative timing or for "cause and effect" investigations.
- Comparison of two waveforms on an X-Y basis with one waveform displayed vertically and the other horizontally.
- Analysis of single occurrences in the single-sweep mode (if available).

For the Space Station application, a light-weight, battery-operated oscilloscope could be used for troubleshooting and maintenance of the various types of laboratory instrumentation. However, lighter weight oscilloscopes usually sacrifice bandwidth, sensitivity, and other more sophisticated features such as dual or multitrace operation.

7.2.2 Digital Multimeter

Digital multimeters are a necessary instrument for troubleshooting of any laboratory electronic instrument. The multimeter should include the following as a minimum:

- Dc voltage ranges from 1 volt full scale to 1000 volts full scale (a 100 mV full scale range is desirable).
- Dc current ranges from 1 mA full scale to 1 ampere full scale.
- Resistance ranges from 1 k full scale to 10 megohms full scale.
- Ac voltage from 1 volt full scale to 1000 volts full scale.

- Over-voltage protection.
- 3-1/2 digit readout (4-1/2 or 5 digit preferred).
- Automatic ranging is desirable but not necessary.

7.2.3 Function Generator

A function generator is a useful instrument for troubleshooting. Signal substitution techniques can be used as follows:

- Test amplifiers for gain, bandwidth, and damping factor with sine or square waves.
- Test digital circuits for proper counting, sequencing, and control with square waves.
- Exercise some servo systems to test proper operation with triangular or square waves.

7.3 LOGISTICS

7.3.1 Packing and Launch

The packing procedures for electronic test equipment should present no problems. Most instruments built for portable operation are inherently more rugged and able to withstand moderate amounts of shock and vibration. The oscilloscope CRT represents the most sensitive component in this instrument. A CRT could be removed and packed separately to better withstand the rigors of launch, then reinstalled later when the instrument is needed. In general, normal packing for commercial-rail-type shipment should suffice for this classification of instruments.

"Nixie" display tubes, which are presently used in the majority of digital multimeters, represent the most fragile component in this equipment. Newer light-emitting diode displays, when they become more widely used, will provide a more rugged digital display for space use.

7.3.2 Installation

Unpacking these instruments and preparing them for use is routine. Some provision is needed to mount the individual equipment in a zero-g environment when used for troubleshooting a laboratory instrument anywhere within the Space Station. When not in use, the equipment requires some type of hold-down or compartment with some provision for recharging of the batteries from the 400 Hz line, if battery-operated portable equipment is selected.

7.3.3 Consumable Supplies

No consumable supplies are required.

7.3.4 Accessories and Spare Parts

Accessories for an oscilloscope should include the following:

- Oscilloscope probes, X1.
- Oscilloscope probes, X10, low-input capacitance for measuring in digital circuit or other critical work.
- Clamp-on current probe to measure higher level current waveforms (ac only) without breaking a connection.
- Miscellaneous cables and test plugs compatible with the test points and test connectors of the various instruments.
- Alignment tool to adjust the oscilloscope gain, dc balance, etc., as required.

Spare parts for an oscilloscope should include the following:

- Fuses.
- Pilot bulbs.
- Hardware for mechanical repairs such as knob repair or replacement.
- Complete set of batteries, precharged.

Accessories for a digital multimeter might include miscellaneous cables and test plugs compatible with the test points and test connectors of the various instruments.

Spare parts for a multimeter should include the following:

- Fuses.
- Replacement display tubes (if "Nixie" type).
- Hardware for mechanical repairs such as knob replacement or repair.
- Complete set of batteries, precharged.

Accessories for a function generator might include miscellaneous cables and test plugs and probes compatible with the test points and test connectors of the various instruments.

Spare parts for a function generator should include the following:

- Fuses
- Pilot bulbs
- Hardware for mechanical repairs
- Complete set of batteries, precharged

7.3.5 Maintenance and Repair

Due to the compact size necessary for portability, maintenance may be difficult other than replacement of fuses, pilot bulbs, display tubes, and batteries.

7.4 OPERATION

7.4.1 Warm-up and Speed-of-Operation

No warm-up is required. Each of the Electronic Test Equipments is immediately ready for operation.

7.4.2 Operation Skills

Space Station operation of the electronic test equipments is possible for technical or professional personnel. However, in the case of the oscilloscope, previous experience or training is necessary. Generally, personnel who are trained to perform maintenance on instrumentation would have the necessary skills.

7.4.3 Operating Procedures

A typical procedure is as follows:

- Mount function generator and oscilloscope or digital multimeter near instrument requiring maintenance.
- Turn on instruments.
- Connect appropriate cables from generator to appropriate circuit location and input to appropriate measurement instrument from test point.
- Adjust measurement instrument and function generator mode, frequency and amplitude as required.
- Interpret and correlate measurements as necessary.

- Turn off instruments when maintenance completed.
- Remove to equipment storage area and recharge batteries.

7.4.4 Sample Preparation and Handling

Not applicable.

7.5 INTERFACE

7.5.1 Interface with Other Laboratory Instruments

The interface between electronic test equipment and the instrument to be repaired is, of course, required. Necessary cables will be provided along with the electronic test equipment.

7.5.2 Interface with Vehicle System

Power from the vehicle will be required for recharging of the batteries or for operation in excess of the battery discharge time ratings. Operation on 400 Hz will present no problem.

7.6 SAFETY

7.6.1 Flame Hazards

Not applicable.

7.6.2 Microbiological Hazards

Not applicable.

7.6.3 Electromagnetic Interference

The oscilloscope presents sources of interference generation in the CRT high-voltage generation circuits and in the sweep-generation circuits. Better

quality portable oscilloscopes are designed to meet the requirements of MIL-I-6181D. A metal mesh filter may be required over the CRT face to meet the high frequency (greater than 25 MHz) requirements.

The digital multimeter presents a source of interference generation in the digital logic circuitry. For 400 Hz operation, line filters may be necessary to control interference conducted back into power line. The use of a well-designed metal case (plastic is not an acceptable material) should reduce radiated interference to acceptable levels.

The function generator presents no significant source of interference generation except that which is required to send the output signal to the instrument under test. Shielded test cables are advised to control radiated interference.

7.6.4 Ionizing Radiation

The high voltage of most oscilloscopes is not sufficiently high to generate ionizing radiation. Portable oscilloscopes will present no problem. Also, function generators and digital multimeters will present no problem.

7.6.5 Physical Hazards to Personnel

Only sharp corners (if any) and protruding knobs present some physical hazard to personnel.

7.7 MODIFICATIONS

Only very minor modifications will be necessary to adapt available portable oscilloscopes, digital multimeters, and function generators to Space Station

use. These modifications will include some provision for mounting and replacement of plastic parts with metal parts.

Additional modifications of the function generator might include the addition of a battery pack and charger for fully portable operation.

7.8 AVAILABLE INSTRUMENTS

The major manufacturers of portable oscilloscopes are as follows:

- Textronix, Inc. (including Sony/Tektronix and Telequipment)
- Hewlett-Packard Co.
- General Atronics Corp.
- Dumont
- Leader Instrument Corp.
- Phillips Electronic Instruments

The major manufacturers of portable battery-operated digital multimeters are as follows:

- Digilin Digital Instruments
- John Fluke Manufacturing Company
- Honeywell
- Hickock Instrumentation Group

Other major manufacturers of digital multimeters (not necessarily battery-operated portable) are as follows:

- California Instruments Corp.
- Keithley Instruments, Inc.
- Non-Linear Systems
- Simpson Electric Company

- Cubic Corp.
- Dana Laboratories, Inc.
- Doric Scientific Corp.
- Dyna Sciences Corp.
- Hewlett-Packard Company
- Systron-Donner Corp.
- United Systems Corp

The major manufacturers of light-weight function generators are as follows:

- Hewlett-Packard Company
- Interstate Electronics Corp.
- Wavetek
- Exact Electronics, Inc.
- Systron-Donner (Datapulse)
- Krohn-Hite Corp.
- Beckman Instruments, Inc.
- E-H Research Laboratories, Inc.
- Honeywell
- Hickock Instrumentation Group
- Chronetics, Inc.

Section 8
ELECTROPHYSIOLOGICAL EQUIPMENT

Electrophysiological recording equipment is used for measuring and recording a broad spectrum of physiological functions in living organisms. From an instrumentation point of view, three general categories of recording situations can be distinguished: biopotential recordings, bioimpedance recordings, and transducer recordings. Biopotential recordings are the most common. They include the electrocardiogram (ECG), the electroencephalogram (EEG), the electrooculogram (EOG), the electromyogram (EMG), and many others. Bioimpedance recordings have been known for many years and are currently finding increased application in the areas of impedance pneumography, impedance cardiography, and measurement of local perfusion. Transducer recordings refer to situations in which a transducer is used to convert biological variables into an electric signal. These include: measurement of body temperature with a thermistor, measurement of respiration with pneumograph, measurement of muscle activity with a strain gage, measurement of blood pressure with a pressure transducer, and many others.

8.1 PRINCIPLES OF OPERATION

Electrophysiological recording equipment consists of a sensor, a signal conditioner/amplifier, and a readout device. The sensors vary widely with the variable measured and type of measurement. The amplifiers have many common features and are often interchangeable for a large number of different types of measurements. Signal conditioning (filtering, integration, attenuation, etc.) is often done in the amplifier or preamplifier. Depending upon the rate

at which a particular phenomena is occurring, the readout from this equipment is either printed out on a strip-chart recorder, or photographed from a CRT (cathode ray tube). In cases where the phenomena occurrence rates are relatively slow, the devices can be interchanged or used together. Meter readout is used only infrequently (for body temperature, for example). In many laboratories, the output is digitized for further data analysis. For this study, it is assumed that the Space Station will be equipped with analog-to-digital converters and a general-purpose digital computer.

8.1.1 Biopotential Recordings

Biopotential measurements are the most common (and diverse) of electrphysiological recordings. A voltage signal related (often quite indirectly) to some physiological function is recorded by conventional electronic methods. The amplifier input is coupled to the animal through an electrode specialized for the type of measurement and the region from which it is recorded. Probably only biopotential skin electrodes will be used on the human subjects. Functionally, these are half cells with a conductive gel between the Ag-AgCl disc and the skin. Implanted electrodes and microelectrodes may be required in some of the animal studies; and these would generally be prepared by the scientist to meet his individual needs.

Although specialized instruments are made for many biopotential measurements, the same results can usually be achieved with general-purpose electronic equipment. The amplifier needed for biopotential recordings varies only slightly with different types of measurements. The general range of characteristics are included in the following: input impedance, one to ten megohms

(100 megohms for microelectrodes); sensitivity to detect signals of millivolts to tens of microvolts; a band pass of 1 to 50 Hz is adequate for most uses, but extreme conditions may include dc to 2 kHz. The amplifier output must drive a galvanometer when a strip-chart recorder is used. (General characteristics of recorders are considered in Section 21).

8.1.2 Bioimpedance Recordings

Bioimpedance recordings show the measurement of changes in the electrical impedance of a tissue segment. The change in impedance results from blood or air entering or leaving the tissue segment. In impedance pneumography, the impedance of the thorax increases during inspiration, while for impedance plethysmography, the impedance of a limb decreases as blood flows into it. Generally, the resistive component of the impedance is measured, although to some investigators the capacitive component is of special interest.

The electronic apparatus necessary to measure bioimpedance signals is considerably more specialized than that used for biopotential recordings. A high-frequency oscillator injects a signal into the preparation. The signal is then detected and demodulated to produce a signal proportional to the tissue impedance. When a 4-electrode system is used, tissue impedances are found in the range of 10 to 100 ohms. The impedance pulsations associated with local blood flow are as low as tenths of ohms. Following demodulation of this impedance signal, a standard amplifier and recorder can be used.

8.1.3 Transducer Recordings

There are several biological phenomena which are detected as mechanical forces or displacements. These are detected by transducers specialized for biological use. Some of these transducers produce an electric signal directly, such as the microphone for phonocardiology. In others, such as blood-pressure transducers, a resistance change must be detected by a bridge circuit. There is currently an increasing number of other transducer techniques being applied to biomedical measurements such as ultrasonic and magnetic measurements, for example. These transducers require their own specialized signal conditioners to match the techniques used and phenomena measured to a standard amplifier and readout.

The category of transducer recordings also includes measurement of body temperature with thermistors. A procedure for making weighted multiple body surface measurements has been proposed for IMBLMS. A made-to-order resistor network would be needed for this, but standard sensors and amplifiers and readout would be adequate for the remainder.

8.2 APPLICATIONS

Electrophysiological recordings are used principally for neurological, behavioral, and cardiovascular measurements on both human subjects and experimental animals. They are particularly called for in the following functional program elements (FPE's):

5.9 Small Vertebrates (Bio D)

5.13 Biomedical and Behavioral Research

5.14 Man/System Integration

5.23 Primates (Bio A)

5.26 Invertebrates (Bio F)

Table 8-1 shows the interrelation between signal conditioners, applications, signal ranges and characteristics, and transducers.

8.2.1 Neurological Applications

The electroencephalogram (EEG) samples aspects of the electrical activity of the brain as detected on the scalp. It is used to assess states of consciousness (sleeping/waking) and a few abnormal neurological conditions. The EEG, together with the electromyogram (EMG) of the neck muscles and the electrooculogram (EOG), are used to assess sleep states and levels of consciousness. For evoked response studies, a signal-averaging device will be needed for the EEG to show the average response in a short time epoch following repeated stimuli. The use of evoked responses in electroencephalography is gaining acceptance and must be included for future plans.

The electrooculogram records movement of the eyes. It is useful in both sleep studies and also human factors research. The first derivative of the EOG can be taken to produce a record of the velocity of eye movements.

The electromyogram is a recording of electrical activity associated with muscular contraction, and is useful on human subjects and experimental animals. The electromyogram signal is often conditioned by an integrating circuit to produce an envelope of muscular activity, rather than the high-frequency bursts normally observed.

Application	Primary Signal Ranges and Characteristics	Transducers Required (Beckman — and Others, as Indicated)	Applicable 9800 Series Input Coupler(s)★
Cardiovascular System			
Blood pressure direct method arterial, large animals	Frequency range: dc to 200 Hz; dc to 60 Hz usually adequate Pressure range, arterial· 40 to 300 mm Hg	Strain gage pressure transducer (Statham P23AA).	9872, 9803, 9853
Blood pressure, direct method, arterial, small animals	Frequency range· dc to 200 Hz; dc to 60 Hz usually adequate	Strain gage pressure tranducer (Statham P23DB)	9872, 9803, 9853
Pulse waves, direct method, arterial	Frequency range dc to 200 Hz, dc to 60 Hz usually adequate	Strain gage pressure transducer (Statham P23 Series)	9803 or 9853
		LVDT pressure transducers	9805B
Pulse waves, indirect method, peripheral artery	Frequency range. 0 1 to 60 Hz usually adequate Pulse trace similar to Blood pressure, direct, but without baseline zero	Beckman Signal Divider, Infraton Pickup (Spinco) (Photocell)	9806A or 9808A (9874 with photocell)
		Other detectors	As required*
Phonocardiogram	Frequency range 5 to 2000 Hz, major diagnostic components lie in 20 to 200 Hz range.†	Crystal, condenser, or magnetic microphone Marco Phonocardiography Microphone (Type 5108).	9806A, 9801, 9802, or 9808A
Plethysmogram (volume measurements)	Frequency range: dc to 30 Hz	Plethysmographs· pressure changes (w/pressure transducer), circumferential changes (w/Mercury Gage), impedance changes (w/impedance plethysmogram)	As required*
Ballistocardiogram	Frequency range: dc to 40 Hz	Infinite period platform with — strain gage accelerometer (Statham A4)	9803, 9853
		— other transducer.	As required*
Heart rate	Average rate, human. 45 to 200 beats/min, lab animal. 50 to 600 beats/min	A computed function, commonly derived from the ECG signal	9857, 9857B (rate dir.), 9854 (rate avg)
		May also be derived from other pulsatile body signals, such as a differentiated pulse wave	As required*
Blood pressure, direct method, venous	Pressure range: 2 to 20 mm Hg	Strain gage pressure transducer (Statham P23BB).	9872, 9803, 9853
Cardiac output	Frequency range: 0 to 60 Hz; 0 to 5 Hz usually adequate	Dye dilution cuvette and amplifier (Spinco Cardio-Densitometer)	9806A, or 9801
Blood pressure, indirect method	Frequency range. dc to 200 Hz, dc to 60 Hz usually adequate. Pressure range· 60 to 300 mm Hg	Sphygmomanometer cuff and pressure transducer (Statham P23AA).	9872, 9803, 9853
Electrocardiogram	Frequency range: 0 05 to 80 Hz Signal range· 10 μV to 5 mV includes fetal range.	Direct recording from Beckman Biopotential Skin Electrodes	9855, 9856, 9806A, 9857, 9857B, 9854, or 9853
Respiratory System			
Flow rate (pneumotachogram)	Frequency components to 40 Hz Normal range: 250 to 500 ml/s; maximum 8 l/s	Fleisch pneumotachograph head with strain gage transducer (Statham PM).	9803, or 9853
		Fleisch pneumotachograph head with LVDT transducer.	9805B
Breathing rate calculated from record (with approximate relative respiratory volume)	Average rate: human, 12 to 20 breaths/min, lab animal, 8 to 60 breaths/min	Strain gage belt (Beckman Type 7001, or other), or mercury-filled rubber tubing	9803, or 9853
		Thermocouple	9827, 9806A, 9801, or 9853
		Thermistor (Yellow Springs Type 511 mounted in flow stream)	9858
		Impedance pneumograph	As required*
Tidal volume (measured per breath or integrated to provide volume per min)	Typical volume, adult human 600 ml/breath, 6 to 8 l/min.	Integral of unidirectional flow only, derived from: — Fleisch pneumotach, head with strain gage transducer (Statham PM Series) — Spirometer with appropriate transducer (Gilford 130 system).	Combination of two channels — 9803 and 9873B
		Flow plus integral of unidirectional flow, when 2-channel readout desired. — Fleisch pneumotach, head with strain gage transducer (Statham PM Series).	9803, 9853 plus 9873B
		— Fleisch pneumotach, head with LVDT transducer	9805B plus 9873B

†Frequency components to 200 Hz can be recorded on all high sensitivity Dynograph recorders; components above these levels require oscilloscope, tape, or optical galvanometer readout

*Choice of coupler depends on transducer used Check transducer output specifications; then select coupler

★9800 Series Input Couplers for use with high sensitivity Dynograph Recorders; Types R, Biomedical R, RS, RP, R-2005, S-II Types KM, T, TC, and TD are complete recorders without couplers.

Table 8-1 (Sheet 1 of 2). Physiological Measurement Guide

Application	Primary Signal Ranges and Characteristics	Transducers Required (Beckman — and Others, as Indicated)	Applicable 9800 Series Input Coupler(s)★
CO_2, N_2O, or halothane concentration in respired air	Normal range, CO_2 0 to 10%, end-tidal CO_2, human, 4 to 6% N_2O 0 to 100% Halothane 0 to 3%	Beckman LB-1 Medical Gas Analyzer amplifier with appropriate pickup head (Spinco).	9806A
Diffusion of inspired gas (using nitrogen)	Normal range of nitrogen concentration differential 0 to 10%	Nitrogen analyzer	9856, 9806A, or 9801
Pulmonary diffusing capacity (using carbon monoxide)	Normal range, human 16 to 35 mlCO/mmHg/min	Beckman 15-A or 215 Infrared Analyzer	9856, 9806A, or 9801
Dissolved Gases and pH			
Partial pressure of dissolved O_2, in vivo or in vitro	Frequency range dc to 1 Hz usually adequate Normal measurement range 0 to 800 mmHg Po_2. Hyperbaric Po_2 range 800 to 3000	Direct recording from Beckman polarographic electrode (Spinco).	9871
		Beckman 160 Physiological Gas Analyzer amplifier with electrode (Spinco).	9806A, or 9801
pH, in vitro	Signal range 0 to ±700 mV covers pH range	Beckman pH Meter with electrode	9806A, or 9801
Partial pressure of dissolved CO_2, in vitro	Normal signal range: 0 to ±150 mV covers range from 1 to 1000 mmHg Pco_2	Beckman 160 Physiological Gas Analyzer amplifier (Spinco) with electrode	9806A, or 9801
Bioelectric Potentials			
Electroencephalogram	Frequency range dc to 100 Hz, major diagnostic components lie in 0 5 to 60 Hz range Normal signal range 15 to 100 μV	Direct recording from Beckman Biopotential Skin Electrodes, or from silver disc electrodes	Types T, TC, EEG Recorders; 9856, 9806A, (Lead selector panel optional)
Cerebral potentials, intracranially recorded	Normal signal range 10 μV to 100 mV. Pulse duration 0 6 ms to 0.1 s	Direct recording from needle or micro electrodes (Recorder response time limits the accuracy of traces for pulses of less than 3 ms duration)	9856, 9806A, or (for micro electrodes) 9808A
Electromyogram (primary signal)	Frequency range 10 to 2000 Hz † Pulse duration 0 6 ms to 20 ms.	Direct recording from Beckman Bipotential Skin Electrodes or needle electrodes (Recorder response time limits the accuracy of traces for pulses of less than 3 ms duration)	9852 (direct)
Electromyogram (averaged)	An average of the primary signal, after full wave rectification.	Derived from direct recording from Beckman Biopotential Skin Electrodes or needle electrodes	9852 (integrate)
Smooth muscle potential (e g , electrogastrogram)	Frequency range dc to 0 6 Hz Normal signal range 0 5 to 80 mV.	Direct recording from electrodes	9856, or 9806A
Electroretinogram	Frequency range: dc to 20 Hz adequate. Normal signal strength: 0 5 μV to 1 mV	Direct recording from corneal electrodes.	9856, or 9806A
Electrocardiogram	(See listing under Cardiovascular System)		
Electronystagmogram	Direct. Frequency range, 0 to 20 Hz Typical signal strength, 100 μV/10° eye movement Derivative or Velocity* Frequency range, 0 to 20 Hz. Signal derived from direct reading.	Beckman Biopotential Skin Electrodes	9859 (direct), 9841 (velocity); Type KM Recorder
Physical Quantities			
Temperature	Full range of signals	Thermistor (Yellow Springs 400 Series).	9858
		Thermocouple	9827, 9806A, or 9801
		Resistance thermometer.	9803, or 9853
Skin Resistance (GSR)	Resistance range: 1 kΩ to 500 kΩ	Direct recording from Beckman Biopotential Skin Electrodes	9892A
Isometric force, dimensional change, body fluid and body cavity pressures	Full range of signals.	Strain gage (resistance or semiconductor). (See Statham Instruments)	9803, or 9853

†Frequency components to 200 Hz can be recorded on all high sensitivity Dynograph recorders, components above these levels require oscilloscope, tape, or optical galvanometer readout

*Choice of coupler depends on transducer used Check transducer output specifications, then select coupler

★9800 Series Input Couplers for use with high sensitivity Dynograph Recorders, Types R, Biomedical R, RS, RP, R-2005, S-II Types KM, T, TC, and TD are complete recorders without couplers.

Table 8-1 (Sheet 2 of 2). Physiological Measurement Guide

8.2.2 Cardiovascular and Pulmonary Applications

The electrocardiogram is the most common of all electrophysiological measurements. For the clinical ECG, a standard set of leads is attached to the arms and legs (sometimes across the chest), switching between leads being done in the preamplifier. The standard ECG is usually written on a strip-chart recorder, although it may also be displayed on a CRT display. The latter display may require some reorientation by the physician or scientist reading it. With a cardiotachometer attachment, the heart rate can be displayed as an analog signal or meter reading, using the standard ECG electrodes.

The vectorcardiogram (VCG) records the ECG signals from an array of electrodes on the thorax. The signals from the individual electrodes of the array are mixed and weighted in a resistor network. The output of two channels of the network are displayed orthogonal to each other as Lissajous figures on a CRT display. This type of display approximates a real-time, two-dimensional projection of the electrical activity of the heart.

Impedance measurements of cardiovascular functions are gaining increasing clinical and experimental use. The impedance plethysmogram records the volume changes in a limb segment as arterial blood-flow pulses to it and venous blood drains from it. The impedance cardiogram (ZCG) records a complex (not yet fully analyzed) pattern of impedance changes of the thorax which are associated with the thoracic blood flows during the cardiac cycle. The impedance pneumogram (ZPG) also uses an impedance technique to measure changes in thoracic

volume associated with ventilation. The impedance changes of the thorax associated with ventilation are considerably larger than those associated with the cardiac cycle.

Measurement of blood pressure with electrophysiological recording equipment typically involves arterial puncture, insertion of a cannula, and connection to a pressure transducer. Automated indirect (noninvasive) methods for recording of arterial blood pressure have been generally unsuccessful. One partially automated indirect method uses a cuff and microphone to automate the clinical method for making blood pressure measurements. As the cuff is automatically inflated, the pressure from the cuff is written on a strip-chart recorder and the Korotkoff sounds are superimposed on the same trace.

The phonocardiogram (PCG) and vibrocardiogram (VbCG) use a microphone or accelerometer to record sounds or movements of the chest wall overlying the heart. The PCG and VbCG are used for timing the cardiac cycle, indicating the opening and closing of the heart valves.

Several other less common cardiovascular measurement techniques are also amenable to electrophysiological recording methods. These include: magnetic and ultrasonic blood-flow measurement, plethysmography, peripheral venous pulse, and oximetry. Each of these diverse techniques requires specialized coupling with the recording system.

The methodology of electrophysiological recordings is also appropriate for pulmonary function recordings. These include impedance pneupography (ZPG), oximetry, and recording the output of gas analysis and flow instruments.

8.2.3 Behavioral Applications

Electrophysiological recording equipment is applicable to a wide variety of behavioral recording needs. Among these are: behavioral events, stimulus-response relations, and physiological correlates of behavior. Most of the neurological measurements (8.1) and cardiovascular and pulmonary measurements (8.3) could be of interest either as behavioral indices or as physiological correlates of behavior. In addition, the galvanic skin response (GSR) is a frequently-used behavioral measure. Given a few basic dimensions of electrophysiological instrumentation, the diversity of measurements appropriate for behavioral research is limited only to the imagination of the experimentor.

8.3 LOGISTICS

Despite the diversity of types and applications of electrophysiological recordings, the actual equipment involved can be considered as one general type, so far as logistics are involved.

8.3.1 Packing and Launch

Electrophysiological recording equipment is essentially rather conventional electronic equipment. Standard packing procedures should be adequate to meet launch requirements. For transport by space shuttle, these should not differ markedly from techniques currently used by major manufacturers for railway shipment.

8.3.2 Installation

Electrophysiological recording equipment requires electric power. Individual items should be rigidly mounted; many items are available for mounting in standard 19-inch instrument racks. Some protection is needed to avoid personnel contact with protruding knobs, sharp corners, or heated writing styli.

8.3.3 Consumable Supplies

Electrophysiological recording equipment uses few consumable supplies. Electrode paste (or perhaps disposable electrodes) are needed for most biopotential and bioimpedance recordings. If the output is to be by strip-chart recorder, considerable paper will be needed. Since heat pens and other nonfluid writing methods are available for electrophysiological recording equipment, ink writing is not recommended.

8.3.4 Accessories and Spare Parts

Many accessories are available for electrophysiological recording equipment. Electrodes and electrode attachment devices are needed for bioimpedance and biopotential recordings. Connecting cables are needed for attaching the electrodes to the equipment and for making interconnections between different parts of the equipment. Selection must be made from the wide variety of transducers and signal conditioners available to support the experiments planned.

Another category of accessories are instruments which, although they could stand alone, are generally used together with electrophysiological recording equipment. These instruments include stimulators, pulse and waveform gener-

ators, and oscilloscope cameras. These instruments are often used together to coordinate presenting the stimuli and collecting the response.

Most spare parts for electrophysiological recording equipment will be standard electronic components. Where permitted by instrument design, repairs should be made by replacement of plug-in printed circuit boards. Spare parts should also include pens, fuses, and extra cables.

8.3.5 Maintenance and Repair

Relatively little maintenance or repair is required of electrophysiological recording equipment. Maintenance should involve little more than occasional calibration and balancing procedures, since failure of electronic components is infrequent. Possible repairs should involve substitution of instruments and subassemblies rather than exhaustive troubleshooting and replacement of components. Maintenance and repairs can all be performed by technical or professional-level personnel trained in electronics.

8.4 OPERATION

8.4.1 Warm-up and Speed-of-Operation

For solid-state electronics, little or no warm-up time is needed except when highly stable long periods of dc recording are to be used.

Some set-up time is needed for electrophysiological recordings. This set-up includes application of the sensing electrodes and transducers, connection of input cables, adjustments of gain and band pass calibrations, and setting baselines and balances. After initial set-up is complete, electrophysiological

recordings are real-time recordings of the experimental events; recordings are concurrent with experimental procedures. When periodic samples of electrophysiological activity are taken, samples of a few seconds to a few minutes are generally adequate.

8.4.2 Operation Skills

Operation of electrophysiological recording equipment is possible by technical or professional-level personnel with previous experience in such measurements. Some pretraining is needed for inexperienced personnel. Interpretation of the recordings requires professional personnel with experience in this area.

8.4.3 Operating Procedure

Because of the diversity of types of electrophysiological measurements, only the highlights of the measurements can be summarized here.

- Apply sensors.
- Connect to recording equipment.
- Adjust gain, baseline, band pass, etc.
- Record.

8.4.4 Sample Preparation and Handling

The samples involved in electrophysiological recordings are electric signals; no preparation is needed. Human subjects can be handled according to normal clinical procedures. Some care is needed in handling experimental animals. They should be tethered when not in their cages.

8.5 INTERFACE

8.5.1 Interface with Other Laboratory Instruments

Electrophysiological recording equipment can be used in conjunction with many other analytical laboratory instruments which provide a proper output. Some experiments may require the coordinated use of several instruments using the electrophysiological recording as a common time base for several measurements. The output of blood gas analyzers, oxygen analyzers, or infrared analyzers, for example, might be used with cardiovascular/pulmonary measurements.

8.5.2 Interface with Vehicle Systems

When used in direct off-the-shelf configurations, electrophysiological recording equipment has only minimal dependence on vehicle systems. Power is needed; some heat must be dissipated; and the instruments must be mounted or otherwise held in place. In this configuration, hard copy would be produced on-line with strip-chart recorders.

In an intermediate configuration, the electrophysiological recording equipment could share recording equipment (strip chart and magnetic tape) with other laboratory instruments. In either of these cases, an oscilloscope could monitor recordings when hard copy was not being made and recording of high-speed phenomena accomplished by photography.

An advanced interface would use the on-board data management system for digitizing and storing electrophysiological recordings. Ideally, this system could provide CRT displays and hard copy on request. A more advanced data management system could also control experiments and analyze results.

8.6 SAFETY

8.6.1 Flame Hazards

There are essentially no flame hazards associated with electrophysiological recording equipment. Even heated writing styli do not present a hazard in a two-gas environment.

8.6.2 Microbiological Hazards

The only possible microbiological hazards involved in electrophysiological recording situations are transfer of microorganisms on the electrodes and culture growths in the electrode pastes. Sterilization of electrodes between uses and bactericidal agents in the electrode paste would avoid these sources of contamination. Use of prepackaged, disposable electrodes would also avoid contamination.

8.6.3 Electromagnetic Interference

Some electrophysiological amplifiers contain 400 Hz mechanical choppers. These choppers might require some shielding to avoid interference with surrounding instruments. Several electrophysiological measurements require high-impedance sources which in themselves are sensitive to electromagnetic fields. Shielding of the preparation is often necessary for terrestrial measurements and may also be necessary for Space Station situations.

8.6.4 Ionizing Radiation

Electrophysiological recording equipment neither produces nor is sensitive to ionizing radiation.

8.6.5 Physical Hazards to Personnel

The principal physical hazards of electrophysiological recording equipment in the nonoperating mode are protruding knobs and sharp corners. When operating, heated styli could be hazardous, but they are generally protected from accidental handling.

During operation of the recording apparatus or peripheral equipment, the subject must be protected from electric shock. National standards for patient safety are to be expected soon. These standards should set limits for acceptable current leakage into the subject. Although many instruments available in the early 1970's cannot meet these new requirements, instruments designed for sale in the mid 1970's should exceed all Space Station requirements. The current procedure of grounding the patient is being discontinued, and the following design techniques will produce far safer instruments: maximum use of isolated inputs, adequate equipment grounding, use of current limiters in patient circuits, use of double insulation, and use of isolated power supplies.

8.7 MODIFICATIONS

No major modifications, beyond rigid mounting and protection from knobs and corners, are needed for use of electrophysiological equipment in a zero-g environment.

For most effective operation, electrophysiological recording equipment would utilize the space station data management system to record, store, and analyze data and to produce hard copy as needed. Lacking this capability, magnetic tape and/or strip-chart recorders would be needed.

8.8 AVAILABLE INSTRUMENTS

Although many specialized instruments are available for recording one or a few physiological variables, only general-purpose instruments with modular operation are recommended for use in a space-station environment. The three major manufacturers of modular instrumentation are Grass Instrument Company (Polygraphs), E and M Instrument Company (Physiographs), and Beckman Instruments, Inc. (Dynographs). The modular signal conditioners and their specifications for these three lines of instruments are shown in Table 8-2.

If a standard chart recorder (Section 21) is to be used rather than hard copy from the data management system, it should use heat writing pens (or some other nonink system), and it should have a roll paper take-up device rather than fan-fold paper stacking.

GRASS INSTRUMENTS COMPANY	POLYGRAPH PLUG-IN UNITS				
Preamp/Signal Conditioner Model or Type No.	Frequency Response	Sensitivity	Input Impedance(1)	Applications	Remarks
Low-level dc Preamplifier Model 7P1	Dc - 47 Hz Variable	10 μV/cm	1	Strain gage, pressure transducer, thermocouple, polarographic O_2, GSR in ac mode, EEG, ECG	Built-in GSR mode with injection to 50 μA current
Wideband ac Preamplifier and Integrator - Model 7P3	0.15 - 5K Hz	25 μV/cm	3	EEG, ECG, EMG	Auxiliary IRIG output. Integrator option with variable time constant. Accepts cathode follower input probe accessory.
Tachograph Preamplifier Model 7P4	0.5 - 5K Hz	50 μV/cm	10	EKG, Cardiotachometer	Internal switching of ECG leads External calibration needed for tachometer
Wideband ac EEG Preamplifier Model 7P5	0.15 - 35K Hz	20 μV/cm	3	EEG, ECG, EMG	Cathode follower input probe available as accessory
EKG Preamplifier Model 7P6	0.04 - 50 kHz	31 mV/cm	10	ECG	Internal switching of ECG leads
Circuit control for Differential Transformer Transducers, Model 7P7	Dc	NA	B(2)	Blood flow meter transducer	
Sphygmomonometer Preamplifier Model 7P8	Unk	Unk	Unk	ECG, pulse plethysmograph indirect blood pressure	Internal switching of ECG leads; pulse plethysmograph needs photoelectric transducer; indirect blood pressure requires pressure cuff and microphone
Photocell Transducer Matching Panel, Model 7P9	Unk	Unk	Unk	Pulse plethysmograph; drop counting	Adjustable time constants
E AND M INSTRUMENTS COMPANY			PHYSIOGRAPH MODULES		
Channel Amplifier	Dc - 16K Hz		See following preamplifier impedance	NA	Uses separate preamplifiers
Recording Channel, Servo Sv-4H or SV-6H	Dc - Hz	1 mV/cm	1	pH meter, dye tracer, gas analyses, thermometer	Servo drives recording pen across entire channels of chart space
Cardiac Preamplifier, 93-100-70	0.1 - 2K Hz	1 mV/cm	6.6	ECG and other biopotentials	Separate preamplifier. No lead switching
Carrier Preamplifier, 93-500-70	Dc	NA	B	For use with pressure transducers strain gage, thermistor bridge, etc.	5 kHz oscillation
Dc Preamplifier, 93-600-71	Dc - 15 kHz	1 mV/cm	2	Biopotential or instrument output	
Ac-dc Preamplifier, 93-600-71	Dc - 15 kHz	1 mV/cm	2	Biopotential, transducers, or instrument output	
GSR Preamplifier, 93-700-70	Dc	300 Ω/cm	B	Galvanic skin response	20 μA current injected
Hi-gain Preamplifier, 93-300-70	0.1 - 12 kHz	30 μV/cm	6.6	ECG, EEG, EMG	
Cardiotachometer, 93-400-70	0 - 500 beats/min	-	-	Heart rate measurement	Used with cardiac preamplifier

(1) Megohms, unless otherwise specified

(2) Bridge type input

Table 8-2. Electrophysiological Signal Conditioners (Sheet 1 of 3)

BECKMAN INSTRUMENTS, INC.	DYNOGRAPH COUPLERS				
Preamp/Signal Conditioner Model or Type No.	Frequency Response	Sensitivity	Input[1] Impedance	Applications	Remarks
Straight-through Coupler ac/dc, Type 9801	Dc - 150 Hz	1 μV/mm	2	Dc or low frequency ac signals, thermocouple output	Presents input directly to preamplifier
Straight-through Coupler ac Type 9802	20 Hz - 5 kHz	1 μV/mm	2	For high-frequency signals	For use with fast frequency response output devices
Strain Gage Coupler Type 9803	Dc - 150 Hz	1 μV/mm	B	Strain gage, 90 - 1000 Ω range	6 V dc excitation. Balance and calibration controls
Reluctance Gage Coupler Type 9804	Dc - 50 Hz	1 μV/mm	B	Reluctance gage coupler	Resistance, reactance, and phase balance controls
Differential Transformer Coupler, Type 9805, 9805B	Dc - 50 Hz	1 μV/mm	B	Differential transformer coupler	
Ac/dc Input Coupler Type 9806A	Dc - 200 Hz Variable	1 μV/mm	2	EEG, EMG, ECG	General-purpose input counter for biopotential and other inputs
Electrometer Coupler Type 9808A	Dc - 150 Hz	0.5 mV/cm	10,000	Microelectrode and other high-impedance sources	
Input and Amplifier Selector Coupler, Type 9809	Dc - 150 Hz	1 μV/cm	1	Switching of different inputs onto same readout channel	Connects to dc or ac preamplifier or direct to power amplifier
Integrating Strain Gage Coupler, Type 9825	Dc - 150 Hz	1 μV/mm	Bridge	Coupler strain gage transducer (range 90 - 1000 Ω)	Integrate output with variable time constant
Integrating Coupler Type 9826	Dc - 20 Hz	40 mV/sec/cm	NA	Integrate ac-dc voltage signal	Integrate or direct operation zero suppression, reset button
Nystagmus Velocity Coupler, Type 9841	Dc - 16 Hz	NA	2	EOG	Differentiates output of 9859 coupler to give signal proportional to velocity of eye movement
EMG Integrator Coupler Type 9852	NA	NA	NA	EMG and integration	Gives direct or integrated EMG
Strain Gage and Straight-through Coupler, Type 9853	Dc - 150 Hz	1 μV/mm	2, B	Strain gage coupler or straight-through potential recording	Controls: sensitivity, balance, polarity, calibrate, direct/bridge
Heat Rate Coupler Type 9854	300 b/min to 400 b/min	Peak ≥ 0.2 mV	Unk	ECG and rate	Has audio monitor; select, direct/rate, and sensitivity adjustments
Electrocardiogram Input Coupler, Type 9855	Dc - 150 Hz	1 μV/mm	2	ECG	Internal switching of ECG leads
EEG lead Selector Input Coupler, Type 9856	Dc - 150 Hz Variable	1 μV/mm	2	EEG, ECG, EMG	Select pairs of 11 leads and ground Select bandpass
Cardiotachometer Coupler Type 9854	30 - 240 beats/min	1 mV/peak	Unk	Heart Rate	Internal calibration frequencies range and centering control
Cardiotachometer Coupler Type 9856 B	30 - 960 beats/min	1 mV/peak	Unk	Heart Rate	Internal calibration frequencies range and centering control
Thermistor Coupler Type 9858	Dependent on Probe	.00025°C/mm	B	Temperature	For use with Yellow-Spring Instrument Co. Series 400 probes (range 1020 - 3300 Ω)

(1) Megohms, unless otherwise specified

Table 8-2. Electrophysiological Signal Conditioners (Sheet 2 of 3)

BECKMAN INSTRUMENTS, INC.

DYNOGRAPH COUPLERS

Preamp/Signal Conditioner Model or Type No.	Frequency Response	Sensitivity	Input[1] Impedance	Applications	Remarks
Straight-through Coupler ac/dc, Type 9801	Dc - 150 Hz	1 μV/mm	2	Dc or low frequency ac signals, thermocouple output	Presents input directly to preamplifier
Straight-through Coupler ac Type 9802	20 Hz - 5 kHz	1 μV/mm	2	For high-frequency signals	For use with fast frequency response output devices
Strain Gage Coupler Type 9803	Dc - 150 Hz	1 μV/mm	B	Strain gage, 90 - 1000 Ω range	6 V dc excitation. Balance and calibration controls
Reluctance Gage Coupler Type 9804	Dc - 50 Hz	1 μV/mm	B	Reluctance gage coupler	Resistance, reactance, and phase balance controls
Differential Transformer Coupler, Type 9805, 9805B	Dc - 50 Hz	1 μV/mm	B	Differential transformer coupler	
Ac/dc Input Coupler Type 9806A	Dc - 200 Hz Variable	1 μV/mm	2	EEG, EMG, ECG	General-purpose input counter for biopotential and other inputs
Electrometer Coupler Type 9808A	Dc - 150 Hz	0.5 mV/cm	10,000	Microelectrode and other high-impedance sources	
Input and Amplifier Selector Coupler, Type 9809	Dc - 150 Hz	1 μV/cm	1	Switching of different inputs onto same readout channel	Connects to dc or ac preamplifier or direct to power amplifier
Integrating Strain Gage Coupler, Type 9825	Dc - 150 Hz	1 μV/mm	Bridge	Coupler strain gage transducer (range 90 - 1000 Ω)	Integrate output with variable time constant
Integrating Coupler Type 9826	Dc - 20 Hz	40 mV/sec/cm	NA	Integrate ac-dc voltage signal	Integrate or direct operation zero suppression, reset button
Nystagmus Velocity Coupler, Type 9841	Dc - 16 Hz	NA	2	EOG	Differentiates output of 9859 coupler to give signal proportional to velocity of eye movement
EMG Integrator Coupler Type 9852	NA	NA	NA	EMG and integration	Gives direct or integrated EMG
Strain Gage and Straight-through Coupler, Type 9853	Dc - 150 Hz	1 μV/mm	2, B	Strain gage coupler or straight-through potential recording	Controls: sensitivity, balance, polarity, calibrate, direct/bridge
Heat Rate Coupler Type 9854	300 b/min to 400 b/min	Peak ≥ 0.2 mV	Unk	ECG and rate	Has audio monitor; select, direct/rate, and sensitivity adjustments
Electrocardiogram Input Coupler, Type 9855	Dc - 150 Hz	1 μV/mm	2	ECG	Internal switching of ECG leads
EEG lead Selector Input Coupler, Type 9856	Dc - 150 Hz Variable	1 μV/mm	2	EEG, ECG, EMG	Select pairs of 11 leads and ground Select bandpass
Cardiotachometer Coupler Type 9854	30 - 240 beats/min	1 mV/peak	Unk	Heart Rate	Internal calibration frequencies range and centering control
Cardiotachometer Coupler Type 9856 B	30 - 960 beats/min	1 mV/peak	Unk	Heart Rate	Internal calibration frequencies range and centering control
Thermistor Coupler Type 9858	Dependent on Probe	.00025°C/mm	B	Temperature	For use with Yellow-Spring Instrument Co. Series 400 probes (range 1020 - 3300 Ω)

(1) Megohms, unless otherwise specified

Table 8-2. Electrophysiological Signal Conditioners (Sheet 3 of 3)

Section 9
EMISSION SPECTROMETERS

9.1 PRINCIPLES OF OPERATION

Quantitative emission spectroscopy is used to determine the constituent elements in minerals, metals and alloys, and, in some instances, for metallic impurities in organic compounds. An emission spectrometer consists of a split dispersing element and a detector, plus a source of excitation for thermally exciting the sample. A detector is needed for measuring the absolute or relative amount of radiation corresponding to each wavelength.

The source or excitation unit can be an arc, spark, gas discharge, or flame system that causes the sample to emit radiation. The energy or radiation analyzed by the spectrometer is detected and measured by two basic methods. The first of these is photography where the exposure, development, and calibration of special film plates and the measurement of the resulting image line densities is made with a photoelectric densitometer. These density readings are then used to calculate actual sample concentration by reference to a simultaneously analyzed standard. The other alternative is direct radiometry where the electrical output of a scanning detector or the outputs of multiple detectors from photocells or photomultiplier tubes can be directly related to the radiation at the wavelength of interest. Sample concentrations may be calculated from these signals or, in some cases, be made to read out directly in concentration units.

9.1.1 Spectra Excitation

An atom of a chemical element can be pictured as a small, compact nucleus consisting of protons and neutrons surrounded by a number of orbital electrons. Each electron carries a unit of negative charge equal and opposite in size to the proton. The neutron is electrically neutral. The neutral atom is one in which the number of protons equals the number of electrons. This number is often called the atomic number, and is characteristic of each element.

The electrons of each of the neutral atoms are located in orbits which are associated with discrete energy levels. When an undisturbed atom has electrons occupying orbits of lowest energy, it is said to be in the ground state.

When the undisturbed atom is excited by fast-moving particles (such as atoms, ions, molecules, electrons, or photons), the outer electrons, which are held more loosely, jump to higher energy levels. This unstable condition causes the electrons to subsequently drop down to their original orbits, and in this process they emit electromagnetic energies equal to the energy difference between the excited orbit and the ground orbit. It is this energy loss which produces spectral lines. Since the electron can return to its original orbit in either one or a series of jumps, either one or a number of spectral lines may be produced as a result of this process. Ions may also undergo the same increase and subsequent loss of energies so that they can emit a spectrum similar to that occurring in neutral atoms. The difference is that a higher energy source such as a spark is required to excite the ions as compared with the relatively low voltage dc and ac arcs required for neutral atoms. Because a neutral atom and an ion have slightly different energy configurations, their spectra are

different with respect to the emitted line intensities. Most lines emitted as a result of dc arc excitation are from the neutral atom; hence, neutral atom lines are generally referred to as arc lines while those emitted by ions are called spark lines.

9.1.2 Excitation Methods

When an element is burned in an arc, spark, or other source, the excitation spectrum that is emitted as a result of the energy transition provides a fingerprint for that particular element. Each element in the periodic table has a fingerprint spectrum which can be uniquely identified. Emission spectroscopy, then, is that process based on analyzing the chemical composition of an unknown material by identification of the spectra produced by excitation of the unknown material.

A number of methods are used for the excitation of elements to obtain this fingerprint spectra for analysis. These methods are reviewed here as the most important aspect of a spaceborne instrument, since the power consumptions involved are sufficiently high to be a limiting factor in the utilization of emission spectrometry.

9.1.2.1 Direct Current

Normally, this type of arc is operated from a dc supply line of 110 to 220 V, or from a rectified ac line. Arc currents in the range from 2 to 20 amperes are normally used with approximately 35 to 80 V across the analytical gap. The remainder of the voltage drop is through a series resistance or inductance coil to stabilize the voltage-current characteristic of the arc. The dc arc method

has very high sensitivity, and is used primarily for determining low concentrations of elements. It is particularly useful in powdered samples.

9.1.2.2 Alternating Current

In this method, an ac discharge of 1000 or more volts with currents from 0.5 to 5 amperes is used. The voltage supply at 60 cycles is connected through a transformer across a large ballast resistance or inductance in series with a 2 to 3-mm electrode gap. This source is primarily used for the analysis of alkaline solutions and alkali salts where the arc can be self-sustaining. For most other materials, the low voltage arcs must be reinitiated each half-cycle by a high frequency discharge. The ac arc does not have as high a sensitivity as the dc arc method or the high-voltage spark.

9.1.2.3 High-Voltage Ac Spark

There are several variations found for high voltage ac spark systems. The first technique is a simple condensed spark. Another common arrangement is a spark circuit in which the analysis gap is triggered by an auxiliary rotating spark gap which enables the discharge to take place near the peak of the secondary voltage. In this variation, one or two spark discharges normally occur during each half-cycle of the alternating current.

Another method is a circuit in which the analysis gap is triggered by an auxiliary spark gap quenched by a flowing air path which gives positive assurance that the spark will break down at constant voltage. This technique has the advantage that the number of breakdowns can be altered from 1 to as many as 20 or 25 discharges per half-cycle. Different parameters of capacitance,

inductance, and secondary resistance are selected depending on whether metals, alloys, or powdered samples are being analyzed. This spark method has relatively poor sensitivity, but has a high degree of reproducibility. Because of these factors, this method is usually the preferred technique for analysis of metallic alloys.

9.1.3 Dispersing Methods for Emission Spectrometers

The key to analysis-by-emission spectrometry is the dispersing technique used. Since emission spectrometers depend on the dispersion of the spectrum, preferably with high resolution to separate individual emission lines, the quality and size of these dispersing elements is very important.

Radiation emitted by the chosen source methods just described is passed through a narrow slit, and the image of this slit is formed at the detector end of the spectrograph or spectrometer. The radiation traveling between the slit and the slit image is collimated and dispersed according to wavelength. This produces a spectrum in the focal plane of the spectrograph. The three types of dispersing elements that are routinely used for emission spectroscopy are prisms, diffraction gratings, and Echelles. Table 9-1 gives the operating theory of each of these three types of dispersing elements.

Although instruments are routinely fabricated from all three types of these dispersing elements, the Echelle combines some of the advantages of the conventional diffraction grating with those of the Echellon Interferometer. The Echelle is capable of producing angular dispersion and theoretical resolving powers considerably in excess of those available from conventional diffraction grating mountings. This is important in that it is possible to build powerful and compact spectrographs with high light gathering power.

Disperse because of variation of index of refraction of common optical materials with wavelength

In a prism the deviation varies with wavelength

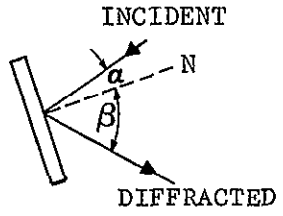
1. Angular dispersion:

$$\frac{d\theta}{d\lambda} = \frac{2 \sin \alpha/2}{\sqrt{1 - n^2 \sin^2 \alpha/2}} \left(\frac{dn}{d\lambda}\right)$$

2. Resolving power:

$$R = t\left(\frac{dn}{d\lambda}\right)$$

Disperse by phenomena of constructive and destructive interference



(a)

1. Angular dispersion:

$$\frac{d\beta}{d\lambda} = \frac{m}{a \cos \beta} = \frac{\sin \lambda \pm \sin \beta}{\lambda \cos \beta}$$

where m = order of interference

a = grating groove width

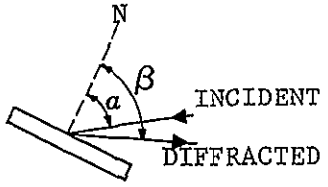
2. Resolving power:

$$R = mN = W \frac{(\sin \alpha \pm \sin \beta)}{\lambda}$$

where N = total number of grooves

Note: In above equations, use + sign when α and β are on same side of the grating and - sign when they are on opposite sides

Disperse by phenomena of conconstructive and destructive interference



(b)

For an echelle used as defined in a Littrow mount, $\alpha \cong \beta$

1. Angular dispersion:

$$\frac{d\beta}{d\lambda} = \frac{2 \tan \beta}{\lambda}$$

2. Resolving power:

$$R = \frac{2W \sin \beta}{\lambda}$$

3. Free spectral range:

$$F_\lambda = \frac{\lambda^2}{2t}$$

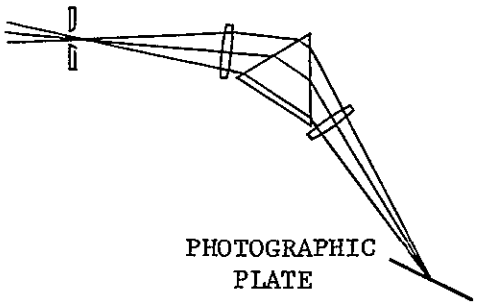
where t = groove interference distance

Table 9-1. General Theory of Dispersing Elements for Spectroscopy

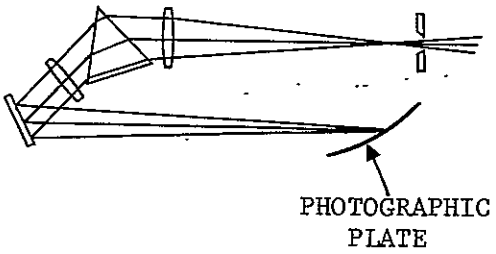
9.1.4 Spectra Measurement

While it is possible to perform certain qualitative and semi-quantitative analyses by visual observation of the spectra, quantitative emission spectrography has grown primarily through the use of photography. Although there is increased usage of photoelectric recording methods, photography continues to be the most used technique found throughout industrial research, development, and quality control. The photographic process is important because of its cumulative nature. This makes it possible to record a very weak spectra through extended exposures and also because it produces a permanent record. When a photographic plate is used to record spectra, it is placed in the focal plane of the spectrograph, as illustrated by the spectrographs shown in Figure 9-1. By careful calibration of the photographic plate and using reproducible development techniques, accurate quantitative analysis can be obtained from the spectra. This is done using a comparator densitometer which is capable of measuring the blackness of the lines on the photographic plate or film. This is routinely done by placing a photocell or phototube behind a slit on the screen over which the photographic plate is mechanically moved to obtain a relative output spectrum. This, in effect, is similar to having a direct recording system except that more sensitivity can be obtained with the in-between photographic process. It is appropriate to note here that if the photographic method were utilized for a flight-emission spectrometer, that the photographic plates could be returned to earth from orbit and measured by ground-based densitometer equipment.

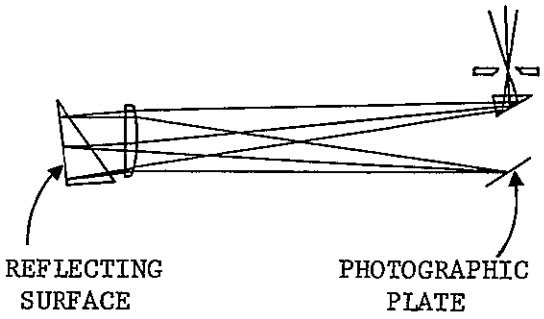
As an analytical technique, emission spectrometry is most important in the measurement of numerous elements in low concentrations, typically, 0.0001 to



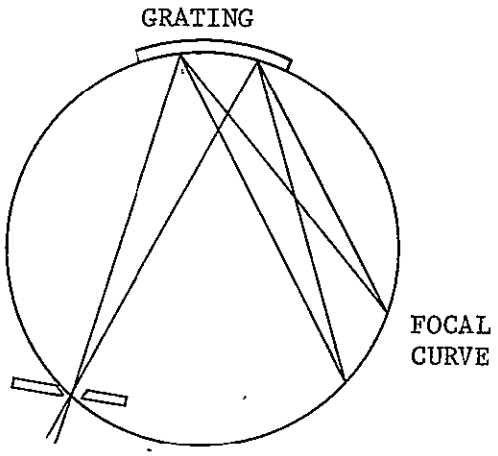
Medium quartz prism spectrograph.



Two-lens quartz prism spectrograph.



Littrow prism spectrograph



The Rowland circle.

Figure 9-1. Optical Variations of Emission Spectrographs

10 percent. In these ranges, the speed, precision, and accuracy for measuring a wide variety of elements are difficult to match by other techniques. The errors are typically in the range of 1 to 5 percent of the actual amount or quantity of the unknown constituent present. This depends to a certain degree on the type of source, the spectrograph or spectrometer, and the skill of the analyst.

9.2 APPLICATIONS

Emission spectrographs and emission spectrometers are an extremely useful analytical instrument since they can be used for the analysis of a wide variety of materials. In addition to wide applications in analysis of metals and solid powders and materials, the emission spectrograph is also useful for the analysis of trace metals in numerous liquids and biochemical materials. The emission techniques are especially useful where a preliminary evaluation of an unknown sample is needed to determine the semiquantitative presence of as many elements and compounds as possible. A specialized form of emission spectrometry is flame emission. This is covered as a separate subject in Sections 2 and 10.

Emission spectrographs and emission spectrometers are applicable to the following functional program elements (FPE's):

5.9 Small Vertebrates (Bio D)
5.10 Plant Specimens (Bio E)
5.13 Biomedical and Behavioral Research
5.16 Materials Science and Processing
5.17 Contamination Measurements
5.26 Invertebrates (Bio F)
5.27 Physics and Chemistry Lab

9.3 LOGISTICS

9.3.1 Packing and Launch

Almost all emission spectrometers and spectrographs are very large but are not typically heavy in proportion to their size, since most of the volume is used to house the optical system. Emission spectrometers often have large gratings or prisms; therefore, substantial care must be taken in launching to assure

that the optical bench and component mounting is structurally strong enough to withstand the rigors of launch and vibration. A substantial bulkhead attachment or tie-down must be provided with this type of equipment because of the bulk involved.

9.3.2 Installation

Because of the large size of most spectrometers/spectrographs, installation in space is not desirable. It might be preferable to launch with the frame attached in its final position in the laboratory. The optical components would be launched in the shuttle, and the instrument assembly completed in space. The power requirements are a major consideration for installation since the dc arc system can draw large amounts of current (for short periods of time). The source arc system must be attached to large bus lines that are capable of handling substantial currents. This would be on the order of 1,000 watts. In the case of a spectrometer, the readout electronics can utilize much lower amounts of power, on the order of 25 watts or less. For a spectrograph, no readout electronics are required with the exception of the photometric or densiometric equipment which may be included with other on-board equipment.

In addition to the high-power requirements, a venting system is required for the burned material occurring during the arcing of the sample. A small blower system or external vacuum vent system will be necessary. Considerable amounts of smoke may be generated during the short arcing period. The need for this venting or blower arrangement is similar to that required for other instruments which have flame sources. (Flame Photometer and Atomic Absorption Spectrophotometer).

9.3.3 Consumable Supplies

The supplies required for spectrographs are primarily the carbon electrodes used and required for the arcing of each individual sample. Standard samples usually available from the National Bureau of Standards should also be taken for purposes of instrument calibration and for preparation of calibration curves. These consumable supplies are of relatively small bulk compared with the actual size of the instrument.

When using spectrographs, the photographic plates are an important consumable supply. The equipment also required for developing these plates can become very bulky so that if a spectrograph is used, some consideration must be made for sufficicent photo development equipment for handling these plates after exposure.

9.3.4 Accessories and Spare Parts

The normal emission spectrometers and spectrographs require few spare parts. Typically, the most commonly used spare parts are additional photomultiplier tube housing assemblies with power supplies for direct reading equipment and, in some cases, different plate holders for photographic plates. Obviously, a number of different source assemblies are available as were discussed under Principles of Operation. Normally, for space applications, only one type of source system would be used.

9.3.5 Maintenance and Repair

In spite of their bulk and size, emission spectrographs are relatively maintenance-free. They have been used in laboratories for many years, and are

routinely used under heavy workload conditions with only minor maintenance required. Most maintenance is involved with the source system, but additional maintenance may be occasionally required in the electronic readout system,if used.

9.4 OPERATION

9.4.1 Warm-up and Speed-of-Operation

If a photodetector is used for measuring the emitted radiation, approximately 5 to 10 minutes may be required for warm-up. If a photographic plate system is utilized, there is no warm-up time for the emission spectrograph. Under these conditions, the sample arcing time is the primary consideration. This may range from as little as a few seconds to up to one minute.

9.4.2 Operation Skills

Normally, a minimal amount of ground-based training is required to operate the emission spectrograph. The primary consideration in operation skill is the sample preparation techniques used. It is certainly desirable that a person be trained in sample preparation and have some prior analytical skills with the emission spectrograph.

9.4.3 Operating Procedures

Typical operating procedures for emission spectrographs/spectrometers would be as follows:

Preparation:	Turn on electronics and allow a 5-minute warm-up. During warm-up, place both calibration standards and samples in the sample holder electrodes.

Calibration: Run the calibration standards and read out the values from the electronic system or expose on photographic plates. At the completion of calibration runs, the plate can also be used for the sample to be analyzed.

Measurement: After completing the calibration sample "burns", the unknown samples should then be "arced or burned." In most cases, a single photographic plate can hold multiple exposures for both sample and standards. If a direct reading system with photomultiplier or phototube detectors is used, the appropriate readings must be made at the end of each run and the system reset.

9.4.4 Sample Preparation and Handling

Samples are normally prepared as powders and placed into the arcing electrode cup. Sample packing preparation for the burn is the most tedious and time-consuming portion of the analysis. Care must be exercised in sample preparation to have a homogeneous mixture. Sample volatility and duration of burn are important characteristics that must be determined. It is here that the analytical skill of the individual preparing the samples and his prior experience is important

9.5 INTERFACE

9.5.1 Interface with Other Laboratory Instruments

The emission spectrometer is a completely separate and independent instrument and does not require any special interface with other instruments. If a direct reading electronic output instrument is used, the signal has sufficient output to be transmitted directly to the data management system or on-board recorder. The output can typically be 0 to 5 V or less for convenient recording or telemetry.

9.5.2 Interface with Vehicle System

There are two vehicle interfaces for the emission spectrometer. One of these is for the necessary power to operate the spark or arc source. This is an intermittent requirement, but requires one or two kilowatts of power. Commercial instrumentation normally operates on 115 V, 60 Hz power. In addition, approximately 20 to 100 watts is required for operation of the photodetector and amplifier if a direct reading system is used.

Some method must be made available for venting the sample during the arcing or sparking process. Sample ignition by these methods requires a small vacuum venting, discharge, or ducting system. This requirement would be similar to that needed in flame photometry.

9.6 SAFETY

9.6.1 Flame Hazards

The emission spectrometer is relatively safe with respect to the detector system. However, the basic dc arc or spark source generates a flame in the ignition of the sample. This means that extreme care must be taken that no hazardous gases are present or, preferably, that an inert gas purge is used during the ignition period. Fortunately, this ignition period is a relatively small period of time, approximately 5 seconds to 1 minute, so that the major flame hazard is only present during actual instrument use.

Very few hazardous materials are involved in the fabrication of the emission spectrograph. Most of the unapproved nonmetallic materials would be items

such as control knobs. However, the main optical bench is typically manufactured from metals and the plastics content is primarily limited to small mechanical parts.

9.6.2 Microbiological Hazards

The analyzer does not present any microbiological hazards to personnel.

9.6.3 Electromagnetic Interference

The emission spectrometer is a major source of radiated EMI during the period when the sample is ignited or burned. Special care would have to be taken in the design to minimize the electromagnetic interference generated. The remaining electronics are not a serious consideration and should have minimal EMI generation characteristics.

9.6.4 Ionizing Radiation

Ionizing radiation is neither produced by nor does it interfere with the operation of emission spectrometers.

9.6.5 Physical Hazards to Personnel

The major hazard presented by the emission spectrometer are the lethal voltages and currents generated in the spark or arc source. Normally, heavy insulation is utilized in all commercial instruments. A careful safety review should be made to insure that equipment chosen for flight use will not have a high voltage safety hazard. Noxious gases might be generated during the arcing operation. However, if venting is available, noxious gases should not provide a physical hazard to on-board personnel.

9.7 MODIFICATIONS

1. The commercial electronic components could be replaced by space or flight-qualified component counterparts.
2. Nonmetallic materials not approved for space should be replaced by approved materials.

9.8 AVAILABLE INSTRUMENTS

Many suppliers sell emission spectrographs and spectrometers commercially. Only a limited number of these would normally be considered for flight applications. They are listed on Table 9-2.

Manufacturer	Model	Cost	Wavelength Region	Power	Weight	Remarks
Spectrex Co.	6A	$2600	400-700 nm	115 V ac, 1000 W	35 lb	Semi-portable system. Paschen mounting, dispersion 40.5 Å/min, visual readout.

There are a number of suppliers of emission spectrographs. As a whole, the instruments are large and bulky and not eminently suitable for flight. Other manufacturers are:

Angstrom, Inc.

Applied Research Labs

Bell & Howell

Engis Equipment Company

General Electric Company

Jarrell-Ash

McPherson Instrument Corp.

National Spectrographic Labs, Inc.

Pitchford Scientific Instruments Corp.

SpectraMetrics, Inc.

Spex Industries, Inc.

Varian Associates

Warner & Swasey Company

Zeebac, Inc.

Table 9-2. Emission Spectrometers

Section 10
FLAME PHOTOMETER

10.1 PRINCIPLES OF OPERATION

The operation of the flame photometer is similar to the emission mode of operation of the atomic absorption spectrophotometer (see Section 2.) A sample is introduced into a flame. As the sample is nebulized, it gives off light at wavelengths characteristic of the atomic species it contains. The intensity of light at the characteristic wavelengths is proportional to their concentration. Flame photometers are generally special-purpose instruments made to analyze only a few elements. The photometer contains a selected group of filters (rather than a grating). It reads the intensity of light passing through the filter with a photomultiplier and amplifier. Flame photometers provide a readout in milliequivalents per liter. This is possible because the sample is diluted into a solution with a known concentration of an ionic species not present in the sample. For blood and urine samples, the comparison is lithium.

By taking simultaneous readings of the light transmitted through three filters, the readings for two elements (sodium and potassium) are each compared with a known concentration of lithium with which the sample was diluted. The outputs are then provided by analog circuitry according to the following logic:

MATHEMATICAL LOGIC

I_{Na} = Current due to Na in Sample from Na Detector

I_{Li} = Current due to Li in Sample from Li Detector

I_{K} = Current due to K in Sample from K Detector

I_{BNa} = Background Current from Na Detector, Flame, etc.

I_{BLi} = Background Current from Li Detector, Flame, etc.

I_{BK} = Background Current from K Detector, Flame, etc.

S = Slope Adjustment

E = = Voltage

$$\text{Na+ Reading} = S \times \frac{I_{Na} + I_{BNa}}{I_{Li} + IBLi}$$

$$\text{K+ Reading} = S \times \frac{I_{K} + I_{BK}}{I_{Li} + I_{BLi}}$$

The zero adjustment, made with the Li standard, electronically subtracts the I_{BNa}, I_{BLi}, I_{BK} from the analog circuits. Then because voltage rather than current drives the digital readouts, the above equations reduce to:

$$\text{Na+ Reading} = S \times \frac{E_{Na}}{E_{Li}}$$

$$\text{K+ Reading} = S \times \frac{E_{K}}{E_{Li}}$$

10.2 APPLICATIONS

Flame photomers are applicable in the following functional program elements (FPE's):

5.9 Small Vertebrates (Bio D)
5.10 Plant Specimens (Bio E)
5.13 Biomedical and Behavioral Research
5.23 Primates (Bio A)
5.25 Microbiology (Bio C)
5.26 Invertebrates (Bio F)

Flame photometers are used for determination of sodium, potassium, calcium in blood serum, and urine.

10.3 LOGISTICS

10.3.1 Packing and Launch

The packing procedures for flame photometers are not particularly different from other precision photo optical instruments. The most fragile part of the instrument is the photomultiplier tube.

10.3.2 Installation

Unpacking and installation of the flame photometer is routine. Mounts are needed to keep the instrument stationary during space operations. Installation is complete with connection of electric power, gas, and venting.

10.3.3 Consumable Supplies

Consumable supplies for flame photometers include gas for the burner and the lithium calibration solution.

10.3.4 Accessories and Spare Parts

An automated sample changer and a strip-chart recorder are the principle accessories for the flame photometer. Neither of these is recommended for space applications.

Spare parts for the flame photometer include the burner assembly, extra filters, a photomultiplier tube, and extra amplifier and logic circuit boards.

10.3.5 Maintenance and Repair

The electronic and optical components of flame photometers present the same general problems as maintenance of other electronic and optical equipment. There are mechanical components in some instruments which present special maintenance problems. These include: choppers, reed switches, and servo-motor control of digital readout counters. The gas and flame systems for flame photometers are clean burning and relatively maintenance-free.

10.4 OPERATION

10.4.1 Warm-up and Speed-of-Operation

Turn-on and warm-up time is negligible for solid-state instruments, and flame ignition is automatic. A method should be included to turn off the flame between measurements when the instrument is not in continuous use. Normally, the instrument will be turned on, measurements made and recorded on a few samples, and the instrument turned off within a few minutes.

10.4.2 Operation Skills

Space Station operation of the flame photometer is possible for either professional or technical personnel. Some previous experience or training is

necessary. More training and experience is required to perform maintenance on the instrument.

10.4.3 Operating Procedure

Flame photometers are designed for simple routine operation. A typical procedure would be as follows:

Preparation:	Turn on instrument/light flame.
Calibration:	Calibrate zero and gain adjustments.
Measurement:	Measure and dilute sample (lithium standard solution used). Introduce sample (from enclosed container). Read values (in milliequivalents per liter) and record.

10.4.4 Sample Preparation and Handling

Samples for analysis by flame photometry must be taken from a closed system, diluted with the lithium standard solution, and introduced into the flame. In a zero-g environment, fluids must be stored in closed containers using pressure differences for fluid transfer. The velocity of gases in the flame provides a negative pressure for aspiration of the sample into the burner.

Automated sample-handling is not recommended for space applications.

10.5 INTERFACE

10.5.1 Interface with Other Laboratory Instruments

Although some earth-based flame photometers are used in conjunction with automated analyzers for clinical chemistry, only instruments which do not depend on these peripheral equipment are recommended for space application. The

flame photometer may be one of several instruments used for biochemical analysis of body fluids.

10.5.2 Interface with Vehicle Systems

The flame photometer will require fuel (propane is normally used), compressed air, and electric power from the vehicle systems. The preferred power to operate a flame photometer in a Space Station environment without major modifications to the electronic and mechanical components is 115 volts, 60 Hz. Modifications to use dc or 400-cycle power would be easy to implement.

Flame venting is required. Maximum safety could be provided by complete separation of the flame from the cabin environment. A vacuum line should also be attached to allow for draining the vaporization chamber.

10.6 SAFETY

10.6.1 Flame Hazards

The presence of an open flame is the major safety hazard of the flame photometer. Flameless methods for sample atomization using a plasma torch or plasma flame present even greater safety hazards. With proper venting and isolation of the flame from the cabin environment, adequate safety can be maintained to allow use of the flame photometer in the Space Station.

10.6.2 Microbiological Hazards

The flame photometer presents no microbiological hazards. Any microorganisms present in the sample would be destroyed in the flame.

10.6.3 Electromagnetic Interference

The flame photometer represents the following possible sources of interference:

- Dc-to-dc converter to convert to 28 V dc operation (if required).
- Flame igniter.
- Solenoid valves for gases.
- Logic in control or readout circuitry.

Sufficient shielding and line filtering will limit radiated and conducted interference to acceptable levels. The flame igniter, typically a spark generator, may be difficult to control even with a moderate amount of shielding and filtering.

However, this is only a single event transient occurring infrequently and, therefore, should present no serious interference problem.

The detectors represent high impedance circuitry and, therefore, may exhibit susceptibility to RF and transient energy without shielding and filtering. The filtering and shielding provided for interference control should suffice.

10.6.4 Ionizing Radiation

Ionizing radiation would be produced by a flame photometer only to the extent that radioisotopes may be present in the samples analyzed. Proper venting of the flame would remove these possible radioactive particles from the cabin environment, leaving their ultimate disposal to the venting system.

10.6.5 Physical Hazards to Personnel

Sharp corners and protruding knobs present on most models of flame photometers present some physical hazards to personnel. These hazards can be reduced by techniques for mounting the instrument and modification of the front panel controls. Separation of the flame from the cabin should include the thermal insulation necessary to avoid burns.

10.7 MODIFICATIONS

The following modifications are needed for adapting a flame photometer for Space Station use.

1. The flame should be vented and separated from the space-station cabin environment.

2. The instrument should be mounted to be stable on the bench and to avoid personnel injury from accidental encounter with edges, corners, or protrusions of the instrument.

3. In Earth-based use, the sample vaporization chamber of the burner drains to prevent accumulation of fluids in the chamber. In a zero-gravity environment, this draining would not occur. The more efficient drawing of the sample into the flame, in the absence of gravity, would probably make this unnecessary. However, until this point is demonstrated in flight, this drain should be connected to a vacuum source to allow evacuation of the vaporization chamber, if necessary.

4. Modification to use on board methane (rather than propane) would reduce supply needs.

5. Sample intake must be made compatible with wet-chemistry procedures.

10.8 AVAILABLE INSTRUMENTS

The major manufacturers of flame photometers are as follows:

Beckman Instruments, Inc.
Coleman Instruments
Evans Electroselenium Ltd.
Instrumentation Laboratory, Inc.
National Instrument Laboratories, Inc.
Yallen Instruments, Inc.

All instruments by these manufacturers have similar external features and capabilities. They aspirate a liquid sample to provide a direct digital reading of concentrations of sodium and potassium (some also give calcium) in blood serum or urine in milliequivalents per liter. Although several current models have vacuum tube circuitry and mechanical components, at least two manufacturers (Beckman Instruments, Inc., and Instrumentation Laboratory) are soon to release improved instruments. The new Beckman Flame Photometer will eliminate mechanical components and provide modular solid-state circuitry.

Section 11
GAS CHROMATOGRAPHS

11.1 PRINCIPLES OF OPERATION

Gas chromatographic separations involve the transport of a sample of a vapor or gas mixture through a chromatographic column. The column contains a substance--the stationary phase--which may consist of a solid where "adsorption" principles are used, or a solid support material with a liquid coating where the principle of "absorption" is used. The transport of the constituents of the sample through the column is effected by a carrier gas--the moving phase. Separation of the gas mixture constituents takes place within the column, "Stationary Phase" (Figure 11-1). The sample molecules are injected at the head of the column and begin to move through the column under the motive forces of the carrier gas, "Moving Phase". The light molecules will travel through the column faster than the heavy ones. Therefore, the time that the light molecules are in the column will be shorter than the time the heavy molecules stay in the column. It is this difference in column retention time for different molecules that provides the separation. A detector is then placed at the outlet of the column and measures the relative concentration of each component while the elution time sequence can be employed to identify each component.

A typical instrument using the principles of gas chromatography consists of the following major elements: a chromatographic column (interchangeable for various applications); a carrier gas flow control; a heated sample inlet system through which measured quantities of sample may be introduced into the system; a thermal

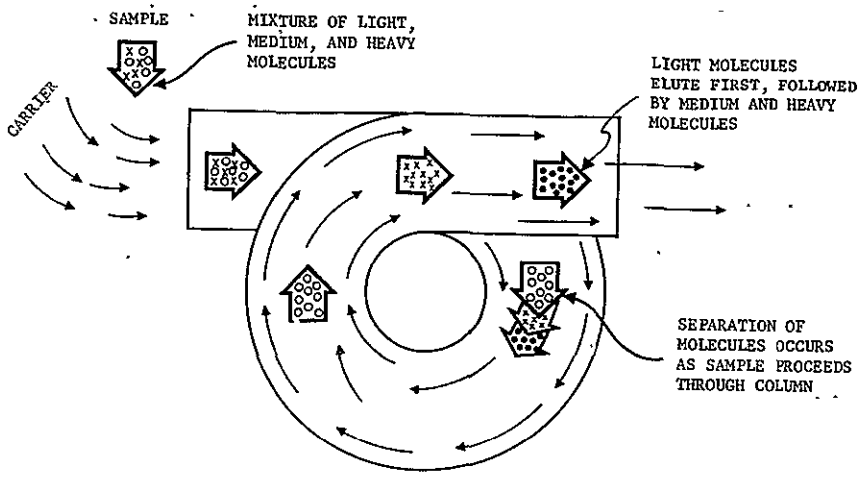


Figure 11-1. Chromatographic Column

An inner insulated compartment, maintained at proper operating temperature by the internal heater, encloses the chromatographic column and the detector. The desired operating temperature is selected by means of a control on the front panel of the instruments; an indicator denotes functioning of the heater system and indicates temperature control during operation.

The sample components are injected into the sample inlet (Figure 11-2) and swept by the carrier gas into the chromatographic column where they are adsorbed (or absorbed) by the column filling-material. As the carrier gas continually flows through the column, it carrier off individual components at different rates.

The gases flow from the column through the sensing side of the detector cell to the exhaust at the rear of the instrument. A corresponding flow of carrier gas flows through the reference side of the cell, first passing through the

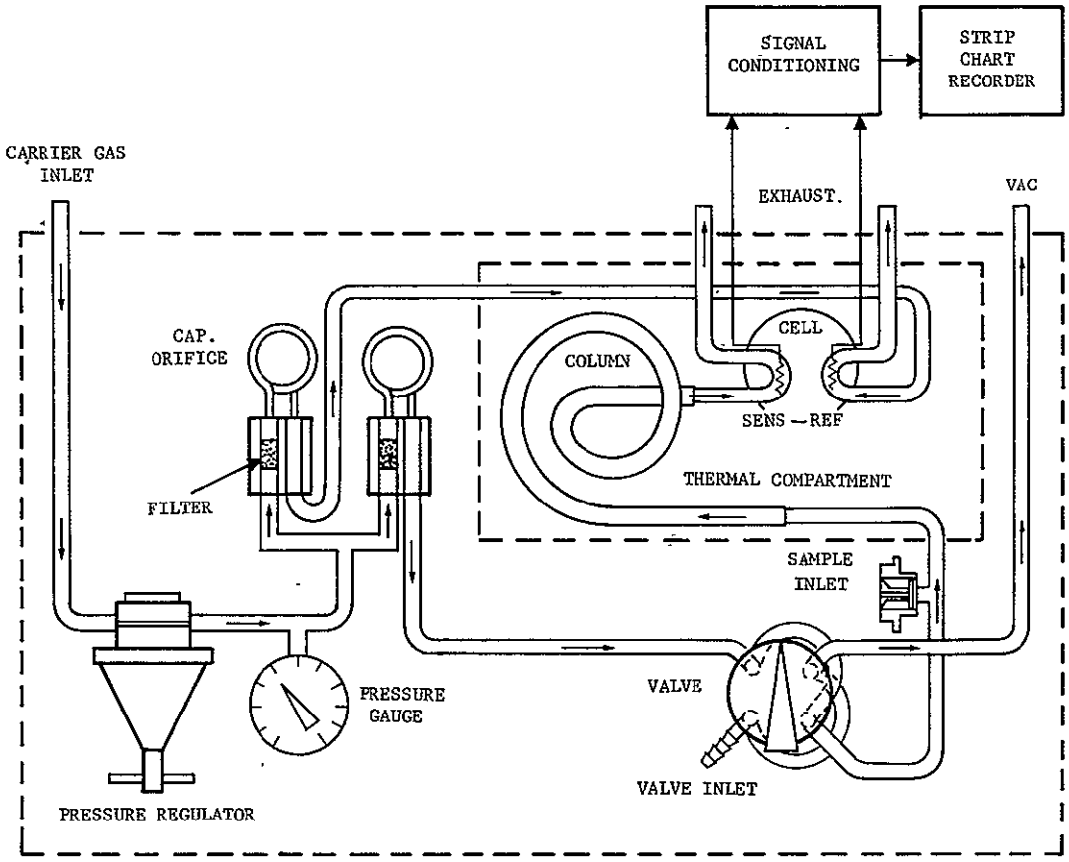


Figure 11-2. Gas Chromatograph Flow and Block Diagram

heated compartment so that it reaches the cell at the same temperature as the sample-carrier gas mixture. Both carrier gas streams are then exhausted.

The difference in thermal conductivity between the carrier gas in the reference side of the detector cell and the sample-carrier gas mixture in the sensing side produces a voltage differential signal. When only carrier gas is flowing through the system, there is no voltage differential--hence, no signal to be indicated by the recorder.

The differential voltage from the detector is transmitted to a recorder which plots a curve showing the separation of the sample into its components (Figure 11-3). The area beneath the recorder trace is proportional to the quantity of the sample component. However, equal quantities of different components yield only approximately equal areas. In many instances, it is possible--and frequently it is more convenient--to use peak height of the trace as a quantitative measure of each component.

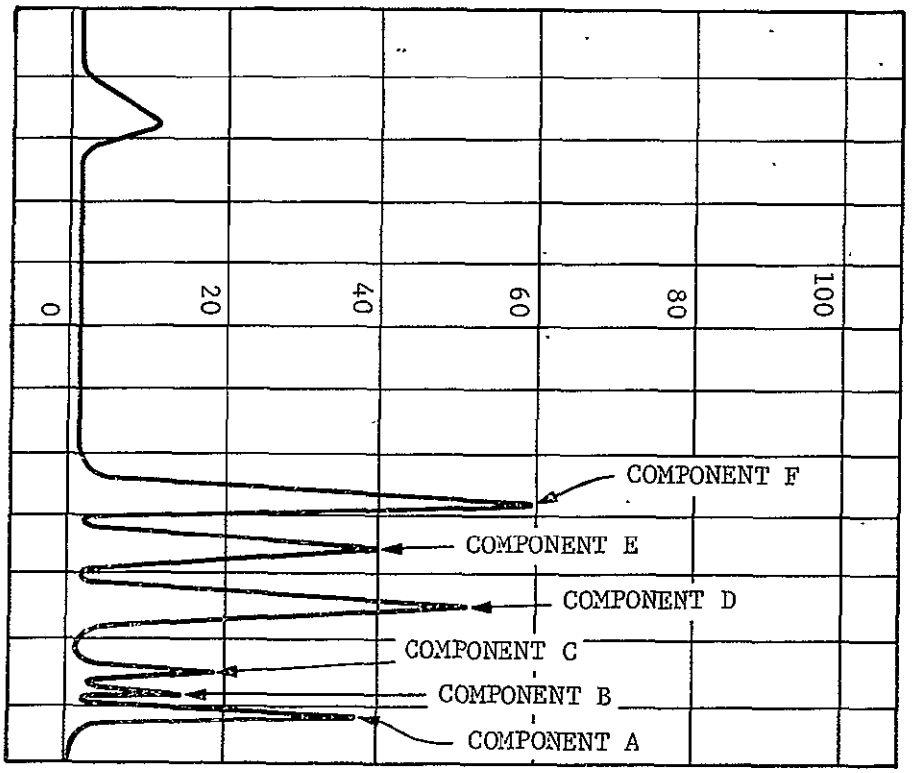


Figure 11-3. Typical Chromatogram

One method of calibrating the instrument is to make trial runs at given flow rates and temperatures with pure components. Another method uses mixtures in which the concentrations of the components used for calibration purposes are known. From the results of these runs, calibration curves may be constructed and used to determine the concentration of each of the components of any sample.

Classification of the type of chromatographic analysis is done on the basis of the moving phase followed by the stationary phase. (For example, gas-solid chromatography, gas-liquid chromatography, etc.) Instruments of the type considered for space applications are designed for gas-solid or gas-liquid analysis.

11.1.1 Gas-Solid Chromatography (Adsorption)

The moving phase is a gas (carrier gas) while the stationary phase is an "active" solid. Separation occurs as a result of the adsorption process. The process of adsorption consists of the adhesion, in an extremely thin layer, of the gas molecules to the surface of solid bodies. When adsorbed, the energy required to break the bond and, therefore, move the sample through the column may be very high. For this reason, the use of gas-solid chromatography is restricted to the analysis of low boiling sample components where volatility of the components is high enough to greatly reduce the strength of the adsorption bond. If the adsorption bond is too strong to be broken by carrier gas flowing through the column, the active solid will eventually become deactivated due to the continued addition of the noneluted components.

Gas-solid columns typically are prepared from 5- to 10-foot lengths of 1/8-inch O.D. stainless-steel tubing and filled with 8-100 mesh particles of molecular

sieves or silica gel. These columns must be "conditioned" at temperatures above 150°C while flowing a dry gas to remove absorbed moisture. A properly conditioned molecular sieve column will separate the light gases in the order of hydrogen, oxygen, nitrogen, methane, and carbon monoxide. Silica gel columns are used primarily for carbon-dioxide separation but can also be used for three carbon (or smaller) organic compounds which are adsorbed on the front of the gas-solid columns. Adsorption of water and organic material in gas-solid columns necessitates periodic reconditioning of the column.

11.1.2 Gas-Liquid Chromatography (Absorption or Partition)

The moving phase is a gas (carrier gas) while the stationary phase is a high boiling-point liquid deposited on an inert solid support. Separation occurs as a result of the absorption process. For example, the sample dissolves into the liquid coating (partition liquid) and separation is due to differences in volatility from solution. The energy required to remove a compound from the solution is much lower than that required to break an absorption bond. Absorption or partition columns thus have a much wider range of application in that they are capable of handling the heavier hydrocarbons.

In gas-liquid columns, the partitioning agents are relatively inert diatomaceous earth particles coated with a thin layer of viscous liquid. The primary retardation which effects separation is absorption of the compounds in the sample into liquid phase. Even compounds with very similar solubility properties can be separated since the absorption/desorption phenomenon occurs thousands of times before a compound is finally eluted. As might be expected, more volatile compounds are eluted earlier than less volatile compounds. The elution order

can be considerably altered by using liquid phases with different characteristics. A nonpolar liquid such as silicone oil will tend to retain nonpolar hydrocarbons much longer than more polar-oxgenated organic compounds. Just the opposite effect is obtained if polar liquid phases such as Carbowax are used. In any case, gas-liquid packed columns usually are limited to separating approximately 20 compounds at any one set of operating conditions. More volatile compounds are eluted as a single peak at the beginning of the run, while less volatile compounds are eluted too late to be of practical use. Compounds in the same volatile region may produce unresolved peaks, but these may be separated by a second run on a different column. The easiest method of increasing the column capability is to use temperature-programming techniques (starting at a lower temperature and gradually increasing to a higher temperature). This technique will permit much more volatile compounds to be separated and will permit less volatile compounds to be eluted in a reasonable time.

Open-tubular columns are a gas-liquid partitioning type in which the liquid phase is evaporated on the inner surface of a capillary tube rather than on an inert support. These columns typically have inside diameters of 0.02 inch and are 200 feet long. They have far better resolution than packed columns and usually do not require temperature programming. They are more susceptible to stripping of the liquid phase and are poorly suited for the large sample sizes needed for trace analyses.

11.1.3 Carrier Gas (Moving Phase)

The carrier gas composition is determined by the type of detector used. Nitrogen and helium are the most commonly-used carrier gases. The flow rate of the carrier

gas is inversely related to the elution times of the various compounds being separated. Maximum resolution will occur at one particular flow rate, with higher or lower flow rates giving somewhat poorer resolution. The optimum flow rate is approximately 15 ml/min for 0.09-inch I.D. packed columns, and 5 ml/min for 0.02-inch I.D. open, tubular columns. The carrier gas flow usually is maintained by a constant-pressure regulator.

The injection valve must maintain a carrier gas flow through the system at all times and periodically inject a small, discrete sample into it. Liquid sample sizes generally are in the 0.1 to 10 µl region, while gaseous sample sizes generally are in the 0.1 to 5-ml region. Small samples provide the best resolution, while large samples provide the best sensitivity. The most common method of injection is by using a manually operated syringe through a rubber septum.

11.1.4 Detectors

A detector is needed to translate the column output into electrical signals. Thermal conductivity detectors, as described in Paragraph 11.1, are most commonly used. When used with helium carrier gas, they respond to all other compounds and typically are capable of detecting down to the 10-ppm level.

Cross-section ionization detectors typically have the same response characteristics as thermal-conductivity detectors. If used with ultrapure helium, they can detect down to 1-ppm levels.

Flame ionization detectors are not responsive to nonflammable gases and thereby have much better signal-to-noise characteristics to organic compounds than do

"universal" detectors. This is the most commonly used trace contaminant detector and typically has a sensitivity limit of 0.1 ppm.

The other commonly used detector is the electron-capture device. With this detector, compounds with strong electronegative characteristics such as halogenated compounds produce a marked reduction in current flow between two electrons. Ordinary hydrocarbons produce no response. If responsive compounds are present in a mixture of hydrocarbons, sensitivities better than one part per billion can be achieved even though no physical separation occurs. This is a widely used detector for pesticides, air pollutants, and other electronegative compounds.

Many other types of detectors are available commercially. These include flame photometers, thermionic-emission detectors, mass spectrometers, coulometric detectors, and others. Most of these devices are special-purpose detectors that have great value for some applications, but not for the more routine applications anticipated for spacecraft missions.

11.2 APPLICATIONS

Gas chromatographs can and have been used for separating and detecting practically all compounds having boiling points lower than 900°C. For the space-mission application, it is anticipated that liquid samples from biological experiments, the drinking water supply, spacecraft wash water, and urine would be of greatest interest. In this case, either a liquid sample injection valve or a microsyringe could be used for injection. The syringe is preferred from the standpoint of peak sharpness and versatility, but may create a hazard if not handled properly.

Gaseous analyses could be performed on samples from the cabin atmosphere. Since numerous simulator experiments have shown that trace contaminant concentrations increase slowly, there is little need to perform automatic, repetitive analyses. Manual injections of the atmosphere, therefore, should be made occasionally. This can be performed with either a gas injection valve or syringe.

Chromatographs are applicable to the following functional program elements:

5.9 Small Vertebrates (Bio D)
5.10 Plant Specimens (Bio E)
5.13 Biomedical and Behavioral Research
5.15 Life Support and Protective Systems
5.16 Materials Science and Processing
5.17 Contamination Measurements
5.23 Primates (Bio A)
5.25 Microbiology (Bio C)
5.26 Invertebrates (Bio F)
5.27 Physics and Chemistry Lab

11.3 LOGISTICS

11.3.1 Packing and Launch

Gas Chromatographs and their accessories are generally quite rugged. The gas bottles should be separately mounted in a cushioned packing case to avoid rupture. If hot-wire thermal conductivity detectors are used, these should also be packed separately. In general, packing for railroad shipment should prove adequate for shipment of Gas Chromatographs by space shuttle.

11.3.2 Installation

Unpacking and installation of gas chromatographs is routine. Mounts are needed to keep the instrument and carrier-gas supply stationary during space operations. The instrument should be mounted such that all gas fittings, columns, detectors, and electronics are readily accessible. A source of 115-V ac, 60-Hz power is required for instrument operation. The carrier-gas supply should be connected to the instrument and all fittings need to be checked for leaks.

11.3.3 Consumable Supplies

Consumable supplies for gas chromatographs are the carrier gases required for instrument operation. Nitrogen and helium are the most commonly used carrier gases. The carrier-gas, whichever type is used, is consumed at approximately 10 ml/min. A supply of replacement rubber septums and manually operated syringes are also required. If a hydrogen flame ionization detector is used, the hydrogen will be consumed at the rate of 20 ml/min. For safety reasons, hydrogen is not recommended. Finally, approximately two feet of chart paper will be required per analysis.

11.3.4 Accessories and Spare Parts

The "standard" gas chromatograph generally consists only of an oven and a septum-type injection port. Various detectors, oven-control units, electronic amplifiers, integrators, injection valves, columns, and gas-control valves are then sold as accessories. Thus, most manufacturers have modular assemblies that can be tailored to a specific application. Spare columns and rubber septums for the injection port should be included as a minimum for spares.

11.3.5 Maintenance and Repair

Major repairs should not be attempted under space-flight conditions. However, major operating components such as detectors, columns, valves, and plug-in electronic modules, could and should be replaced in the event of failure. Due to the requirement for good temperature stability, the oven heaters should remain on if the instrument is to remain stable for long periods of time. Daily and weekly performance and calibration checks are necessary. Also, the sample valves require periodic cleaning and lubrication.

11.4 OPERATION

11.4.1 Warm-up and Speed-of-Operation

The oven should be brought to temperature with a normal carrier-gas flow rate for at least one hour prior to use. This permits the elution of volatile decomposition products for the column.

11.4.2 Operation Skills

Very little skill is required to perform routine analyses. Interpretation of the chromatograph from simple analyses generally can be accomplished after a few hours training. However, utilization of the wide variety of types of analysis possible with a gas chromatograph requires considerable operator experience.

11.4.3 Operating Procedure

A typical analysis would be as follows:

- Set the oven temperature to the correct temperature.
- Select the proper column and detector.
- Inject the sample.
- Interpret the resultant peaks from a strip chart recorder.

11.4.4 Sample Preparation and Handling

For most applications, no chemical reactions need be performed prior to analysis. For atmosphere trace-contaminant analysis, some form of concentration will be required to achieve adequate sensitivity and reliability. Most concentration is performed by passing air through a tube immersed in liquid nitrogen. This only results in about a hundred-fold concentration and is difficult to perform in a spacecraft.

11.5 INTERFACE

11.5.1 Interface with Other Laboratory Instruments

The separated components of the output of the Gas Chromatograph can be further analyzed by many other laboratory instruments. These include mass spectrometers, infrared analyzers, radiation counters, and spectrophotometers.

11.5.2 Interface with Vehicle Systems

The Gas Chromatograph will require a source of 110 V ac, or suitable modifications to utilize other forms of power. A source of compressed carrier-gas also will be required. Some heat dissipation may be needed.

11.6 SAFETY

11.6.1 Flame Hazards

The use of a hydrogen flame ionization detector represents the greatest safety hazard. With proper venting of the flame and isolation of the flame from the cabin atmosphere, adequate safety might be maintained in the space station. A flame is not needed, however, if suitable concentration of the samples can be obtained.

Hot-wire filament detectors are capable of ignition in an oxygen atmosphere. This can be prevented by ventilating the detector outlet to space, or by using a sensor to ensure the presence of inert carrier-gas before current is allowed to flow in the detector.

11.6.2 Microbiological Hazards

Gas Chromatographs present no microbiological hazards. Any microorganism present in a sample would be destroyed by instrument operating temperatures.

11.6.3 Electromagnetic Interference

Electromagnetic interference is not produced by gas chromatographs nor interfere with the operation of gas chromatographs.

11.6.4 Ionizing Radiation

Ionizing radiation is neither produced by nor interferes with the operation of these instruments.

11.6.5 Physical Hazards to Personnel

Sharp corners and protruding holes present some problems on Gas Chromatographs. These hazards can be reduced by slight alterations of the front panel controls and instrument shape.

11.7 MODIFICATIONS

The following modifications are needed to adapt gas chromatographs for space-station use:

- The flame from hydrogen flame detectors (if used) should be vented.
- The effluent from a hot-wire detector should be vented.
- Sharp knobs and corners should be modified.

11.8 AVAILABLE INSTRUMENTS

The major available instruments and their specifications are listed in Table 11-1.

Company	Model	Price w/o Recorder	Type of Detector	Max Column Temp (C)	Sub Ambient Operation	Carrier Gas	Multiple Column Operation	Multiple Detector Operation	Temp Programmed	Principle Application
Barber-Colman Company	5000 (coiled columns)	$4,345 & up	TC, HF, Ar, EC, P, R	500 (180 C min opt)	Yes	Any	Up to 3 including capillary	21 comb of 2, or 35 comb of 3	Yes	GA, TA, PA
	5000	$7,535	HF dual; TC, EC, Ar, P & R (opt)	500	No	Any	2	21 comb of 2, or 35 comb of 3	Yes	GA, TA
Beckman Instruments, Inc.	GC-4	$4,620 to $10,000	F, TC, EC, Hel	500	Yes	Any	3	3 at a time (choice of 4)	Yes	GA, TA, PA, PR
	GC-5	$4,200 to $6,500	F, TC, EC, Hel	400	Yes	Any	2	2 at a time (choice of 4)	Yes	GA, TA, PA, PR
	GC-45	$4,900 to $9,800	F, TC, EC	400	No	Any	2	2 at a time (choice of 3)	Yes	GA, TA, PA, PR
Bendix Corp.	CL-2400	$18,000	Dual T/C, dual flame	350	No	He, N_2 Most other common carrier gases	No	No	No	PA
	CL-2500 Series	$4,065	F,DFID,T/C,ECD	400	No	Any	4 simultaneous	4 simultaneous	Yes	GA, TA, PA
Fischer Scientific Co.	4400	$4,000 to $8,000	FID,TC,EC (63Ni)	450	Yes	Any	Yes, up to 4	Yes		GA
Hewlett-Packard Co.	7626A	$8,375	Dual Fl, dual TC/EC	500	Yes	He or Ar + CH_4	2	3	Yes	GA, TA
675	6756B	$6,500	Dual Fl, dual TC/EC	500	No	He or Ar + CH_4	2	3	Yes	GA, TA
	402	$4,000	Dual Fl	500	No	He	2	2	Yes	GA, TA
Leeds & Northrup Co.	7851 Chromomax II	$2,460	T	210	No	He	2	NG	No	PA
Packard Instrument Co.	7424 GC	$5,000	HF, EC	500	No	Any	2	2	No	GA, TA. PA
	7400	$3,100 to $7,050	TC(F),FI,EC(H^3 or Ni^{63}),Av,P,GD,dual FC,dual EC, EC/FC, EC/TC, FC/TC, FC/dual TC	500	Yes	Any	2	2 or 3 at a time (choice of 7)	Yes	GA, TA, PA
Perkin-Elmer Corp.	900	$4,800 to $8,800	TC(F),Fl,EC(H^3) or Ni^{63}), thermionic	400	Yes	He,N_2,Ar-CH_4	Yes	Yes	Yes	CA, TA
Pye Unicam Ltd.	104	$2,600 to $5,600	Fl, TC, Tl, EC	500	No	Ar, N_2, He	Up to 2	Up to 2	Opt	GA, TA
Shimadzu Seisakusho Ltd.	GC-1C	$4,400	F, Ar, EC, HF	420	No	Any	3	4	Yes	GA, TA, PA, PR
Varian Aerograph	711/712/713	$4,600 to $7,500	HF	400	No	N_2,He,Ar	NA	NA	Yes	GA, PA
	1868 series	$5,600 to $7,200	TC(F or T),GD,HF EC(H^3 or Ni^{63}),P	400	No	N_2, He. H_2, Ar	2	Dual	Yes	GA, TA, PA, PR

NOTES:

GA - General Chem Analysis
TA - Trace Analysis
PA - Preparative
PR - Process

F - Filament
TC - Thermal Conductivity
EC - Electron Capture
FI - Flame Ionization

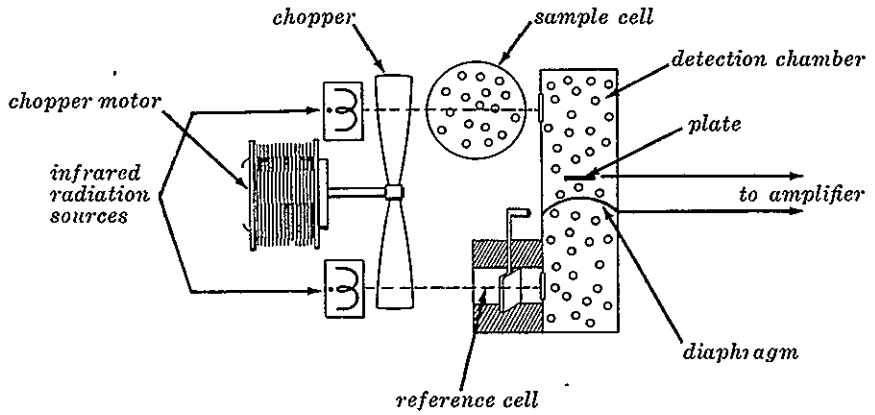
Table 11-1. Gas Chromatographs

Section 12
INFRARED ANALYZERS

12.1 PRINCIPLES OF OPERATION

Infrared analyzers are specific component analyzers which provide a continuous determination of the concentration of a selected constituent in a gaseous or liquid stream (Figure 12-1). The most common types use two light beams which pass through a reference cell and sample cell, respectively. The sample cell contains the gas or liquid to be analyzed, and the reference cell normally contains a nonabsorbing gas or a known concentration of a selected component. The infrared radiation passing through the sample cell is absorbed by the component of interest only in the wavelength regions where that component has infrared absorption bands. The percent of radiation absorbed is proportional to the concentration of the component of interest in the sample stream.

Due to the difference in the gas absorption in the reference and sample cells, the amount of energy entering the detector from the reference cell is greater than the amount of energy entering the sample side of the detector. This is due to absorption of a portion of the IR source energy by the selected component. The detector is a sealed container consisting of two compartments of equal volume separated by a thin, flexible metal diaphragm. Both compartments of the detector are filled with the specific gas or vapor of the component to be measured. The infrared radiation passing through the reference cell enters one compartment of the detector while the radiation passing through the sample cell enters the other compartment. This causes the gas contained in each



operation

Two identical infrared sources in the pickup head emit beams of radiation that are pulsed by a motor-driven chopper. One beam passes through a sample cell, the other beam through a reference cell, and both beams enter opposite ends of the detection chamber.

The detection chamber is a permanently sealed unit divided into two compartments by a thin, metal diaphragm. Both compartments are charged to the same pressure with the gas being measured.

When the gas being measured enters the sample cell, it absorbs infrared radiation at the same wavelengths as the gas in the detection chamber. This reduces the amount of radiation reaching the gas in the sample side of the detection chamber and produces a lower pressure in that side. The diaphragm bends toward the side of lower pressure, and this movement is converted into electrical impulses which are translated by the amplifier into meter readings and recorder signals.

Figure 12-1. Nondispersive Infrared Analyzer Diagram

compartment to be heated by the incoming energy resulting in an increase in pressure proportional to the temperature rise. The pressure rise is greater in the compartment receiving the radiation from the reference cell since a portion of the radiation transmitted to the sample cell has been absorbed by the selected component contained in the sample cell. The unequal pressures between the two compartments cause the diaphragm to expand unequally. The amount of expansion is proportional to the difference in pressure between the two compartments, and is monitored by measuring the change in capacitance between the diaphragm and a fixed button contained within the detector.

An optical chopper is normally placed between the infrared radiation sources and the two sample cells causing modulation of the beam. When the chopper blocks both beams, the pressure in the two compartments of the detector is equalized and the diaphragm returns to a normal position. Each time the beams hit the detector, the pressure again becomes unequal, causing the diaphragm to move at a rate equal to the modulating frequency.

The infrared analyzer; normally called a nondispersive analyzer, has several unique advantages over spectrophotometers for specific gas analyses. The fact that the detector can be charged with a single component makes the analyzer highly specific to a selected constituent. Secondly, additional filters can be placed in the reference or sample beam to insure better selectivity and increased attenuation for specific interfering components in the sample stream. The output is linear with concentration, and the sensitivity is normally 50 to 100 times higher for a given sample pathlength than a spectrophotometer.

A single-beam version of the nondispersive analyzer is also available which uses two or sometimes three detector compartments in series. This type of analyzer has certain advantages for selected applications and results in a more compact analyzer. The elimination of the reference cell makes the instrument less sensitive to degradation of the sample cell windows and eliminates the requirements for matching two IR sources. By using different charges in the detector compartments, better interference rejection can often be realized.

12.2 APPLICATIONS

Infrared analyzers are directly applicable to the following functional program elements (FPE's):

5.9 Small Vertebrates (Bio D)
5.10 Plant Specimens (Bio E)
5.13 Biomedical and Behavioral Research
5.15 Life Support and Protective Systems
5.16 Materials Science and Processing
5.17 Contamination Measurements
5.23 Primates (Bio A)
5.27 Physics and Chemistry Lab

The infrared analyzer is particularly useful for the continuous analysis of a single constituent in a multicomponent gas mixture. The analyzers are ideally suited for monitoring components of low concentration, and are most easily applied where the measured component is of considerably different composition than the background components.

For space applications, the infrared analyzer would be ideal for monitoring cabin atmospheres for trace components, such as carbon monoxide or hydrocarbons.

Fast-responding versions are available for measurement of exhaled breath. The analyzers are generally more useful in stream applications where the sample cell is permanently connected to a closed system. However, with the addition of a sample-handling system, batch samples of gas or liquid can be analyzed. The instruments can be readily changed to monitor different components by substituting detectors and, in some cases, by addition of an auxiliary filter. Multichannel instruments are routinely used for monitoring submarine atmospheres where up to five different trace components are continually monitored using a single sample pump with multiple sources and detectors.

12.3 LOGISTICS

12.3.1 Packing and Launch

Generally, infrared analyzers are extremely rugged, requiring only a small additional precaution to insure surviving the launch environment. The instrument is not normally operable under conditions of high vibration and shock due to movement of the diaphragm in the detector. A specially-designed packing crate should be adequate for the infrared analyzer to survive the rigor of launch.

12.3.2 Installation

The instrument should not normally be affected by the absence of a gravitational field, although it is expected that this could result in a zero shift or span change. Inflight calibration, using known gases, would eliminate any problem associated with such a shift. About 150 watts of power is normally required, and a warm-up time of several hours is necessary to permit stabilization of operating temperature. Sample lines must be connected during installation

either to the closed-process system to be analyzed or to a suitably-designed sample-handling accessory. Source operating temperatures exceeding 800°C are used. For this reason, the instrument should only be installed in environments where self-ignition of the atmosphere is not possible.

An RF oscillator is used in some instruments for measurement of the capacitance change of the diaphragm. This could cause potential EMI. Additional shielding may be necessary to prevent unwanted radiation. A connection to space vacuum may be necessary if flushing of the sample or reference cells is required. This is normally necessary between calibration cycles or if batch-type sampling is desired. The instrument housing interface with the spacecraft is dependent upon whether a process or laboratory-type instrument is used. Process versions are preferred where permanent plumbing to a sample stream is desired. Laboratory versions are more adaptable to changes in detectors or sample streams.

12.3.3 Consumable Supplies

Consumable supplies are not normally required for operation of an infrared analyzer with the exception of calibration gases which may be necessary, depending upon the application and desired accuracy.

12.3.4 Accessories and Spare Parts

The major accessory required for infrared analyzers is a sample-handling system for introduction of calibration gases or for changing sample streams. If the unit is to be used for measurement of more than one component, spare detectors charged with the other components of interest must be made available. In some cases, it may be necessary to provide a number of sample cells, particularly

where wide concentration ranges are to be monitored. Special filters are often necessary as accessories for eliminating interferences for specific applications. Spare sources are useful in the event of a burn-out. Most commercial instruments are packaged so that detectors, sources, and/or sample cells are easily interchangeable since this is normally required to achieve versatility.

The spare parts list should include a quantity of fittings and tubing to permit interconnection of the analyzer with various sample streams. Particulate filters may be a valuable addition, particularly for space applications where the absence of gravity could result in unnecessarily high concentrations of solid particulates in the atmosphere. Other spares should include additional windows for the sample cells where interchangeable, and a set of gaskets and seals for the gas sampling and plumbing portions.

12.3.5 Maintenance and Repair

Major repair should not be attempted under space-flight conditions. However, major operating components, such as detectors, sources, sample cells, or plug-in electronic modules, could and should be replaced in the event of a failure or, routinely, where more than one sample component is to be monitored. Due to the requirement for good temperature stability, the instrument heaters should never be turned off if the instrument is to remain stable for long periods of time. A sufficient quantity of calibration gases and filters should be made available so that a simple check can be made on instrument performance. Generally, it is necessary to check both the instrument zero and span. The zero is checked by placing a nonabsorbing sample gas in the cell. The span is tested by placing a known concentration of the specific

constituent, to which the analyzer is sensitized, into the cell. Electronic checks can be performed by blocking one beam or often by internal test switches.

12.4 OPERATION

12.4.1 Warm-up and Speed-of-Operation

The major cause of drift for the infrared analyzer is changes in temperature. Therefore, it is generally necessary to stabilize the operating temperature by warming up the instrument for periods of 30 minutes. This is particularly necessary for applications where trace contaminants are to be analyzed. The sources should be on during this period since a major portion of the total heat into the instrument is from the sources.

Once operating temperature has been achieved, the warm-up for the electronic portion is relatively fast, particularly for units utilizing solid-state electronics.

The output reading is a dc voltage and no spectral scan is required. Time constants on the order of 2 to 10 seconds are typical for standard instruments. Special-purpose instruments such as breath analyzers provide a response of 0.1 second or better. When fast response is required, greater care must be exercised in the handling of the sample lines since this is the major cause of lengthening response time.

12.4.2 Operating Skills

The infrared analyzer is simple to operate and should present no difficulty in a spaceborne laboratory. The operator should be familiar with calibration

techniques which are essential where good accuracy is required. A specially-designed sampling system will probably be required in most cases to interface the sample stream with the instrument cell and the spacecraft vacuum system.

When the instrument has been properly calibrated and the sample introduced, the output reading is continuous and requires no further operator participation. Where more than one component is to be monitored, the operator must familiarize himself with the methods of changing detectors and/or adding filters.

12.4.3 Operating Procedures

The infrared analyzers are relatively simple to operate. The procedure normally involves warming the instrument up to operating temperature and turning on the standby switch. A calibration sample is normally placed in the sample cell so that both span and zero can be adjusted to the desired range. It is then only necessary to substitute the desired sample to be monitored and note the reading on the output meter or recorder. For batch-type sampling, the only operating controls are those required to change samples and flush out the sample cell. Where the instrument is to be sensitized to a different constituent, the operation requires changing the detectors and filters. This is not normally performed during a sample analysis, but is considered a relatively major overhaul requiring recalibration and an additional warm-up step.

12.4.4 Sample Preparation and Handling

Very little sample preparation is generally required prior to introduction into the sample cell of the infrared analyzer. Sample pressures are normally atmospheric, but can range from several atmospheres to vacuum. The sample cell

length must be selected to provide the desired concentration range for a given constituent. Two versions of sample handling are normally available. The process version provides a permanent connection of the sample cell to a closed stream, such as the environmental control system of the spacecraft. The laboratory version requires connection to a sample-handling accessory so that batch sampling can be utilized. It is advisable to include valves and stream splitters in either version so that calibration gases can be supplied. In operation, the sample cell is evacuated and a nonabsorbing gas, such as nitrogen, admitted to the cell. The instrument zero can then be set. A calibration gas is then substituted for the nitrogen and the full-scale sensitivity adjusted to match a known concentration. This gas is then flushed out of the cell and the sample gas of interest admitted.

For liquid analyses, a spectrophotometer is generally more advisable, although there are specific applications where liquid streams are continuously monitored by an infrared analyzer.

12.5 INTERFACE

12.5.1 Interface with Other Laboratory Instruments

The infrared analyzer can be interfaced with any other laboratory instrument which uses a gas sample. The cell volume is relatively large for most gas samples, however, making it less desirable than other techniques for use as a gas chromatograph detector. Small cell versions, such as the CO_2 breath analyzer models, would be quite useful for measuring the effluent of a gas chromatograph, however. The sample is not destroyed; therefore, once the analysis has been

performed using the infrared analyzer, the sample can be transferred to another instrument, such as the mass spectrometer, without degradation of sample characteristics.

The output of the infrared analyzer is generally an analog voltage, which is displayed on a meter or recorder. It can also be made available to the spacecraft data management system for telemetry or storage.

Major interfaces will probably include special sampling systems and interconnection to readout consoles or the spacecraft telemetry system. In some cases, the instrument may require a special interface for a specific medical experiment, such as a respiratory measurement where the inlet system would be connected to a face mask and associated hardware.

12.5.2 Interface with Vehicle Systems

Services required for the infrared analyzer are 115 V, 60 Hz power, and a source of vacuum for the sample-handling system where calibration or sample flushing is necessary. The power should be left on continuously or, as a minimum, the standby power maintained in order to insure constant temperature control.

12.6 SAFETY

12.6.1 Flame Hazards

Infrared analyzers do not present flame hazards.

12.6.2 Sample System Hazards

The sample accessory will normally include a vacuum connection to provide proper flushing of sample lines and interchange of samples. Connections to the sampling accessory could be broken and should be constructed of stainless steel tubing wherever possible. Portions of the inlet system may be connected to space vacuum, and any break in this sampling line could result in a hazard to the laboratory atmosphere. A pressure-monitoring device should be added, including the necessary alarms in the event of rapid pressure drop.

A leak from the calibration gas cylinders could represent a potential hazard if utilized in a closed atmosphere of small volume. This should be prevented by sizing the calibration gas cylinders so that even a catastrophic leak could not result in a toxic or unsafe atmosphere.

12.6.3 Electromagnetic Interference

Most infrared analyzers use an RF oscillator for measurement of detector diaphragm movement. This could result in EMI, particularly since most commercial instruments use the commercial radio frequency band. Adding more shielding and filters to the power line will prevent serious interference with radio equipment.

Operation of transmitter equipment in the frequency bands associated with the detector electronics would result in possible interference causing sizeable tional shielding where necessary. This may be of no consequence if the communication center of the laboratory is more than 20 or 30 feet away from the measurement laboratory.

12.6.4 Ionizing Radiation

Ionizing radiation is neither produced by nor does it interfere with the operation of the infrared analyzers.

12.6.5 Physical Hazards to Personnel

Exposed vacuum lines which could be broken by accidental contact could represent a physical hazard to personnel. The operating controls of the infrared analyzers should be reviewed for ease-of-operation under zero-gravity conditions, and may require replacement of knobs or relocation in some cases.

The presence of high-temperature sources within the instrument case represents a hazard if physical contact is made with the source or its housings during operation. The rotating chopper wheel could be a hazard to fingers during operation. Generally, operating voltages are sufficiently low to not present any hazard except at the power interface with the spacecraft.

12.7 MODIFICATIONS

Following are typical modifications which are needed for most commercial infrared analyzers to permit operation in the Space Station:

1. Instruments containing sample pumps should be replaced with suitable vacuum lines and valving to permit space-vacuum operation.

2. A human-factors analysis should be made for each instrument, and any sharp corners eliminated and operating controls carefully reviewed to insure ease of operation under conditions of zero gravity.

3. Special sample-handling and calibration equipment should be designed to eliminate requirements for loose gas bottles and other hazards associated with space environments. An all-stainless steel system is preferred with permanent hard lines to calibration volumes and instrument sample cells.

4. Additional clamps and straps may be necessary to tie down circuit boards and analyzer components during launch.

5. All materials exposed to the cabin environment should be reviewed for possible outgassing and contamination of the breathing atmosphere.

6. Additional shielding and light filters must be added to reduce electromagnetic interference.

7. The effect of zero gravity on the thermal characteristics should be considered, particularly since good temperature control is required for stable operation.

12.8 AVAILABLE INSTRUMENTS

Following is a list of manufacturers of infrared analyzers which might be useful for the spaceborne laboratories:

Beckman Instruments, Inc.
Leesona Corp.
Mine Safety Appliances

Section 13
MASS SPECTROMETERS

13.1 PRINCIPLES OF OPERATION

The mass spectrometer is based on the physical separation of electrically charged particles according to their mass by the action of magnetic and/or electric fields. Ions are formed by bombardment of the sample gas with a sharply defined electron beam in an ionization chamber operating at vacuum pressures. The resultant ions are drawn from the ionization region by means of negatively charged plates and focused into a narrow beam. In the magnetic-sector-type instrument, the ions emerging from the ionizing region are curved by the action of the magnetic field and ions of different masses are separated into beams of different radii. The radius of curvature (r) is related to the ion mass (m), the accelerating voltage (v) and the magnetic field (H) by the equation $H^2r^2 = KMV$. A collector slit is used in front of a collector plate so that only ions of the desired mass are allowed to strike the plate and discharge. The resulting current is proportional to the quantity of ions striking the collector. The quantity of ions striking the collector produces a signal current proportional to the quantity of the input sample with a particular mass. A mass scan is achieved by varying either the magnetic field strength or accelerating voltage.

The quadrupole is the most popular type of electric field analyzer. This analyzer consists of four rods to which dc and RF potentials are applied. Ions are directed down the space between the rods and parallel with them. For

a given RF frequency and applied dc potential, only one mass can reach the collector plate placed at the end of the rods, all other ions striking the rods become non-synchronous with the RF field. A mass scan is achieved by varying the dc/RF voltage ratio.

The resolving power of a mass spectrometer refers to the instrument's ability to separate adjacent masses. Instruments for light gas analysis require resolution up to mass 100, whereas those for high molecular weight determinations require resolutions of several thousand. Since the operation of the mass spectrometer must be at low pressures (below 10^{-5} mm of Hg), it is necessary to condition the sample prior to introduction. This is performed either by expanding the sample to lower pressures in combination with a molecular leak or by using a capillary and leak system which permits atmospheric samples to be introduced continuously.

Mass spectrometers require operation under high vacuum. This normally requires that a mechanical forepump be used ahead of the molecular pump. It is desirable to consider the use of space vacuum to supply the fore-pressure, since a mechanical pump will not operate under zero gravity conditions. Diffusion pumps cannot be used since the pump fluid will not "percolate" properly without gravity. An ion pump is the only suitable device.

There are generally two types of detectors used in mass spectrometry. The majority of instruments use a collector plate in series with a high value resistor. Ions are discharged upon hitting the plate and the resultant flow of electrons produces a voltage across the resistor which is amplified by means of an electrometer. Typical currents range from 10^{-9} to 10^{-14} amperes.

Quadrupole type instruments generally use an electron multiplier detector. The collector plate consists of an alkaline Earth surface which produces secondary emission. The electrons are collected on a series of dynodes similar to a photomultiplier. Although this detector requires a high voltage for operation, the output current is on the order of 10^{-6} amperes maximum and less amplification is required. The noise level and instability of the electron multiplier is generally higher than the electrometer type but much faster response times can be achieved.

The output of a mass spectrometer can be displayed on a meter or a recorder chart. Where a mass scan is desired, it is useful to generate the mass spectrum by recording the entire mass scan. For applications where only one or two components are to be monitored, a step scan can be supplied or the instrument may be tuned to a single mass and a continuous measurement obtained. In some applications it may be desired to interface the mass spectrometer with the data management system.

13.2 APPLICATION

The mass spectrometer could be used in the following functional program elements (FPE's):

- 5.9 Small Vertebrates (Bio D)
- 5.13 Biomedical and Behavioral Research
- 5.15 Life Support and Protective Systems
- 5.17 Contamination Measurements
- 5.18 Exposure Experiments
- 5.23 Primates (Bio A)
- 5.27 Physics and Chemistry Lab

The mass spectrometer is particularly useful for light gas analysis and is a qualitative detector for a gas chromatograph. Applications in space might include trace contaminant analysis, atmospheric control, and breath analysis for biomedical experiments.

The mass spectrometer is particularly useful for the analysis of gas mixtures where a separate mass peak can be selected for each component of interest without superposition or overlap. In the case of atmospheric monitoring, for example, separate peaks can be selected for oxygen, nitrogen, carbon dioxide, and water. Carbon monoxide, on the other hand, cannot be monitored in the presence of nitrogen since both peaks overlap and the large N_2 peak would swamp the presence of a CO contaminant.

For trace contaminant identification, the mass spectrometer should be combined with another analytical technique because of the larger number of fragmentation peaks which occur when analyzing complex mixtures of higher molecular weight.

With proper signal conditioning and sampling design, the speed of response of the mass spectrometer can be made very fast so that breath-by-breath analysis is feasible for medical applications. The gas consumption of the sample gas is extremely small, making the mass spectrometer particularly useful for experiments involving gas analysis of effluents from biological experiments.

13.3 LOGISTICS

13.3.1 Packing and Launch

During launch, any shock mounts used must be tied down securely to prevent damage. Prior to launch, the instrument will normally be pumped down and sealed off to maintain a clean vacuum system. The instrument is generally quite rugged and would require no special packing other than that normally provided for railroad shipment.

13.3.2 Installation

Standard commercial mass spectrometers are generally bulky and require external power for operation and heating of the vacuum system during the bake-out procedure. Space vacuum for sample handling and roughing should be supplied at vacuum pressures below 10^{-3} mm Hg, since it is not expected that mechanical forepumps could be used in a zero gravity environment.

The instrument analyzer is not sensitive to gravitational fields and, therefore, can be mounted in any position in the spacecraft. The large size and mass of commercial mass spectrometer units would require that they be suitably fastened to the spacecraft bulkheads.

The readout device can be conveniently located separately from the mass spectrometer console. The type and location of the installation is dependent upon the application of the instrument and the type of mass spectrometer being supplied. Magnetic units, for example, should be located away from experiments where changing magnetic fields could affect the results. RF fields from the quadrupole type may require additional shielding to meet EMI requirements for

the laboratory; however, the envelope of the vacuum chamber provides considerable shielding.

13.3.3 Consumable Supplies

Consumable supplies are not required for operation of a mass spectrometer with the exception of calibration gases which may be desirable, depending upon the application of the instrument. The extremely small gas consumption of the mass spectrometer permits the use of small volumes for calibration gases.

13.3.4 Accessories and Spare Parts

Major accessories for the mass spectrometer will be sampling devices. The type of application for which the instrument will be used determines the sampling system configuration. For atmospheric monitoring, capillary inlets are generally satisfactory for reducing the sample pressure to the required vacuum pressure. An external vacuum is usually necessary for introducing the test sample into the sampling system. This should be available from space vacuum, however, since the size of the line required to provide the necessary flow is less than 1/4 inch in diameter. Other accessories would include a valving manifold for introducing calibration gases or special adapters, such as breathing masks for biomedical experiments.

Spare parts would normally include additional filaments for the ionization chamber, although it may be more practical to replace the entire analyzer head rather than the filament so that the delicate task of changing filaments is not performed under space conditions.

A readout device, such as a recorder, is particularly useful for initial setup and calibration of the mass spectrometer. In operation, the instrument may be set on a single peak or the scan may be telemetered or recorded on magnetic tape. In either case, it is necessary to observe the mass spectrum prior to operation to insure proper resolution, sensitivity, and mass range. The quadrupole type of instrument generally scans sufficiently fast so that an oscilloscope can be used for this purpose.

Additional spares would include molecular leaks for the inlet system which can be easily clogged by samples containing aerosols or particulates. A leak detector is often a valuable accessory, although if adequate vacuum can be maintained, the mass spectrometer itself is an excellent leak detector if a tracer gas such as helium is available.

A set of gaskets, O-rings, and vacuum valves is a valuable addition to the spare parts kit. Special wrenches required to dismantle the analyzer should be included in the spare parts kit.

13.3.5 Maintenance and Repair

Vacuum system repair is the major maintenance problem associated with mass spectrometers. Even small vacuum leaks can cause major problems in operation. The maintenance cycle must include a schedule for baking out the instrument and calibrating it at least on a weekly basis. Ionizing filaments require changing frequently, if the instrument is used to sample the atmosphere or any gas containing a large amount of oxygen. For space maintenance, it is desirable to change entire subassemblies, such as the analyzer head or

detector and preamplifier. Spare circuit boards should be available where plug-in design is used. In the case of quadrupole instruments, the entire analyzer is generally replaceable, although major recalibration is often necessary if such a drastic modification is made. The instrument should be routinely placed in a standby operation mode when not being used for gas sampling. This involvolves closing the inlet system and possibly turning off the filament and electronics. The vacuum system should never be turned off if at all possible.

13.4 OPERATION

13.4.1 Warm-up and Speed-of-Operation

If vacuum pressure has been established, the instrument vacuum system should never be turned off except during maintenance cycles. Under these conditions, the warm-up time is less than 10 minutes, again depending upon the mass spectrometer design and application. The ionization chamber warms up after the filament is turned on and generally requires up to one hour to stabilize in temperature. This is of particular concern where high accuracy is required. In cases where the vacuum system must be shut down between experiments, at least one hour should be allowed for pumping down the instrument. Where heated inlet systems are used, at least 30 minutes is required to attain the proper operating temperature.

A mass scan can be achieved in milliseconds using a quadrupole instrument or may require up to one hour for the higher resolution, wide mass range instruments. For light gas applications, a mass scan can generally be achieved in seconds unless extremely high sensitivity is required. Resolution and

response time are generally inversely proportional to each other. The maximum response speed is generally limited by the acceptable noise for the system.

The sampling system can be designed with less than 0.1-second response characteristic, although sample lag times are generally 1 to 2 seconds. For applications where the instrument is set to a single mass peak, the response time is generally limited only by the sampling system characteristic and the time constant of the electrometer amplifier.

13.4.2 Operation Skills

The operation of a mass spectrometer in a spaceborne laboratory should be no more difficult than an earth-based unit. The operator must be experienced with the operation of a mass spectrometer and the interpretation of the mass spectrum. In particular, he must be skilled with the operation of vacuum systems since proper operating pressures can only be obtained if all of the operating valves and pumps are set to the proper modes in a preselected sequence. When the instrument has been set up and calibrated, operation for specific experiments is relatively simple and requires only a few hours of training for inexperienced personnel. High resolution instruments generally include increased versatility in operating modes such as mass range selection, resolution, sensitivity, range, operating pressures, ionization currents, scanning speeds, etc. The training required for these more complex instruments is considerably longer. Mass spectrometers have been designed specifically for space applications and require only the operation of two to three switches and valves. This reduced versatility obviously makes the

instrument useful for fewer applications and, in general, for only one selected application.

13.4.3 Operating Procedure

The operating procedures of a mass spectrometer are dependent upon the type of sampling system and the desired scanning mode. Generally, the sample gas is admitted continuously by means of a capillary inlet system and the instrument is either set on a single mass peak to monitor a single component or it is permitted to scan over a selected mass range. Where unknown components are to be determined, it is generally necessary to calibrate the instrument for sensitivity and mass range and to run a background spectrum so that gas components adhere to the walls of the vacuum system and do not affect the analysis of the sample gas.

A typical operating procedure might be as follows:

Preparation:	Turn on vacuum system. Insure proper vacuum pressure. Turn on filament. Open inlet valve and set sample pressure. Set mass range and resolution. Set sensitivity desired.
Calibration:	Introduce known calibration gas into inlet. Perform mass scan. Record peak heights and set attenuation accordingly.
Measurement:	Introduce sample gas. Turn on mass scan. Observe for proper resolution and on-scale sensitivity. Record.

The versatility of the mass spectrometer permits a wide variation in sampling technique, mass range selection, sensitivity, resolution, and specificity; therefore, no single procedure is adequate for all applications. In all cases, the instrument should be calibrated with a known gas which is selected to provide an indication of resolution and sensitivity over the desired mass range.

13.4.4 Sample Preparation and Handling

Only a gas sample is being considered since solid sampling is relatively complex and probably has little application for the spaceborne laboratories. Liquid samples must be vaporized prior to introduction into the instrument. This can be performed by expanding to lower pressures using a sample handling system containing multiple flask volumes.

For most applications, sampling will be performed directly. The sample line is a small capillary tube, one end of which is connected to the molecular leak which introduces the sample into the ionization chamber. A larger tube is connected at this point to space vacuum for purposes of drawing the test sample through the capillary and providing a sample pressure at the molecular leak of 1 mm Hg or less. When sampling gases, such as the atmosphere or the head space of closed environment experiments, no sample preparation is generally required. To properly set the operating characteristics, the instrument must be capable of sampling known calibration gases. No special precautions are necessary for sample handling under conditions of zero gravity with the recommended capillary inlet system. If liquids are to be sampled, a specially designed sample handling system can easily be provided to condition the sample to the proper operating pressure. Where large amounts of water

vapor are to be sampled, it is desirable to heat the inlet capillary system to prevent condensation on the walls which could interfere with the response time of the instrument and cause excessive background readings for water vapor.

13.5 INTERFACE

13.5.1 Interface with Other Laboratory Instruments

The mass spectrometer can be interfaced with any other laboratory instrument which utilizes a gas sample. The instrument is particularly useful for monitoring the effluent of a gas chromatograph since the gas chromatograph column provides the separation of each constituent in the sample gas. The mass spectrometer in this case observes a single constituent at a time thereby providing maximum quantitative accuracy. The mass spectrometer is also useful for analyzing samples from an infrared analyzer. In this case, the infrared spectrum can be compared with the observed mass peaks and relatively complex molecules rapidly identified.

The output of the mass spectrometer is generally an analog voltage which can be displayed on an oscilloscope or recorder, or it can be made available to the spacecraft data management system. Where single peaks are being monitored, a meter is often adequate. The output is linear with partial pressure for most gases.

An additional interface will probably be required to the readout console and/or telemetry. There may be additional interfaces to specific experiments such as a Metabolic Analyzer, for example, where the inlet system would be connected to a face mask for sampling breath.

13.5.2 Interface with Vehicle Systems

Services required for the mass spectrometer are 115 V, 60-Hz power, and a source of vacuum for the sample inlet system and the forepressure. In operation, the inlet system requires a continual vacuum source with a flow rate of 0.5 to 2 cc per second at STP. With the use of a molecular ion pump, no external vacuum is required except during the roughing stage. A vacuum line of at least 1.5 inches diameter and no more than 2 feet long to space is desirable to achieve the operating pressure prior to operation of the molecular pump.

13.6 SAFETY

13.6.1 Vacuum System Hazards

Portions of the mass spectrometer inlet system may utilize glass or flexible tubing which can be easily broken. Protection must be provided so that the vacuum is not broken during operation. Since portions of the inlet system will be connected to space vacuum, any break in this sampling line could result in a hazard to the laboratory atmosphere. Vacuum gauges will be necessary to continuously monitor operating pressure and should provide an alarm in the event of vacuum failure. The vacuum pressure monitoring equipment should be in operation at all times or whenever the instrument is connected to space vacuum.

13.6.2 Electromagnetic Interference

The magnetic mass spectrometer may produce a large varying magnetic field in the case of the electromagnetic units. Where a permanent magnetic is used, a varying dc voltage is applied for scanning purposes. With the quadrupole

units, a high frequency RF oscillator is required which is changing amplitude and/or frequency with the scan. It would be necessary to provide additional shielding for standard commercial units since none of them provide adequate electromagnetic interference protection for space vehicle use.

If the laboratory is located adjacent to high power transmitters or radar, additional shielding may be necessary to prevent operation of this equipment from affecting the performance of the mass spectrometer. Additional line filtering will be necessary to prevent RF currents from entering the main power lines. EMI requirements will, in general, have to be reduced or considerable redesign of commercial mass spectrometers would be required. It may be possible to plan operating times of various laboratory equipments so that mutual interferences do not exist. It may also be possible to screen the laboratory environment so that relatively high interference signals can be handled.

13.6.3 Ionizing Radiation

The mass spectrometer will not produce any ionizing radiation. However, the presence of cosmic rays or other high energy particles in the space environment could produce additional noise since the detection system of a mass spectrometer can respond to the presence of any ions which strike the collector plate. This may be particularly important with the use of electron multiplier detectors where the presence of additional collector or dynode plates can produce a large multiplication of stray ions.

13.6.4 Physical Hazards to Personnel

Exposed vacuum lines which could be broken by accidental contact could represent a physical hazard to flight personnel, particularly if glass or shatterable material is used. The operating controls of each mass spectrometer should be reviewed in terms of ease-of-operation under zero gravity conditions and, in most cases, will require replacement by more suitable knobs. Concise operating procedures should be included to prevent accidental breakage of the vacuum system by personnel relatively unfamiliar with the instruments.

The presence of high voltage for the multiplier detector and ion pumps could represent a hazard. More protection from these high voltages should be provided in some instruments.

13.7 MODIFICATIONS

Following are typical modifications which are needed for most commercial mass spectrometers to permit operation in the Space Station:

1. Units containing a diffusion pump must be replaced by a molecular or ion pump.
2. Sampling systems utilizing mercury sealing must be redesigned to eliminate the mercury.
3. All forepumps should be removed from the equipment and replaced with suitable vacuum lines and valving for space vacuum operation.
4. All inlet systems and vacuum monitoring equipment should be rebuilt to eliminate glass wherever possible and any requirement for liquid coolant.

5. A human-factors analysis should be made for each instrument and any sharp corners eliminated and operating controls carefully reviewed to insure ease of operation under conditions of zero gravity.

6. Special sample handling and calibration equipment should be designed to eliminate requirements for loose gas bottles and other hazards associated with space environments. An all-stainless-steel system is preferred with permanent hard lines to calibration volumes.

7. Any requirement for cooling water should be eliminated be replacement of the component and/or substitution of adequate trapping.

8. Additional clamps and straps may be necessary to tie down circuit boards and analyzer components during launch. Any components which utilize gravity for operation or positioning must be remounted.

9. All materials exposed to a cabin environment should be reviewed for possible out-gassing and contamination of the breathing atmosphere. This is particularly necessary for heating blankets which can evolve toxic materials during the bake-out procedure.

13.8 AVAILABLE INSTRUMENTS

Following is a list of manufacturers of mass spectrometer equipment which might be useful for the spaceborne laboratories:

Manufacturer	Type
Aerovac Corporation	Magnetic
Bell and Howell Company	Magnetic
Bendix Corporation	Time of Flight
Electronic Associates, Inc.	Quadrupole

Manufacturer	Type
Extranuclear Products	Quadrupole
Finnigan Instruments, Inc.	Quadrupole
GCA Corporation	Magnetic
Jeolco, Inc.	Magnetic

Table 13-1 shows appropriate instruments and their specifications.

Company	Model	Price ($)	Type	Mass Range (amu)	Resolution at mass 28 (nitrogen)
Aero Vac Corp	685	17,000	SF (MAG)	2 - 300 amu	250
Bell & Howell Co.	CEC 21-104	40,000 to 60,000	SF	1 - 2,000	1/2, 500
Bendix Corp.	MA-2	27,500	Time-of-flight	1 - 1,500	500
Electronic Assoc., Inc.	Quad 300	20,495	Q	1 - 800	Unit
Finnigan Instruments Corp.	1015	19,900 to 25,000	Q	750, 1000 opt	56
Nuclide Corp.	12-90-G & 12-90-G(DF)	40,000 to 60,000 SF; 75,000 to 100,000 DF	SF, DF	1 to over 6,000	10,000 SF, 35,000 DF
Perkin-Elmer Corp.	801-1523	7,950	Q	1 - 300 amu	$m/\Delta m = 2$
	RMS-4	25,000	SF	1 - 1,200	1,000
Picker Corp.	MS10c2	22,000	SF	1 - 600	400
Varian Associates	CH-7	24,000 to 35,000	SF	1 - 3,600	3,000 (10% valley)

Table 13-1. Mass Spectrometers

Section 14
MICROSCOPES

14.1 PRINCIPLES OF OPERATION

The operation of optical microscopes is sufficiently widely understood that it need not be discussed in detail here. An image of small objects or a thin layer of material is optically magnified for direct viewing or photography. The typical laboratory microscope has an assortment of objectives (4X to 100X) which are usually mounted on a turret for rapid interchange and interchangeable eyepieces (5X to 20X). The optical magnification of the microscope is the product of the power of the objective and eyepieces. The image of the standard laboratory microscope is reversed, and the light source normally shines through the specimen into the optical system.

In the metallographic microscope, the specimen is illuminated by reflected rather than transmitted light. The light source is introduced at right angles to the optical path and is thereafter concentric with the optical path, converging on the specimen along the same axis as the optic path. Special objectives are used to handle both the illuminating light and the optical image; with some microscopes, there is a choice of bright field or dark field illumination.

The inverted microscope is a variant of the standard laboratory microscope particularly adapted to tissue culture observation. The stage is accessible in a relative open area, and the objectives are below the stage. The placement of the stage with respect to the optical and illumination systems is particularly

advantageous for keeping the specimen in an incubator or glove box during operation. Although the inverted microscope does not have the conventional microscope configuration, it should not be excluded from consideration as a general-purpose instrument. It accepts many of the accessories of the standard microscope and is more convenient to use for many applications.

An additional type of microscope in common use is the stereo or dissecting microscope. The stereomicroscope typically has lower magnification and longer working distance than a standard microscope. They have dual-optical systems and present unreversed, true stereoscopic image. Many standard microscopes have twin eyepieces but do not produce a true stereo image.

14.2 APPLICATIONS

Microscopes are applicable in the following functional program elements (FPE's):

- 5.9 Small Vertebrates (Bio D)
- 5.10 Plant Specimens (Bio E)
- 5.13 Biomedical and Behavioral Research
- 5.15 Life Support and Protective Systems
- 5.16 Materials Science and Processing
- 5.17 Contamination
- 5.18 Exposure Experiments
- 5.23 Primates (Bio A)
- 5.25 Microbiology (Bio C)
- 5.26 Invertebrates (Bio F)
- 5.27 Physics and Chemistry Lab

These instruments could also be used in the Bioscience, Biomedical, general-purpose, and physics and chemistry laboratories.

Microscopes are applicable for use in a wide spectrum of biological and physical experiments. They are used to view and photograph living, fresh, and stained biological specimens, and they are used to examine surfaces and small particles.

Dissecting microscopes are invaluable for visual feedback during fine manipulations whether dissecting, assembling, or analyzing. A wide range of attachments and accessories (Paragraph 14.3.4) are available to extend the microscope's versatility (Figure 14-1).

Phase-contrast and interference techniques should be given particular attention with respect to biological observations. These techniques allow detailed observation of unstained tissue and cellular samples, thereby decreasing the need for complicated zero-g staining techniques.

Unstained specimens such as bacteria, living cells, tissue cultures, smears, and thin tissue sections can be made visible in phase contrast illumination and viewing conditions, thus permitting clear viewing of the shape and structure of the specimen. Interference contrast techniques provide similar advantages and additionally provide a "shadow-cast" effect. The shallow depth of field characteristic of interference methods provides an "optical sectioning" of relatively thick objects.

14.3 LOGISTICS

14.3.1 Packing and Launch

Microscopes do not require particularly different packing procedures than other precision photo-optical instruments. Standard shipping containers should be adequate for the microscopes and all accessories.

14.3.2 Installation

Unpacking a microscope is routine. Since microscope accessories include many small parts, storage containers will be needed. Foam-filled containers with cut-outs for individual pieces are often used for earth-based laboratories; storage in such containers would provide both impact-resistance and also a small amount of friction to keep the pieces in their holes. The same container could be used for shipping.

Some hold-down device, not normally present on earth-based instruments, would be needed to keep the microscope on the workbench. This could be mechanical or magnetic; it need not resist much force.

14.3.3 Consumable Supplies

The only consumable supplies needed for microscopes would be immersion oil, but even this could be unusable in a zero-gravity environment. Other consumables would be experiment-oriented rather than instrument-oriented. Film would be needed for microphotography.

14.3.4 Accessories and Spare Parts

There is a large array of accessories available for most lines of general-purpose microscopes. There are dozens of objectives, eyepieces, and condensers in different magnifications and for different applications. Phase-contrast,

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*Immersion oil is used as an optical coupling between a slide and a high-magnification objective. It eliminates air-glass interfaces and contributes its retractive index to the optical system.

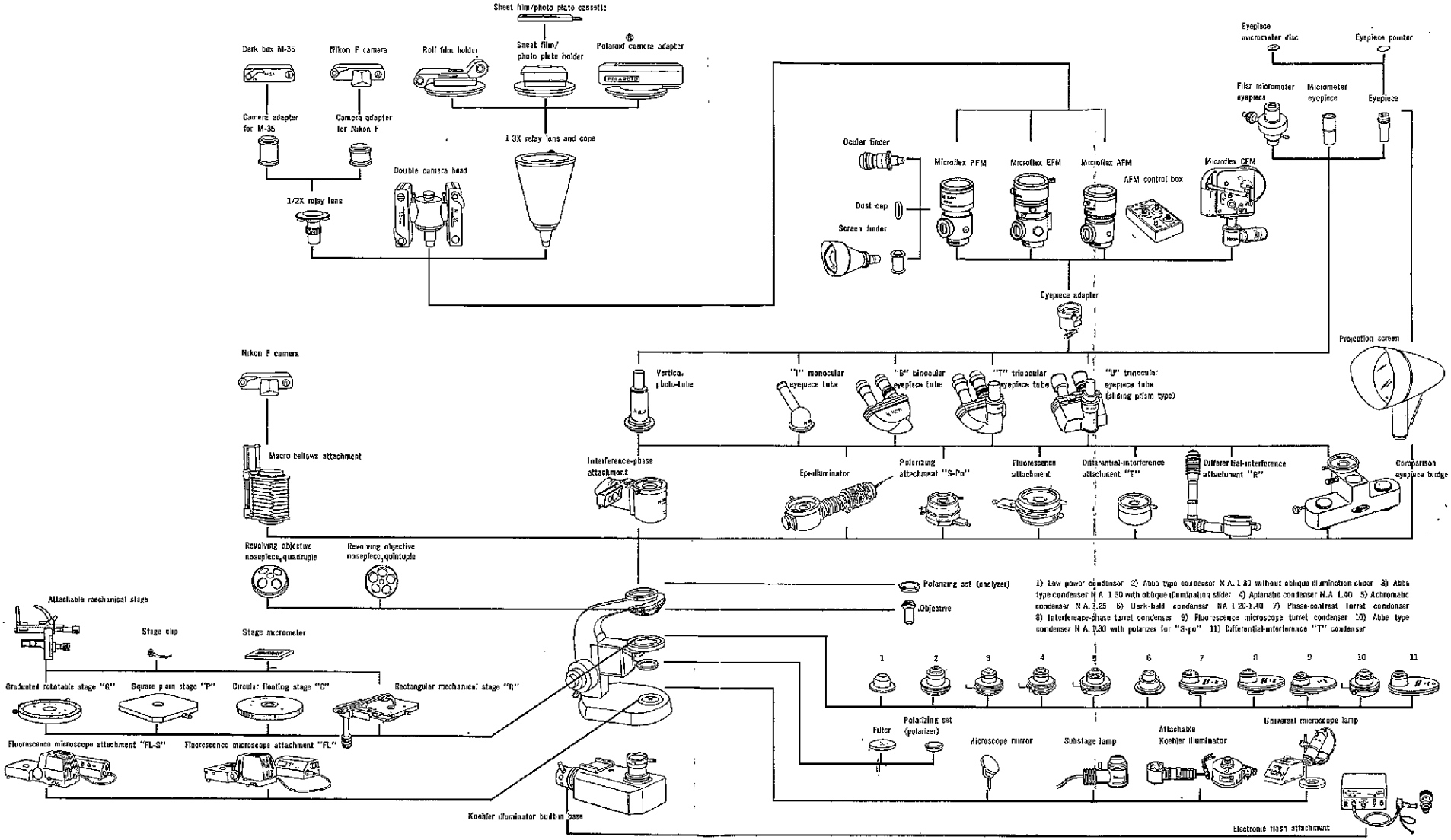


Figure 14-1. Microscope Applications

FOLDOUT FRAME 1

FOLDOUT FRAME 2

interference-phase, differential-interference, and polarizing modes of operation are available for many instruments with attachments to the basic instrument. (Phase and interference techniques are extremely useful for viewing unstained tissue samples). Micrometer attachments are available for measuring small objects and distances. Most general-purpose microscopes provide for microphotography, directly or with attachments, onto most standard formats (16 mm, 35 mm, 120, 4 x 5 in., Polaroid, and video). Special lighting, color balance adjustment, light intensity measurement, and automatic exposure are features or accessories used in conjunction with microphotography.

The major spare parts needed for microscopes are extra bulbs for the various illuminators. Most other microscope parts neither break nor wear out with normal useage, although a complete backup instrument may be desirable.

14.3.5 Maintenance and Repair

Adjustment and calibration of microscopes require skilled technical personnel and the optics test facility (see Section 16). The frequency of need for service would decrease with higher priced and higher weight microscopes. The decreased need for service, especially in high-precision applications, should be considered a worthwhile trade-off for the increased price and weight.

14.4 OPERATION

14.4.1 Warm-up and Speed-of-Operation

No warm-up time is needed for microscopes. Speed of operation does not particularly depend upon instrument characteristics; it is, rather, a perceptual

function, varying with the individual and the task. Some tens of minutes may be required for set-up and adjustments.

Microphotography may require exposures up to several minutes. Time lapse photography may require several hours to produce a few minutes of footage.

14.4.2 Operation Skills

Only a relatively brief period is necessary to train personnel to obtain an image on a microscope. Much professional experience is needed for more sophisticated use of a microscope. Certainly years of training and experience are required to effectively use the information derived from the microscopel

14.4.3 Operating Procedure

The operating procedures tend to be inseparable from the skills of the scientist using them. Operating a microscope, like operating a pencil, is on the one hand so trivial and on the other so diverse that it cannot be discussed briefly.

14.4.4 Sample Preparation and Handling

Sample preparation, especially in the case of biological specimens, is a major aspect of microscopy generally. A thin layer is prepared on a slide by sectioning a tissue sample (Section 15, Microtomes) or spreading a layer of liquid on a glass slide. This is then stained for viewing; the problems of tissue staining in a zero-gravity environment are considered in Section 25.

In an earth-bound laboratory, a stained glass slide is placed on the microscope stage and is held there by gravity; it may be moved by the arms of a mechanical

stage. For space station use, it must be actively held in the focal plane (the stage surface); a slight increase in friction may be adequate for this. This friction could be provided by rubber pads on the arms of the mechanical stage.

There are also problems in holding loose specimens on the microscope stage for observation. Solutions to this problem must follow from the nature of the specimen and the need to move it under the microscope. Solutions to these individual problems will include hold-down clamps, magnets, adhesives, etc.

14.5 INTERFACE

14.5.1 Interface with Other Laboratory Instruments

Microscopes are generally used alone, although in some instances they may be used with, or as a part of, some other laboratory instrument. Examples of the latter would include the microspectrophotometer, the use of a stereomicroscope for positioning microelectrodes for physiological recording, or other micromanipulations.

14.5.2 Interface with Vehicle Systems

For most applications, microscopes require only electric power for their lamps. Some situations may include an incubator or glove box surrounding the microscope stage. This would require interface with the LS/EC System. If the standard high-pressure mercury bulb is used for an ultraviolet light source, it would be well to isolate this from the cabin environment.

14.6 SAFETY

14.6.1 Flame Hazards

Microscopes present no flame or flamability hazard; however, the mercury lamp used for fluorencence techniques does present some potential explosion hazard. This high-pressure bulb should be isolated from the cabin environment.

14.6.2 Microbiological Hazards

The presence of microorganisms presents a hazard only if they are allowed to float from their culture containers into the spacecraft cabin environment. Care should be taken to be sure that this situation does not occur.

14.6.3 Electromagnetic Interference

Standard microscopy techniques neither produce nor are interfered with by electromagnetic interference.

14.6.4 Ionizing Radiation

Standard microscopy techniques neither produce nor are interfered with by ionizing radiation. Autoradiographic preparations may require additional shielding during exposure.

14.6.5 Physical Hazards to Personnel

The major physical hazards to personnel arise from protruding parts of the microscope or hot-lamp housings.

14.7 MODIFICATIONS

The following modifications are recommended for microscopes used in the space-station environment:

1. A positive latch or increased friction to hold eyepieces in their tubes (they are normally held in by gravity).
2. Stage modification to hold slides and specimens.
3. Isolation of high-pressure mercury bulb from cabin environment.
4. Device to hold microscope on work bench.

14.8 AVAILABLE INSTRUMENTS

Because of the flexibility of general-purpose microscopes and the diversity of configurations available with available accessories (Figure 14-1), this survey does not attempt to tabulate instruments and specifications. Generally, any configuration and mode of operation can be achieved by interchanging the parts of instruments available from any one of the major manufacturers. This does not imply that all microscopes are equal in performance. There are qualitative differences between instruments which are generally (but not completely) related to their price. The following manufacturers produce laboratory-quality, general-purpose microscopes which will fill any anticipated need for space-station instrumentation:

American Optical Company	Unitron Instrument Co.
Bausch & Lomb	Vickers Instrument Co.
E. Leitz	Wild Heerbrug Instruments
Nikon	Carl Zeiss
Olympus Corp.	

One particularly different instrument is the Model H Portable Microscope, manufactured by Nikon. This instrument might find several applications because of its portability.

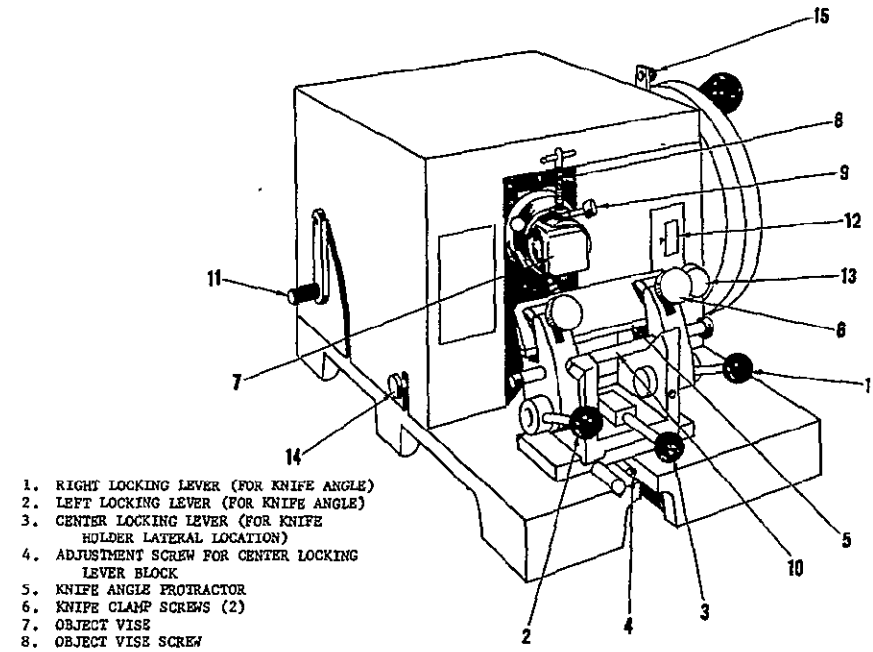
Section 15
MICROTOMES

15.1 PRINCIPLES OF OPERATION

Microtomes are devices for cutting fixed (embedded or frozen) tissue specimens into thin (1 to 50 micron) sections. The two basic types of microtomes are rotary and sliding (Figure 15-1). Rotary microtomes operate by holding the knife fixed and moving the specimen across it. The specimen is moved manually, rotating a flywheel connected by cams and levers to the specimen stage. Sliding microtomes move the knife across the fixed specimen. With both types, the specimen is moved a fixed distance before the beginning of each cutting stroke. Since electron microscopes have not been considered in this survey, ultramicroscopes have been excluded.

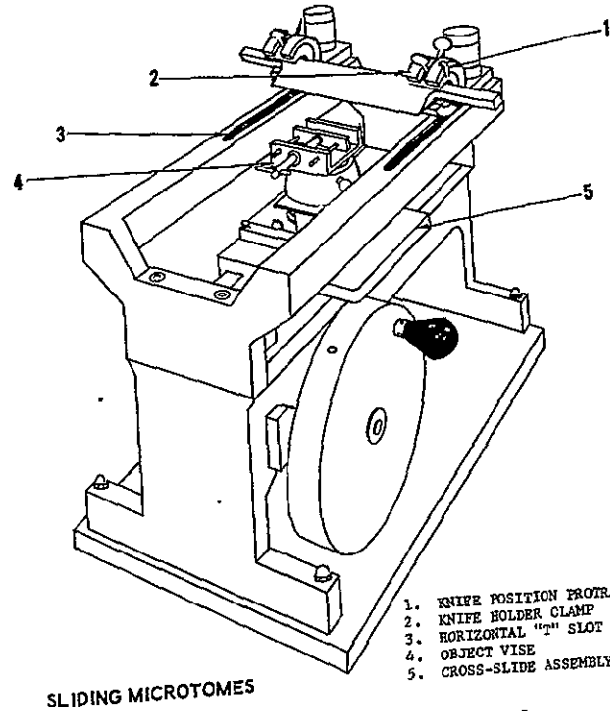
Several microtomes are supplied with freezing stages which allow sectioning of fresh tissue frozen on the stage. Standard freezing stages use a CO_2 coolant, but some new models cool by the Peltier effect; considerable current is required for the latter.

A new type of vibrating microtome, the Vibratome, is manufactured by Oxford laboratories. This device cuts fresh, unfixed tissue with common injector razor blades. The device operates by vibrating the blade along its long axis, thereby sawing rather than cutting the specimen.



1. RIGHT LOCKING LEVER (FOR KNIFE ANGLE)
2. LEFT LOCKING LEVER (FOR KNIFE ANGLE)
3. CENTER LOCKING LEVER (FOR KNIFE HOLDER LATERAL LOCATION)
4. ADJUSTMENT SCREW FOR CENTER LOCKING LEVER BLOCK
5. KNIFE ANGLE PROTRACTOR
6. KNIFE CLAMP SCREWS (2)
7. OBJECT VISE
8. OBJECT VISE SCREW
9. OBJECT VISE ORIENTATION SCREWS (3)
10. KNIFE RAISER CAM
11. FORWARD FEED AND REVERSE CRANK
12. MICRON INDICATOR
13. MICRON ADJUSTMENT DIAL
14. COVER LOCKING SCREW
15. HAND WHEEL LOCKING LEVER

ROTARY MICROTOMES



1. KNIFE POSITION PROTRACTOR
2. KNIFE HOLDER CLAMP
3. HORIZONTAL "T" SLOT
4. OBJECT VISE
5. CROSS-SLIDE ASSEMBLY

SLIDING MICROTOMES

Figure 15-1. Microtomes

15.2 APPLICATIONS

Capable of preparing thin sections for microscopic observation, microtomes are particularly appropriate in disciplines such as histology, cytochemistry, autoradiography, etc. Microtomes are applicable to the following functional program elements (FPE's):

5.9 Small Vertebrates (Bio D)
5.10 Plant Specimens (Bio E)
5.26 Invertebrates (Bio F)

15.3 LOGISTICS

15.3.1 Packing and Launch

No particular packing or launch requirements exist for microtomes beyond those applicable to precision mechanical instruments. Standard packing procedures should be adequate.

15.3.2 Installation

Unpacking and installation of microtomes is routine. Mounts are needed to keep the instrument stationary; moderate mechanical forces are applied to a microtome during normal operation.

15.3.3 Consumable Supplies

The only consumable supplies needed for microtomes are the razor blades occasionally used to replace the microtome blade. Carbon dioxide would probably not be the recommended coolant for the freezing microtome.

15.3.4 Accessories and Spare Parts

Accessories for microtomes include blades, specimen stages and clamps, embedding blocks, freezing equipment, section-handling equipment (forceps, brushes, probes), and blade-sharpening equipment. A tool kit should also be provided for maintenance of the microtome.

15.3.5 Maintenance and Repair

Microtomes are relatively trouble-free mechanical devices. Cleaning and lubrication should be routine. Occasional adjustments and minor repairs can be done by a mechanical technician aided by a maintenance manual. When properly cared for, a microtome knife can be used for considerable periods between sharpenings. When in need of sharpening, however, a microtome knife requires both extensive equipment and skill for proper sharpening. Microtome blades being relatively small could be stocked on the Space Station in quantities of five or ten, and returned to earth for resharpening. A hone would then be adequate for maintenance in the Space Station.

15.4 OPERATION

15.4.1 Warm-up and Speed-of-Operation

The only warm-up time needed for microtomes is that required for specimen freezing in the freezing microtome.

Speed-of-operation of a microtome varies with the skill of the operator and the nature of the sections being made. Initially, all Space Station operators will be unskilled in zero-gravity microtome use. The actual cutting procedures occupy only a portion of the time needed for operation. After a section (or

series of sections) has been cut, it must be picked up and placed on a microscope slide.

15.4.2 Operation Skills

Operation of a microtome in space will require extensive earth-based experience with microtome use and, in addition, the ability to adapt known techniques to an entirely new environment.

15.4.3 Operating Procedure

The basic steps of the operation of a microtome will be the same as for earth-based use: specimen preparation, cutting, and application to microscope slides. Detailed procedures, however, will differ considerably, and perhaps unexpectedly.

15.4.4 Sample Preparation and Handling

Preparation of frozen samples will involve placing the specimen on the freezing stage and holding it there while it cools. Use of embedded samples, on the other hand, will require development of zero-g embedding techniques.

Handling the cut sections and placing them on the slides will require a completely different set of techniques than currently used. The lack of gravity will require different techniques. A ribbon of paraffin sections, for example, normally hangs down over the knife as it is cut. In free-fall conditions, it may hang in the air waiting for a slide to be placed against it. Also, a collodion section, vigorously cut on a sliding microtome, may be projected into a trajectory across the spacecraft. New procedures must be developed to deal with these and other, as yet unsuspected, possibilities. New tech-

niques will also be needed to keep the specimens on the slides until cover slips are attached; adhesive coatings may be needed for this.

15.5 INTERFACE

15.5.1 Interface with Other Laboratory Instruments

The microtome is a necessary instrument for preparation of tissue sections for microscopy.

15.5.2 Interface with Vehicle Systems

Except for cooling techniques, microtomes are manually operated instruments which do not interfere with vehicle systems. Although compressed CO_2 is commonly used for freezing microtomes, Peltier cooling methods are recommended for Space Station application (available instruments use a few hundred watts). Cryogenic cooling could be used with some instrument modifications and vehicle interface. Laminar air flow across the cutting area may be needed to collect particulate material when cutting some types of specimens.

15.6 SAFETY

Microtomes present no flame hazards, microbiological hazards, electromagnetic interference, or ionizing radiation. Protruding parts of the instrument and the knife blades present some physical hazards to personnel. A rigid cover should be kept over the instrument when it is not in use. With some types of specimens, control of particulate material may be required.

15.7 MODIFICATIONS

No major modifications are needed for Space Station use of microtomes. They should be firmly mounted and covered when not in use. If CO_2 cooling is used (not recommended), venting is necessary to avoid undue stress on the EC/LS system.

15.8 AVAILABLE INSTRUMENTS

The different manufacturers produce microtomes with similar features and applications. Quality may differ between different product lines. The choice of a rotary or sliding microtome, or both, will depend upon the applications and the preferences of the principle investigator. The major microtome manufacturers are:

American Optical Corporation
Leitz Wetzlar
Lipshaw Manufacturing Company
MSE Incorporated
Reichert Optische Werke

The Vibratome of Oxford Laboratories is an interesting alternative (or supplement) to a conventional microtome. However, the requirement for cutting to occur in a water bath might require modification for Space Station application.

Section 16
OPTICAL TEST EQUIPMENT

Optical test instruments to be used in an optical test facility are discussed in this section. The coverage is necessarily incomplete because of the limited scope of the present survey. Detailed consideration of the optics test facility is recommended for further study. In addition to the type of instruments discussed in Paragraph 16.1, the following may be considered as useful in an optical test facility: Microdensitometer, Spectrograph, Spectrophotometers, Laser, Microprojector.

16.1 PRINCIPLES OF OPERATION

16.1.1 Optical Bench

An optical bench is a precision support for both the optical test equipment and the optics being tested. An optical bench restrains optical element movement to a single degree of freedom (a geometrical line). Movement along the bench is generally unrestricted over a linear range of 1 to 2 meters. The test equipment used on an optical bench will often provide other degrees of freedom relative to the single degree of freedom provided by the ways of the optical bench. These auxiliary movements are generally only a few centimeters, and seldom over a quarter meter.

16.1.2 Surface Plate

A surface plate is frequently used as a precision support for optical test equipment and optics being tested. The surface plate restrains optical element movement to two degrees of freedom (a geometrical plane). Movement on a surface plate is generally unrestricted over a surface area of several meters square.

The test equipment used on the bench will often provide other degrees of freedom relative to the flat surface of the surface plate. These auxiliary movements are generally only a few centimeters, and seldom over a quarter meter.

16.1.3 Autocollimator

An autocollimator is an ultraprecision telescope having an infinite front focal distance. It has two separated back focal planes which are formed by an optical beam splitter. A back-illuminated reticle is located on one of these two back focal planes, and the second back focal plane is the front focal plane of an ocular which is used to visually observe the second back focal plane.

In operation, an image of the back-illuminated reticle is projected by the telescope of that autocollimator to infinity. When a flat mirror is positioned in front of the autocollimator within its collimated beam and normal to the autocollimator optical axis, the back-illuminated reticle image is returned to the second back focal plane from which it is viewed through the ocular. A nonilluminated reticle is supported on this second back focal plane. The lateral position of the returned back-illuminator reticle can now be composed visually with that of the nonilluminated reticle. Small, angular displacements of the flat mirror in front of the autocollimator is in the order of a few seconds of arc (in some cases even fractions of a second of arc). This is seen as a shift in the position of the back-illuminated reticle image relative to the nonilluminated reticle.

16.1.4 Alignment Telescope

An alignment telescope has the same features as an autocollimator, with the additional capability of being able to vary its front focal distance from

infinity to some very short focal length. Some commercially available alignment telescopes can actually bring their focal distance to within the telescope itself. The alignment telescope can be used to view visually each of the optical elements and optical images within a compound optical system. This latter function cannot be performed by an autocollimator.

16.1.5 Interferometer

There are many types of interferometers, but they are all used to delineate the vertical surface height differences between two or more optical surfaces by analysis of the interference fringe patterns produced. A one-fringe error, using mercury green light, between two surfaces represents a vertical surface deviation between these two optical surfaces, which is slightly greater than 0.25 micron (10 microinches). High-quality optical surfaces are often made to match each other to an accuracy of 1/10 fringe, or 1/40 micron (1 microinch).

16.1.6 Reflex Microscope

A good-quality microscope is generally required to analyze the images produced by lens systems. This microscope should be a reflex type such as used for metallurgical analysis. The microscope is then capable of projecting a reticle image into an optical system, and analyzing the return image produced when a return mirror is placed at the opposite end of the optical system. Good-quality flat-field metallurgical objective lenses should be used.

16.1.7 Circular Table

A circular table is a precision mechanical support which can accurately hold an optical system and rotate it about one axis or, in some cases, two axes.

Commercial circular tables are available with angular readouts which are accurate to 1 second of arc and better.

16.1.8 Height Gage

A precision height gage can measure lengths to 0.0001-inch accuracy, and can be used on a surface plate to check the mechanical dimensions of an optical system to that accuracy. Commercial height gages are available with measuring ranges well over a meter.

16.1.9 Modulation Transfer Function (MTF) Measuring Equipment

A real lens system has a different Modulation Transfer Function for every combination of angular field, front focal distance, spectral bandpass, and F/number. The modulation transfer function shows essentially the contrast ratio between the images of adjacent black and white bars with a given spacing in line pairs/mm. There are a variety of commercial systems available, and most can be mounted to either an optical bench or a surface plate.

16.2 APPLICATIONS

Optical testing and alignment is generally performed on either an optical bench or a surface plate. Some of the most important optical tests and alignment procedures are as follows:

1. The lens and mirror performance measurements:
    a. Focal length
    b. Surface accuracy and quality

c. Image analysis

1) Direct visual image analysis with a microscope.
2) Photographic recording with visual microscopic analysis.
3) MTF (Modulation Transfer Function) using photometric detection.
4) Interferometer.

d. Spectral transmittance or reflectance

2. An optical system alignment is generally accomplished with one or more of the following devices:

a. Autocollimator

b. Alignment telescope

c. Precision circular table

d. Precision height gage

e. Microscope

3. Photometric or spectrophotometric measurements of the optical system performance is made with one or more of the following devices:

a. Spectrograph (see Section 9)

b. Densitometer

c. Spectrophotometer (see Section 23)

1) Absorption
2) Fluorescent
3) Phosphorescent
4) Polarization
5) Raman
6) Atomic Absorbance (see Section 2)

d. Spectroradiometer (see Section 20)

16.3 LOGISTICS

16.3.1 Packing and Launch

Optical test instruments are either large and heavy (optical benches or surface plates, for example), or small and fragile (autocollimator or reflex microscope, for example). The heavy items must be securely tied down during launch and transported to avoid damage to nearby items. Small fragile instruments need the protection of foam-filled cases.

16.3.2 Installation

Installation of the large pieces of equipment in the optics facility will require some care because of the mass of the items used. They will be easy to move but hard to stop. Secure tie-down will be needed in the spacecraft. Small and fragile instruments should be stored safely when not in use. The foam-filled shipping cases should be appropriate for on-board storage.

16.3.3 Consumable Supplies

Consumable supplies for optical test equipment are lens cleaning fluids and tissues. In some cases these items are not to be used because of delicate lens coatings which could be damaged.

16.3.4 Accessories and Spare Parts

The accessories needed for optical test equipment are many and interrelated. Some instruments are accessories for others. For instruments such as the optical bench, there are extensive lines of accessories and their use is recommended as the topic for a future study.

Spare parts should be taken for all breakable optical components of the optical test facility. A complete supply of light sources will be needed.

16.3.5 Maintenance and Repair

Since these instruments are themselves test and maintenance instruments, their upkeep will be an integral part of their operation. Cleaning of optical surfaces and lubrication of mechanical parts is a continuing aspect of maintenance of optical test equipment.

16.4 OPERATION

16.4.1 Warm-up and Speed-of-Operation

Warm-up time is applicable only to light sources. Full operating output is achieved in less than one minute. Operating time is extremely variable with the type of measurements being made. Generally, more time is used for set-up of the desired configuration of the apparatus than the taking of readings. Operating times vary from a few minutes to a few hours.

16.4.2 Operation Skills

Optical test equipment can be used by either professional or highly trained technical-level personnel. In-flight training of inexperienced personnel is not recommended.

16.4.3 Operating Procedure

The operating procedure for optical test equipment varies with the test being performed. Measurement of lens performance might include a test of spectral transmission of the lens, which would include the following steps:

- Mount light source and spectroreflectometer along optical bench.
- Mount lens to be tested in front of integrating sphere of reflectometer.
- Perform spectral scan.
- Compare observed spectral transmission with standard or previous performance of the lens.

Additional tests on the lens might include determination of resolution or assessment of resolution degradation by use of modulation transfer equipment. Measurement would be made at different F/stops and at different positions in the angular field.

16.4.4 Sample Preparation and Handling

Sample handling with optical test equipment involves the mounting and illumination of the optical components under test. This is a matter of skill, experience, and training; there are no unique problems in a zero-g environment.

16.5 INTERFACE

16.5.1 Interface with Other Laboratory Instruments

Optical test equipment can be used to test, calibrate, and align the optical subsystems of many other laboratory instruments. This would include cameras, microscopes, telescopes, spectrophotometers, monochromators, etc.

16.5.2 Interface with Vehicle Systems

The only interface required with vehicle systems is electricity and cooling for the light sources used with optical test equipment. Access to the on-board data management system will be useful for some calculations, but for most cases on-line processing will not be required.

16.6 SAFETY

16.6.1 Flame Hazards

The only flame hazards presented by optical test equipment are those which might be associated with lasers or mercury arc lamps.

16.6.2 Microbiological Hazards

There are no microbiological hazards associated with optical test equipment.

16.6.3 Electromagnetic Interference

The only electromagnetic interference which might be associated with optical test equipment is that which might arise from the power supplies of the light sources.

16.6.4 Ionizing Radiation

Optical test equipment does not produce ionizing radiation. Some optical components, however, may show degradation with prolonged exposure to radiation.

16.6.5 Physical Hazards to Personnel

The major physical hazards to personnel arising from optical test equipment are those associated with lasers, mercury arc lamps, hot parts on lamp sources, and protruding pieces of equipment.

16.7 MODIFICATIONS

Optical test equipment requires no modification for zero-g operation beyond the replacement of mounting devices which depend on gravity with positive hold-down devices.

16.8 AVAILABLE INSTRUMENTS

Optical Bench

Beckman Instruments, Inc.	J. Noertl Optical Co.
Gaertner	Oriel
Lasico	Ardel Instrument Co.

Surface Plate

Modern Optics	Microflat Co.
Gaertner	
Do All	

Autocollimator

Vought	Moller
Nikon	Davidson Optronics, Inc.
Kollmorgen	Leitz

Alignment Telescope

Kollmorgen	Nikon
Moller	Davidson

Interferometer

Group 128, Inc.	Itek
Kollmorgen	Davidson
Engis	Tropel

Reflex Microscope

Nikon	Wild
Zeiss	Leitz

Circular Table

Leitz	A A Industries

Height Gage

Cadillac Gage Co.	Brown & Sharp

Modulation Transfer Function (MFT) Measuring Equipment

Weiser Robodyne	Itek
Zoomar	Spectra Physics

Section 17
OSMOMETERS

17.1 PRINCIPLES OF OPERATION

The presence of a solute in a solvent changes some of the colligative properties of the solvent. The boiling point increases, the freezing point decreases, and an osmotic pressure is developed when a semipermeable membrane separates the solution from a sample of pure solvent. The amount of these changes is proportional to the total number of particles, disassociated ions, and dissolved molecules in the solution. The number of particles, in moles, dissolved per kilogram of solution is the osmolality of the solution. Three types of osmometers are available commercially: membrane osmometers, vapor-pressure osmometers, and cryoscopic osmometers.

17.1.1 Membrane Osmometers

When a semipermeable membrane separates two compartments, one containing a solvent and the other a solution of solvent plus solute, molecules of solvent will tend to cross the membrane to dilute the solution. If, however, a hydrostatic pressure is applied to the solution, the movement of solvent modules is inhibited and there is no net exchange across the membrane. At equilibrium, the hydrostatic pressure equals the osmotic pressure. The osmotic pressure π is related to concentration C and molecular weight M in the following relationship:

$$\pi = C R T/M \qquad (1)$$

in which T is the absolute temperature and R the gas constant.

Commercially available membrane osmometers use a variety of automated and semiautomated techniques to determine the hydrostatic pressure necessary to equilibrate with the osmotic pressure. In some models, detection of the equilibrium is achieved by allowing the pressure differences to displace a membrane (not the semipermeable membrane) which acts as one plate of a capacitor. Changes in capacitance are then detected by a bridge circuit. Pressure is adjusted mechanically and automatically with servo motors for some instruments. Although some models adjust and detect pressure with the height of a column of liquid, some newer instruments use servo motors and pressure transducers for these functions.

17.1.2 Cryoscopic Osmometers

Cryoscopic osmometry is based on the principle of depression of freezing point in solutions of nonvolatile solutes. The following relationship generally holds:

$$\Delta T_f = \frac{RT^2}{H_f} m \times 10^{-3} \qquad (2)$$

where ΔT_f is the freezing point depression, R is the gas constant, T_f is the freezing point of the pure solvent on an absolute scale, H_f the latent heat of fusion per gram of the solvent, and m is the molal concentration of the solution.

When the concentration is large enough to cause a freezing point depression of more than one or two degrees, the following equation is more precise:

$$\frac{d \ln (1-x)}{dT} = \frac{H^1_{f_2}}{RT_0} \qquad (3)$$

Here $\underline{x}$ is the mole fraction of solute. H^1_f is the molal heat of fusion and would be expressed as a function of the temperature before the equation is

integrated. For dilute solutions, the integral of this equation becomes equivalent to equation (2).

The equations shown relate to ideal solutions. In actual solutions, ionization and association effects cause departures from predicted values. The value of ΔT_f is then a measure of the effective number of particles in the solution and, in osmotic work, of the osmolality.

The instrumentation is, in principle, simple. A freezing bath (air or liquid) is provided. A tube containing a sample with a thermistor probe inserted, is placed into the freezing bath. The thermistor forms one arm of a calibrated Wheatstone bridge. The response of the bridge is calibrated, using saline solution of known osmolality. The output of the bridge can be applied to a sensitive null indicator, to a mirror galvanometer, to an amplifier followed by an "electric eye" tube, or to a conventional d'Arsonval meter.

In a practical instrument, maximum precision is obtained if careful consideration is given to design and technique. One requirement is that only a modest fraction of the total sample volume be frozen. Otherwise, the solution remaining unfrozen becomes excessively concentrated and unrepresentative to the original value. Correspondingly, the equilibrium temperature is depressed excessively. It is desirable, in any event, that both the saline standards (of osmolality approximating the samples) and all samples be frozen to about the same extent if the results are to be reproducible and the calibration valid.

A convenient way to control the extent of freezing is to supercool the solution to a moderate and reproducible extent. Typically, the sample is taken down to

about -3°C, at which time it is violently agitated either by a vibrating wire striking the inner tube wall or by ultrasonic vibration. Another technique is to seed the solution with an AgI crystal. In the freezing process, the excess heat which was earlier removed during supercooling is largely restored by latent heat of fusion from the frozen portion of solvent. However, the true freezing point is not quite attained since (a) the solution in equilibrium with the ice has been somewhat concentrated, thus lowering the equilibrium temperature, and (b) warming caused by the latent heat must compete with the continuing cooling action of the freezing bath. If temperature gradients in the sample, container, and immersed probe are made reproducible, then the slope of the calibration curve ($\Delta T_f / \Delta$ osmolality) using saline solutions is close to the theoretical value. A further necessary precaution is precooling of all samples to a uniform temperature. This is usually done in a well adjoining the freezer, where several samples may be stored prior to measurement.

The molal freezing point depression for aqueous solutions is 1.858°C. Since commercial cryoscopic osmometers claim a precision of ±1 to ±3 milliosmoles, they are measuring freezing point temperatures to a precision of ±0.002 to ±0.006°C. This is only 1/20th to 1/60th the precision attained in conventional osmometers of the vapor-pressure type. In making these comparisons, however, the effective molal ΔT in the vapor-pressure osmometer is only about 1/6 that of the cryoscopic method.

The precision of ±1 milliosmole in the cryoscopic method is usually obtained with a 2 ml sample. If a smaller sample tube is used, reducing sample size to 0.2 ml, there is a two-fold reduction in precision. This is due to the

reduced thermal "buffering" or thermal capacitance of the sample and its tube, decreased control of temperature gradients, and the short duration of the "plateau" during which ice and solute are in equilibrium.

17.1.3 Vapor Pressure Osmometer

If a droplet of solution of a nonvolatile (or relatively nonvolatile) substance in a volatile solvent is suspended in a space saturated with the solvent vapor, the solution droplet equilibrates to a temperature slightly higher than that of the ambient vapor. This elevated temperature tends toward a value at which the increase of vapor pressure just offsets the initial lowering of vapor pressure due to the presence of solute. The elevation is proportional to the molar concentration m and may in practice approach 90 percent or more of a theoretical ΔT (degrees C) given by the familiar ebullioscopic formula

$$\Delta T = \frac{RT^2}{q} \; m \times 10^{-3} \qquad (4)$$

where R is the gas constant, T the experimental temperature (absolute scale), and q is the latent heat of vaporization per gram of solvent at the experimental temperature.

In the practical determination of osmolality by the vapor-pressure method, the solution to be measured is suspended in the vapor space on a thermistor bead. A drop of pure solvent is suspended on a nearby, separate, reference thermistor in the same vapor space. The thermistors are connected in opposite arms of a Wheatsone bridge, the output of which is applied to a low-drift null amplifier. The change of setting of the bridge required for rebalance, after previously

balancing with pure solvent on both thermistors, is a measure of the temperature differential.

A simple mercury cell and voltage-dropping resistor serve as the bridge supply. Current through the thermistors is very low, about 35 microamperes, and self-heating in the thermistors due to this current raises their temperature only about 0.001°C. Excessive heating would cause disturbing convection currents and is undesirable.

The thermal assembly of the conventional instrument is shown schematically in Figure 17-1. The thermal block, usually of aluminum, is nested in insulation. A resistance heater is wound on the block for close thermostatic control of its temperature, this being sensed by a resistance thermometer or thermistor inserted within the body of the block. The signal from this sensor acts via a thermostatic controller to regulate the heater current as required. A typical operating temperature for the thermal block is 37°C. A viewing tube penetrates the block and permits inspection of the thermistors and hanging drops. The cavity in the block is maintained saturated with the solvent vapor by a solvent-containing cup and an upward-extending cylindrical wick. Projecting into the cavity are the probe, carrying the sample and reference thermistors, and two or more syringes. The thermistor beads are about 1 mm in diameter. One of the syringes, loaded with solvent, is positioned with its tip very close to the reference thermistor. The tips of the remaining syringes are close to the sample thermistor. Both thermistors are initially loaded with solvent for zeroing. About 2 to 3 microliters cling to each thermistor. Subsequently, the droplet on the sample thermistor may be replaced by drops of as many as

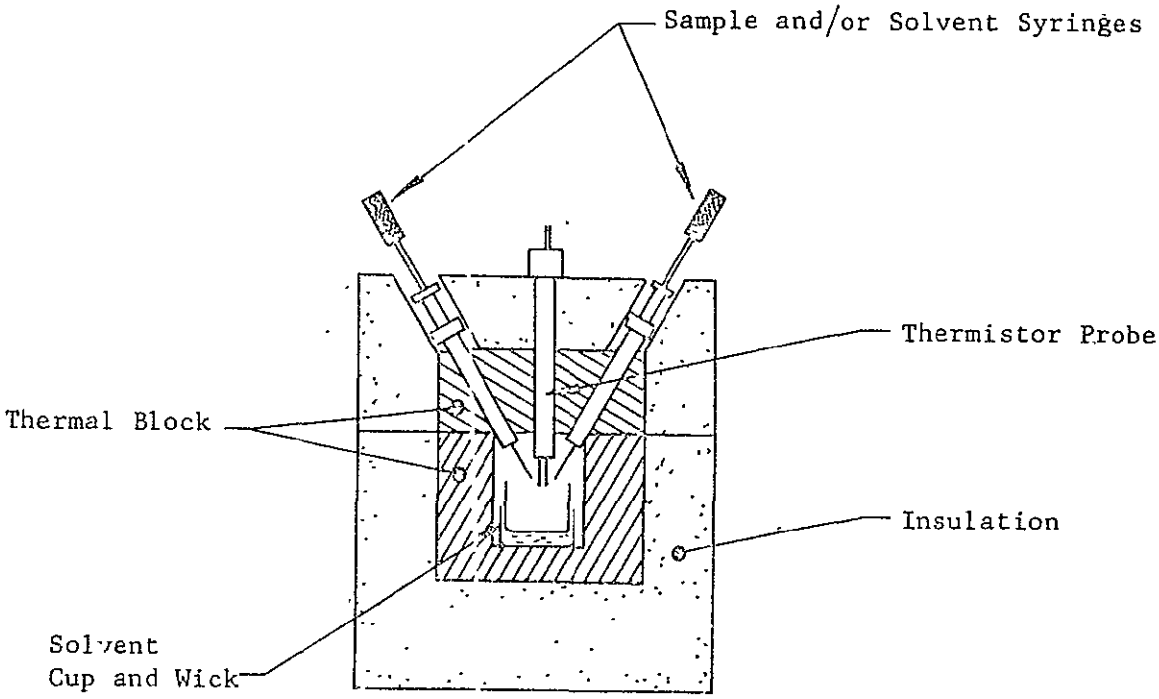


Figure 17-1. Vapor Pressure Osmometer--Conventional Thermal Assembly, Schematic Diagram

four different samples in sequence, each delivered from a separate, preloaded syringe. Typical equilibration time for the sample drop to reach a stable temperature is about two minutes.

The instrument is calibrated by use of a solute of known molecular weight dissolved in a known concentration in the same solvent as that of the unknown. Since most solutes are associated to some extent, generally in increasing de degree as concentration is increased, a series of standards of varying concentrations may be used. The ratio $\Delta R/m$, i.e., ratio of resistance change for rebalancing the bridge to the molal concentration, is plotted against molarity and extrapolated to $m = 0$ to determine the instrument constant K. The molarity of any unknown is then given by ΔR, the resistance change of the unknown, divided by K. If, however, standardization is done with a mannitol solution whose association is negligible at molal concentrations of clinical interest, calibration with a single solution of known concentration will suffice.

17.2 APPLICATIONS

Osmometers are used to determine the osmotic pressure and, thereby, the osmolality of solutions. Osmolality determination of blood, urine, and other body fluids is particularly significant in the physiology of the kidneys as well as water and mineral metabolism generally. Determination of osmolality may also be of interest in plant physiology and microbiology. In addition to measurement of osmotic pressure, osmometers can be used for determination of the molecular weight of a substance. The colligative properties of solutions, on which osmometry depends, are altered by the total particle (dissociated ions and dissolved molecules) content of the solute in a solution. Thus, if the

number of particles of a known mass of solute is determined, the molecular weight of the particles can be determined.

Cryoscopic and vapor pressure osmometers are used for measurement of body fluids and determination of the molecular weight of substances below around 10,000, while membrane osmometers are used for determination of molecular weights from 10,000 to 1,000,000. Osmometers could find application in the following functional program elements (FPE's):

5.9 Small Vertebrates (Bio D)
5.10 Plant Specimens (Bio E)
5.13 Biomedical and Behavioral Research
5.23 Primates (Bio A)
5.25 Microbiology (Bio F)
5.26 Invertebrates (Bio F)
5.27 Physics and Chemistry Laboratory

17.3 LOGISTICS

17.3.1 Packing and Launch

The packing procedures for the electronic and optical components of osmometers should be little different from those for other precision instruments. The mechanical components, however, may require special and separate packing for launch and transport.

17.3.2 Installation

Installation of osmometers would include reassembly of mechanical components. The instrument must also be made secure in the working area. Connection to electric power, and perhaps vacuum, is needed for operation.

17.3.3 Consumable Supplies

Consumable supplies for osmometers would include solvents (if other than aqueous solutions are used) and standard solutions.

17.3.4 Accessories and Spare Parts

The major accessory for osmometers is a strip-chart recorder. The recorder can be used to document the equilibration time of different types of instruments, but it is not needed for Space Station application. Automated sample changers are also available, but not recommended.

Spare parts for osmometers should include most functional assemblies and components of the instrument used. In addition, a considerably supply of membranes for the membrane osmometer, syringes for the vapor-pressure osmometer, and sample types for the cryoscopic osmometer are needed. A tool kit should be included for mechanical installation and repairs.

17.3.5 Maintenance and Repair

Osmometers should be checked for accuracy with standard solutions during the measurement procedures. Repairs, when needed, should be approached by replacement of entire subassemblies.

17.4 OPERATION

17.4.1 Warm-up and Speed-of-Operation

Although electronic warm-up time is negligible for most osmometers, the equilibria which must be established in the different types of instruments can contribute to the time before the instrument can be fully operational.

These equilibria times include cooling time for cryoscopic osmometers, sample-cell saturation for vapor-pressure osmometers, and fluid flow to establish osmotic equilibrium for membrane osmometers. These times may vary from a few minutes to an hour or more.

17.4.2 Operational Skills

Osmometers can be operated by any professional or technical personnel with previous experience with these instruments. Some preflight training may be needed for personnel not familiar with the specific model of osmometers used in the Space Station.

17.4.3 Membrane Osmometer Operation

The basic steps in the use of a membrane osmometer are as follows:

- Moisten, smooth out, and install membrane.
- Introduce solvent to one side of membrane and solution to the other side.
- Adjust pressure on solvent to inhibit fluid movement into solution.
- Read pressure needed to maintain equilibrium and calculate desired values.

Several commercial instruments perform these operations automatically after introduction of the sample solution, presenting the operator with a direct reading. The block diagram of a typical automated instrument is shown in Figure 17-2.

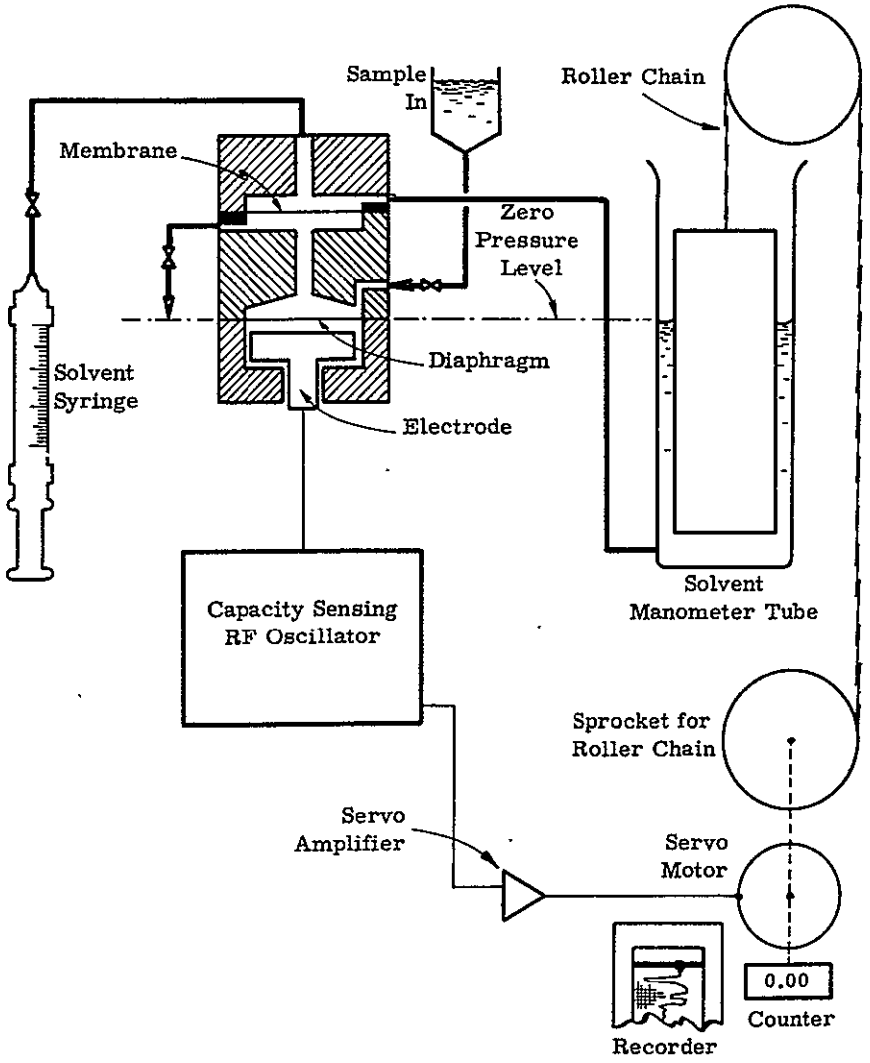


Figure 17-2. Typical Automated Osmometer--Block Diagram

17.4.4 Vapor-Pressure Osmometer Operation

The basic steps in operation of a vapor-pressure osmometer are as follows:

- Dilute samples and fill syringes.
- Place a drop of solvent on each thermistor bead.
- Press amplifier zero button and center null meter with amplifier zero control.
- After two minutes, center null meter with bridge zero control.
- Rinse sample bead with the first sample dilution, leaving a drop in place.
- After a minute, check amplifier zero and adjust if necessary.
- At end of two minutes, center null meter with bridge output control reading ΔV.
- Repeat application of sample and reading of ΔV for other sample dilutions.

17.4.5 Cryoscopic Osmometer Operation

- For calibration, make up several concentrations saline solutions in the range of expected sample osmolality.
- Precool 2 ml volumes of these standards in sample tubes and make freezing point measurements.
- Immerse the tube in the freezing bath and start sample stirrer.
- Allow temperature to fall to a predetermined level in the supercooled region.
- Nucleate freezing by strongly vibrating a wire against the inside tube wall.

NOTE

The temperature now climbs to a plateau, varying only slowly during one to two minutes as it passes through a maximum.

- Switch bridge potentiometer to maximum sensitivity, and adjust bridge to bring the meter to a zero position.
- Record the setting of the bridge balance control.

NOTE

In direct-reading instruments, the knob indicating the osmolality is set to the known value of the saline solution, while a supplementary bridge control brings the null indicator to zero.

- Measure standard solution according to the same procedures.

17.4.6 Sample Preparation and Handling

Osmometry in the Space Station depends heavily upon wet-chemistry techniques and apparatus. Cryoscopic osmometers will present the fewest sample-handling problems in this environment. A closed cell of flush-through design appears to be an appropriate solution.

Vapor-pressure osmometers present the dual problem of maintaining saturation of vapor pressure in the analysis chamber and application of the sample and solvent drops to the thermistors. Saturation of the analysis chamber might be accomplished by use of a damp sponge rather than an open fluid container and reduction of interchamber pressure to allow saturation. Application of drops of fluid to the thermistors may be possible by careful manipulation, drops being held on by capillary action and surface tension. Removal of the

drops from the thermistors could present a problem requiring additional reduction of chamber pressure and some flushing between samples.

For membrane osmometers, introduction of the sample (and solvent) to make proper contact with the membrane could be an extremely difficult problem. Solution of the problem may require extensive instrument modification. The experimental value of a membrane osmometer is doubtful if extensive modifications are required for use in a zero-g environment.

17.5 INTERFACE

17.5.1 Interface with Other Laboratory Instruments

In several applications of osmometers, not only is the osmolality of the sample wanted, but also the chemical composition. Qualitative analysis may require an atomic absorption spectrophotometer, a flame photometer, a spectrophotometer, or specific ion electrodes.

17.5.2 Interface with Vehicle Systems

Osmometers generally require 115 V, 60 Hz power. Vacuum connection may be needed for modification of some of the instruments. Although most instruments provide direct-reading capabilities, an output could be provided for the data management system, if desired.

17.6 SAFETY

17.6.1 Flame Hazards

Osmometers neither present flame hazards nor are they considered to be made of inflammable substances.

17.6.2 Microbiological Hazards

Osmometers present no particular microbiological hazards beyond those associated with microorganisms which may be contained in the sample.

17.6.3 Electromagnetic Interference

Osmometers present no more electromagnetic interference than other electronic or electrooptical instruments.

17.6.4 Ionizing Radiation

Osmometers present no particular radiation hazards beyond those associated with the sample itself.

17.6.5 Physical Hazards to Personnel

The sharp corners and protruding knobs on the operating panel of osmometers may present some hazard to personnel. Most available instruments provide adequate protection from internal sections which may be heated or cooled during analysis.

17.7 MODIFICATIONS

17.7.1 Cryoscopic Osmometer Modification

The major modification needed for a cryoscopic osmometer would be the development of a flush-through sample cell containing a stirrer and a sensing thermistor.

17.7.2 Vapor-Pressure Osmometer Modification

The vapor-pressure osmometer appears adaptable to zero-gravity conditions without great operational difficulty. The pool of solvent normally contained in the bottom of the vapor chamber may be replaced by a wetted sponge. A

moist sponge layer may also line the cavity wall. In the conventional instrument, when sample solution or solvent is used to rinse off the thermistor beads, the rinse liquid normally falls into the solvent pool. In the space-adapted instrument, this liquid may be picked up by blotting action. Thus, a rod with a blotting pad may be inserted into the chamber and brought up close to one side of the thermistor bead. The rinse liquid would be applied with one of the syringes, on an opposite side of the bead.

Retention of the solvent or sample droplet on the thermistor bead under zero gravity, as in the earth's gravitational field, should present no particular difficulty.

17.7.3 Membrane Osmometer Modification

Two problems associated with the lack of gravity face the membrane osmometer. The first is the adjustment of hydrostatic pressure in the solvent to balance the osmotic pressure in the sample solution. Most commercially available instruments rely on the effect of gravity on a column of fluid. However, one available instrument uses a pressure transducer. The second problem, considerably more serious, involves introducing and removing fluids from the chambers on either side of the membrane. Although bubbles can be tolerated in either chamber, good contact must be made over the entire surface of the membrane. Considerable developmental effort may be needed to solve this problem. Until it is solved, the feasibility of this type of osmometer for zero-g application is questionable.

17.8 AVAILABLE INSTRUMENTS

Cryoscopic osmometers are manufactured by the following companies:

Advanced Instruments, Inc.
Fiske Associates

Membrane osmometers are manufactured by the following companies:

Scheicher & Scuell, Inc.
Hewlett-Packard
Hallikainen Instruments
Melabs

Vapor-pressure osmometers are manufactured by the following company:

Hewlett-Packard

Section 18
OXYGEN ANALYZERS

18.1 PRINCIPLES OF OPERATION

Because oxygen fulfills an essential role in life support, oxidation and combustion reactions, substantial effort has been devoted to the analysis of this gas. A number of analysis methods have been used successfully in several different basic types of commercial analyzers. These methods are discussed in the following paragraphs.

18.1.1 Paramagnetic

Oxygen has a large paramagnetic susceptibility compared with other common gases and vapors. This characteristic allows direct measurement by determining the amount of displacement that a test body makes in a magnetic field as a result of the presence of varying quantities of oxygen. Indirect oxygen determination is accomplished by measuring the cooling effect of a heated wire by oxygen in a magnetic field.

18.1.2 Catalytic Combustion

If another combustant such as hydrogen is added to oxygen, the reaction completion or temperature rise produced is a useful parameter by which oxygen concentration is determined.

18.1.3 Thermal Conductivity

Oxygen concentration can be measured by adding hydrogen to a sample and measuring the thermal conductivity before and after a continuous combustion.

18.1.4 Electrochemical

Oxygen concentration can be determined by measuring the current output of an electrochemical cell when operated either in a galvanic, polarographic, or amperometric mode. In common usage, a thin oxygen permeable plastic membrane is used over the oxygen-sensitive electrode to allow the sensor to be completely self-contained.

18.1.5 Summary

Numerous other techniques have been proposed and utilized for the analysis of oxygen. Included in these other methods are mass spectrometers and gas chromatographs which are the subject of separate surveys (Sections 11 and 13). These would only be used for multicomponent gas analysis since they are significantly more complex and, hence, less reliable than the single-purpose instruments.

The methods enumerated above represent the majority of the commercial analyzers available today. Of the above four types, the electrochemical oxygen analyzers are the only units having undergone substantial flight testing and qualification in past years. For example, electrochemical oxygen analyzers have been qualified and flown on the BIOS program, qualified for Apollo spacecraft, and additional development and testing has been done under the MOL, Gemini B, and Airlock programs. These electrochemical analyzers were chosen because they require very small power, are low in weight and size, and are relatively uncomplicated. In addition, they measure the partial pressure of oxygen which is the most important requirement for life support. They can be very stable at room tem-

perature for extended periods of time. To date, they are substantially better than other commercial analyzers for long-term stability. The most difficult requirement is for a precise automatic temperature compensation; the permeable membrane does give the oxygen sensor a substantial temperature coefficient. A simplified diagram of the permeable membrane electrochemical sensors for oxygen is shown in Figure 18-1.

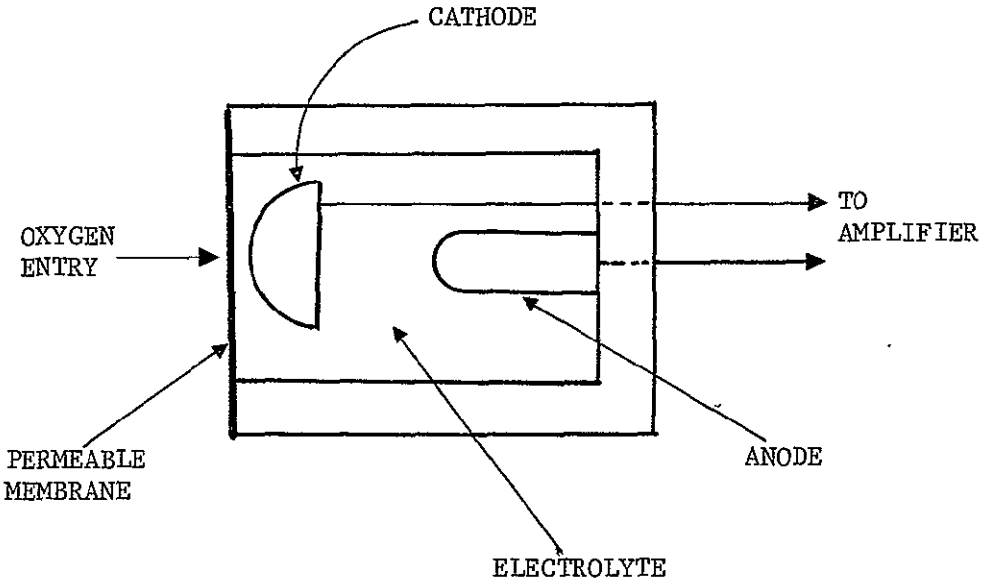


Figure 18-1. Typical Oxygen Sensor

In operation, atmospheric oxygen diffuses through the permeable membrane and reacts electrochemically at the cathode. Within the cell, an electrolyte surrounds the anode and cathode. The reduction of oxygen at the cathode produces a current flow which is directly proportional to the oxygen partial pressure diffusing through the membrane.

Various materials have been successfully used in the construction of electrochemical oxygen sensors. Typical membranes used are cellophane, polyethylene, polypropylene, silicone rubber, and RFE and FEP types of Teflon. The types of cathode materials used have included platinum, silver, and gold. Possible electrolytes are potassium, chloride, potassium hydroxide, sodium chloride, and ammonium chloride. Anode materials include calomel (HgCl/Hg), lead (Ag_2O/Hg), tungsten, cadmium, and AgCl/Ag.

These electrochemical cells may be galvanic or polarographic (more correctly "amperometric"). In the latter case, an EMF (0.5 to 0.8 volts) is applied to the electrodes. The most common galvanic cell is the gold-potassium hydroxide-lead system, while the most popular polarographic system is the gold-potassium chloride-silver system.

The completed sensor is typically very small--1/2 to 2 inches long and approximately 3/8 to 1-1/2-inch in diameter. The output current from the sensors ranges from 0.1 to 100 microamperes for atmospheric oxygen, depending mostly on the active area of the cathode and the membrane thickness. The output of the sensor is either amplified by a current amplifier or has sufficient output to drive a meter directly.

18.2 APPLICATIONS

Oxygen analyzers can be used to determine the oxygen partial pressure in cages, incubators, experimental enclosures, and in the expired air of human and experimental animals. The following functional program elements (FPE's) are directly applicable:

5.9 Small Vertebrates (Bio D)
5.10 Plant Specimens (Bio E)
5.13 Biomedical and Behavioral Research
5.14 Man/System Integration
5.16 Materials Science and Processing
5.23 Primates (Bio A)
5.25 Microbiology (Bio C)
5.26 Invertebrates (Bio F)
5.27 Physics and Chemistry Lab

Oxygen analyzers are of critical importance in the EC/LS system. Air contains approximately 160 mm Hg partial pressure of oxygen. When the partial pressure of oxygen drops to in the region of 70 to 100 mm Hg, an hypoxic condition occurs in which the oxygen supply is too limited to support life. Under these circumstances, blackout occurs followed by complete oxygen starvation and death. If the oxygen partial pressure rises too high, over-oxygenation can occur and induce convulsions and brain damage. Fortunately, this high-level tolerance is very high (1500 mm Hg), and a human not expending a great amount of energy can exist in a 100-percent oxygen atmosphere without risk for brief periods of time.

Mass Spectrometers (Section 13) and Gas Chromatographs (Section 11) can also be used for the analysis of oxygen. These analyzers are very much more complex

than the simple electrochemical oxygen analyzers. They would typically be used only where multicomponent gas analysis is required at one time. If an analysis of oxygen only is required, electrochemical sensors are substantially more simple, reliable, lighter, and less costly in terms of power consumption and investment.

18.3 LOGISTICS

18.3.1 Packing and Launch

Both the sensor and the electronics of electrochemical oxygen analyzers are highly resistant to shock and vibration. They can be launched without special packing precautions, and need only be fastened securely to sustain the full stress of launch. The only component sensitive to shock and vibration would be the readout meter.

18.3.2 Installation

Installation of the electrochemical oxygen analyzer is very simple. Attachment to electric power would be the only requirement. With portable instrumentation, power is self-contained. Since this instrument is very compact, the only necessary requirement for installation would be to locate the sensor/amplifier in an appropriate place to obtain a continuous oxygen readout.

18.3.3 Consumable Supplies

The only consumable supplies required are gases for calibration of the oxygen sensor. These gases would typically be a single-calibration gas for oxygen span and a zero gas such as nitrogen or helium for checking the sensor residual

current. Since the sensor is very linear to oxygen partial pressure, these two gases would suffice for performing on-board calibration.

18.3.4 Accessories and Spare Parts

Spare sensors are the only components required for support of the oxygen analyzer. Sensor life is limited typically by the amount of consumable electrolyte within the sensor. Although the sensors normally have a long life, in excess of one year on air, replacement sensors may be necessary. Many sensors are available with recharge kits for renewing the electrolyte. This feature is neither recommended nor desirable for spacecraft operation.

18.3.5 Repair and Maintenance

Because of the small size and simplicity of the electrochemical oxygen analyzer, repair and maintenance should be absolutely minimal. Spare sensors should satisfy the major maintenance requirement--even this on a relatively long maintenance cycle. Oxygen analyzers are generally small enough to permit including a spare unit on board for backup.

18.4 OPERATION

18.4.1 Warm-up and Speed-of-Operation

Although the oxygen analyzer will give readings during warm-up, approximately 20 minutes are needed before accurate readings are obtained. Once in operation, the unit can be left operating continuously. Even with small batteries, the operating life can be in excess of 1,000 to 2,000 hours.

The speed of response for 90 percent step change is typically 5 to 20 seconds at room temperature. This is primarily determined by the thickness and the type of membrane material used. This speed is very adequate for cabin air monitoring requirements. It may be marginal if used for closed-suit loop applications where speed of response might be an important factor in maintaining environmental safety.

18.4.2 Operation Skills

Space-station operation of the electrochemical oxygen analyzer is possible for unskilled personnel. Calibration of the equipment with standard gases can be accomplished by anyone with minimal technical capability. With the exception of the calibration procedure, preflight training required for this device would be negligible.

18.4.3 Operating Procedure

Typical operating procedures for oxygen analyzers would be as follows:

Preparation:	Allow a 20-minute warm-up period.
Calibration:	Introduce span gas. Adjust span control to read correct value using a simple computation involving accurate cabin pressure readings. Introduce zero gas to check sensor residual current. Recheck span gas to repeat prior span setting. Remove calibration gas supply.
Measure:	After removal of calibration gas, the oxygen analyzer will automatically give continuous oxygen partial pressure readout from the ambient gas.

18.4.4 Sample Preparation and Handling

For oxygen partial pressures in the range of 150 to 760 mm full scale, absolutely no sample preparation is required. The sensor monitors the oxygen partial pressure by simply being exposed to the gas. The oxygen usage by the electrochemical sensor is extremely small and does not deplete the life-support environment.

18.5 INTERFACE

18.5.1 Interface with Other Laboratory Instruments

The oxygen analyzer is a completely separate and independent instrument and does not require any special interface with other instruments. If the sample is in a liquid medium for dissolved oxygen measurement, it is necessary to incorporate a flow chamber and the appropriate seals to completely enclose the sensor tip. This would normally not be used for dissolved oxygen measurements except in blood gas analysis as covered in Section 3. The output of the oxygen analyzer can be transmitted to either a standard laboratory potentiometric recorder, or to the on-board data system. The output can typically be 0 to 5 volts or less for convenient recording or telemetry.

18.5.2 Interface with Vehicle System

The only interface requirement for the oxygen analyzer is a very small amount of power, readout as required, and calibration gas as previously discussed. If a zero gas such as nitrogen, propane, helium, or argon is present in the vehicle system, it could be utilized with a simple valve and small tube for zeroing the oxygen analyzer system. An oxygen calibration gas will be required also, and here again an available on-board source of air or other upscale calibration gas

could be used, if available. In either case, small, separate calibration cylinders may be used and supplied for this purpose. Manual calibration can easily be accomplished using very small hand-held cylinders.

18.6 SAFETY

18.6.1 Flame Hazards

Electrochemical oxygen analyzers present no flame hazard since only low voltage, low current, dc supplies are required. The sensors are usually fabricated from plastic materials which may not be an approved nonmetallic material. The flammability of these sensors, if ignited, may be a consideration in their choice.

18.6.2 Microbiological Hazards

Possible microbial growth in the electrolyte solution used in the sensor can be controlled with bactericidal agents. The analyzer does not in itself present any microbiological hazards to personnel.

18.6.3 Electromagnetic Interference

An electrochemical oxygen transducer represents no source of interference generation unless a dc-to-dc converter is needed to isolate an analog output signal and associated circuits from the power input line. LC and Feedthrough bypass filtering will bring converter-generated interference well under control. Power-line filtering will provide sufficient freedom from susceptibility to conducted RF and transient energy on the power input lines. Battery operation would be ideal. The actual electrochemical cell should be shielded by a metallic enclosure to prevent undesirable pickup of radiated energy.

18.6.4 Ionizing Radiation

Ionizing radiation is neither produced by nor interferes with the operation of these analyzers.

18.6.5 Physical Hazards to Personnel

The oxygen analyzers are very safe with the exception that if potassium hydroxide electrolyte is used in the sensor, care must be taken in the event of a membrane or sensor housing breakage. Neutral electrolytes such as sodium chloride and potassium chloride do not present safety problems. The oxygen analyzers are small and rugged and would typically pose no mechanical or electrical hazards to operating personnel.

18.7 MODIFICATION

1. The commercial electronic components should be replaced by space or flight-qualified counterparts.

2. The range of the oxygen analyzer may require modification for other ranges of interest. This range-change would only normally be required where a 0 to 760 mm Hg (100 percent) oxygen range may require conversion to a 0 to 250 mm Hg oxygen partial pressure range. This modification is simple for most electrochemical oxygen analyzers.

18.8 AVAILABLE INSTRUMENTS

A list of commercially available electrochemical oxygen analyzers and their manufacturers is listed in Tables 18-1 and 18-2.

	Company	Model	Price	Sensor	Temp Comp	Alarms	Output	Power	Size
1.	Beckman	OM-10	$650	Polarographic Sealed/ Rechargeable	Yes	Audio & Visual	Alarm & IV FS	1 W (not inc Alarms)	9x5x9 in.
2.	Beckman	100800	495	Polarographic	Yes	No	Meter	-	11-1/2 x 5-1/2 x 9
3.	IMI, Div. of Beckton-Dickinson	-	495	Polarographic	No	Audio & Visual	-	-	-
4.	Teledyne	320C	-	Galvanic	Yes	Audio & Visual	Alarm & IV FS	-	5x8x11 in.

Table 18-1. Line-Operated Electrochemical Oxygen Analyzers

	Company	Model	Price ($)	Sensor	Temp Comp	Alarms	Output	Battery Life	Size (inches)
1.	Beckman	100801	395	Polarographic	No Has Temp Probe	No	Meter & Recorder Output	240 hrs	11-1/2x5-1/2x9
2.	Bio Marine Industries	OM300	600	Galvanic	Yes	-	Meter	-	-
		OMC400	790	-	-	-	-	-	-
3.	Instrumentation Laboratory	IL402	-	Long Life--One Year	Yes	Yes IL404 Dual Alarm	Meter & 100 mV FS	4500 hrs	6 x 4 x 3
4.	Johnson-Williams	K2500	-	Galvanic	No	No	Meter	50 hrs	6 x 4-1/2 x 3
5.	Magna Corp	1070	240	Short Life--Galvanic	No	No	Meter, Needs Special Adapter for Recorder	None Required	3 x 2-1/2 x 12
6.	Precision Scientific	68850	395	Galvanic	No	No	Meter	None Required	14 x 13 x 5
7.	Teledyne	320B	-	Sealed/ Replaceable Galvanic	Yes	No	Meter & 100 mV FS	3-4 mos.	5 x 8 x 11
		320B/RC	-	Sealed/ Replaceable Galvanic	Yes	No	Meter & 100 mV FS	-	5 x 8 x 11
		330A	250	Sealed/ Replaceable Galvanic	Yes	Yes	Meter, no Recorder Output	-	5 x 2 x 1

Table 18-2. Portable (Battery) Operated Electrochemical Oxygen Analyzers

Section 19
RADIATION COUNTERS

19.1 PRINCIPLES OF OPERATION

Three general types of radiation counting instruments were considered: liquid scintillation counters, planchet counters, and gamma counters. These three types are used as analytical instruments. Dosimeters and radiation flux detectors were not considered in this section because they are not analytical instruments. Each of the three analytical instruments detects nuclear disintegrations indirectly by detecting "counts". The percentage of counts detected for a given number of disintegrations is an index of the efficiency of each instrument. The concentration of isotopes is inferred from the observed rate of disintegrations and knowledge of the isotope.

Since the accuracy of the counting aspect of a nuclear experiment is dependent solely on the number of counts observed, the performance of counting instruments is evaluated on the basis of the time required to count to a given accuracy. A number representing this then evaluates both the time required by the instrument to count a sample to a given accuracy, and the accuracy that can be achieved in a given time. This number is the figure of merit:

$$F = \frac{S^2}{S + 2B}$$

S = sample count rate observed and is the disintegrations per minute at the sample times the counting efficiency, DPM x E

This is often seen as $F = \frac{E^2}{B}$

B = background counts per minute

19.1.1 Liquid Scintillation Counters

Liquid scintillation counters use fluorescent materials in a liquid state to detect nuclear disintegrations. The β particles stimulate photon emission from the fluorescent materials (cocktails). Photomultiplier tubes detect the light flashes, and electronic circuits accumulate the number of flashes (counts) over a period of time.

19.1.2 Planchet Counters

Planchet counters measure radioactivity more directly than scintillation counters. A metallic sample container, the planchet, is placed under the detector cell. The detector is a Geiger-Muller-type cell which detects the ionization of gas molecules around high-voltage electrodes. A small current pulse is produced for each count detected. The same electronic circuitry can be used for detecting the counts from liquid scintillation counters and planchet counters.

19.1.3 Gamma Counters

Gamma counters use an operating principle similar to the liquid scintillation systems. The sample in a test tube is placed into a depression in a scintillating crystal (NaI), and light flashes are detected by a photomultiplier tube. The output pulses are again accumulated over a given period of time.

19.1.4 Signal Conditioning

For any given count, all pulses contributing to the count are treated as equivalent. However, different energy-level particles can be distinguished. In liquid scintillation counting, the brightness of the flash is related to the particle's energy. It is possible to select upper and lower thresholds for flashes included in the count and thus select the isotope for which the count is made. In multiple channel instruments, two or more energy ranges can be counted simultaneously. In planchet counters, sensitivity is maximized for particular energy particles by adjustment of the electrode voltage of the detector cell.

A different type of electronic signal conditioning is the use of coincidence and anti-coincidence techniques to improve accuracy of counts. Photomultiplier tubes, when used for detection of extremely low light flashes, have an internal noise level comparable to the signals they produce. Impulses related to actual light flashes are distinguished from tube noise by coincidence circuitry; a count is recorded only if impulses are produced simultaneously from two different photomultiplier tubes. The gas detector cells of planchet counters solve a similar problem with the opposite logic. Counts from the sample must be distinguished from cosmic rays. Two detectors are used, one in contact with the sample and the other away from it, but in the same cosmic ray tract. In this situation, an anti-coincidence circuit is used to reject pulses which occur simultaneously from both cells, counting only those from the sample cell.

19.2 APPLICATIONS

Liquid scintillation counters are used principally for counting beta particles as emitted by isotopes such as Tritum, C^{14}, P^{32}, and I^{131}. These are used

extensively as tracers in biomedical and biochemical research. In some cases, such as calibration from an internal standard, a liquid scintillation counter can be used as a Cerenkov counter for gamma radiation. Since the actual sensor of the liquid scintillation counter is actually a photomultiplier tube, the instrument can be used to detect or measure light-producing processes. An example of this would be the luciferin/luciferase reaction (from firefly enzymes) which occurs in the presence of ATP; this is the principle of the bacteria counter discussed in Section 4.

Liquid scintillation counting also is usable for higher energy isotopes. The exclusive photomultiplier yields higher E^2/B values at room temperature than can be obtained with temperatures of 5°C or lower. This advanced transducer also produces the highest tritium efficiency ever obtained in a commercial scintillation system, thus substantially reducing counting time, or improving accuracy.

Typical applications of planchet counting are: the counting of Phosphorous-32 labeled nucleic acids on filter paper, measuring airborne radioactivity from microporous filters, determining Strontium-90 in milk, and checking the environment for latent radioactivity from fallout and reactor leakage. Planchet counting also is exmployed in many phases of biological and environmental research.

Gamma radiation detectors have more limited use requiring isotopes such as I^{125}, Co^{60}, Cs^{137}, and Ba^{133} to produce gamma radiation.

Radiation counters may find application in the following functional program elements (FPE's):

5.9 Small Vertebrates (Bio D)
5.10 Plant Specimens (Bio E)
5.13 Biomedical and Behavioral Research
5.17 Contamination Measurements
5.23 Primates (Bio A)
5.26 Invertebrates (Bio F)
5.27 Physics and Chemistry Lab

19.3 LOGISTICS

19.3.1 Packing and Launch

Radiation counters cannot be shipped in their operating configuration. Detector cells are fragile and must be separately packed for maximum protection against acceleration and vibration. Current trends in the development of solid-state radiation detectors suggest that such devices may become available in commercial instruments in the near future. Solid-state radiation detectors will make a major contribution in improving the sensitivity and ruggedness of radiation counters. The shielding (lead) has considerable mass and must be kept stationary to protect other items near it. The radiation standards should be shipped in shielded compartments to prevent radiation leakage. Other components of radiation counters can be shipped by standard procedures.

19.3.2 Installation

After unpacking, the detectors and shielding must be reinstalled in the instrument. A zero-g environment will be particularly advantageous for moving the shielding. Gas and electrical connections must also be made. Modifications

for moving the calibration standard may require addition of vacuum and compressed air connections.

19.3.3 Consumable Supplies

Considerable consumable supplies are needed for radiation counters, the actual amounts, however, varying with the volume of work done. Supplies for liquid scintillation counters include: toluene, fluors, solubilizing agents, standard solutions, vials, etc. The supplies for planchet counters include: planchets, planchet holders, standards, and gas (methane with Argon). The rate of gas use during operation is a few tenths ft^3/hour. Supplies for gamma counters include test tubes and standards.

19.3.4 Accessories and Spare Parts

Extra detectors are advisable for all types of instruments. For the detector of planchet counters, a supply of gold window membranes is needed. Extra shielding may be needed for the Space Station environment. A gas regulator is needed. For one line of instruments, the planchet counter and gamma counter are accessories to the basic liquid scintillation counter. A modified version of a discrete sampling flow cell for the liquid scintillation counter would solve some sample-handling problems.

19.3.5 Maintenance and Repair

Maintenance and repair should be handled on a replacement basis. Spares should be available for the detectors and for the amplifier, logic, and power-supply modules. Mechanical components should be modified or eliminated.

19.4 OPERATION

19.4.1 Warm-Up and Speed-of-Operation

Warm-up time is negligible for most components of radiation counters. The exception is the counter cell of the planchet counter. This cell must be uniformly gas-filled for correct operation. Approximately one hour is needed to purge the cell if it is allowed to become empty. When the planchet counter is in frequent use, a slow flow is maintained through the tube at all times the instrument is not in operation.

19.4.2 Operation Skills

Radiation counters can be operated by any technical or professional level personnel with previous experience on these instruments. Preflight training is needed for inexperienced personnel.

19.4.3 Operating Procedures

Operation procedures differ considerably for the three types of instruments. Major operation steps for liquid scintillation counters are as follows:

- Calibration with quenched series (of known activity) for each sample type.
- Determination of external standard number.
- Make plot of external standard number to determine efficiency.
- Count background of sample with similar consistence except lacking radioisotope.
- Count sample for given time interval.
- Calculate rate of disintegrations and concentration of tagged isotope.

Major operation steps for planchet counters are as follows:

- Adjust gas flow in detector tube.
- Set detector electrode voltage for plateau of particle to be counted.
- Count background for empty planchet.
- Count standard.
- Count sample to preset time.
- Calculate rate of disintegrations and concentration of tagged isotope.

Major operation steps for gamma counters are as follows:

- Set operating voltage for photomultiplier tube (trade-off of efficiency vs. background)
- Count background activity for empty test tube.
- Count standard.
- Count sample.
- Count sample to preset time.
- Calculate rate of disintegrations and concentration of tagged isotope.

19.4.4 Sample Preparation and Handling

Radiation counters present a variety of sample-handling problems. For both liquid scintillation counters and gamma counters, a liquid sample must be introduced into the sample tube. One problem here is venting the air from the tube while filling it. Use of a collapsed plastic bag would aid filling, but the strong solvents used in the scintillation cocktails dissolve most appropriate plastics. Complete removal of air bubbles is probably not essential for the fluid samples for either scintillation or gamma counters.

The sample vial in liquid scintillation counters must be lowered into the counting chamber on elevators. With earth-based instruments, the vial is held on the elevator platform by gravity. In a zero-g environment, hold-down clamps must be provided. Magnets are not permitted in or near the counting chamber.

Although automated sample-changing devices have not been recommended for most instruments in this survey, the flow cell available for some liquid scintillation counters would solve several of the sample-handling problems for these instruments. This device mixes the sample and cocktail ingredients, pumps them into a flow cell in the counting chamber, times the counting period, flushes the flow cell for the next sample, and recycles. This device, in present form, is not usable in a zero-g environment without modifications.

Sample handling in planchet counters is also gravity-dependent in earth-based instruments. The cell window of the planchet counter is on ultra thin gold foil membrane. Any contact planchet or sample with this membrane could cause ripping or contamination. Gravity holds the sample in the planchet and the planchet in the planchet holder. A vacuum system can perhaps be used to keep the planchet in the planchet holder. Use of a plastic membrane to keep the sample in the planchet holder would allow alpha particle counting but exclude beta particle counting. Samples stuck to the bottom of the planchet by evaporation of a solvent would be usable. For other solid and particulate samples, some adhesive method might be developed for a free-fall environment.

Some models of counters use as an internal standard a small radioactive pellet. This pellet is moved in the instrument with air pressure and drops back into its

shielding by gravity. This procedure would need modification to use negative pressure to return the pellet to its shielded case.

19.5 INTERFACE

19.5.1 Interface with Other Laboratory Instruments

Radiation counters can be used in conjunction with other laboratory instruments. Two examples are paper chromatography and spectrophotometry. The presence of tagged compounds in particular regions of a chromatograph strip can be detected by placing strips of the paper in a planchet for direct counting, or by dissolving it in a "cocktail" or liquid scintillation counting. The liquid scintillation counter is also a powerful companion instrument for a spectrophotometer.

19.5.2 Interface with Vehicle Systems

Radiation counters require power and provision for heat dissipation. Planchet counters require a gas supply for their detector cells. For instruments which normally move an internal standard (a small radioactive pellet) into position with an air pump, compressed air is needed to replace the pump and a vacuum source to return the standard to its shield (it normally falls back by gravity). The final count is available as a digital or analog signal for interface with the data management system. Some modifications may be desirable to allow the data management system to control counting periods and perform other calculations. Storage and disposal of radio chemicals is a problem which must be handled by the vehicle system. Disposal of consumable supplies presents similar problems.

19.6 SAFETY

19.6.1 Flame Hazards

There are no open flames used and these instruments present no inflammable hazards.

19.6.2 Microbiological Hazards

Radiation counting instruments do not introduce any microbiological hazards beyond those inherent in the samples being measured.

19.6.3 Electromagnetic Interference

The high-frequency transformer in the power supply of some instruments may require some shielding to avoid EMI. It should also be noted that the detectors of radiation detectors are extremely sensitive to magnetic fields and cannot tolerate magnets or magnetic fields in their vicinity.

19.6.4 Ionizing Radiation

The standard solutions for liquid scintillation and planchet counters do not emit radiation of sufficient energy to penetrate the plastic containers they are stored in. However, care must be taken to keep these standard solutions in their containers. The standard solutions for gamma counters emit gamma rays and must be shielded for storage. The internal standard for one model of liquid scintillation counter is a Cesium-137 pellet (40 μ curies) which is normally stored in a lead shield within the instrument, remotely moved to a position under the sample vial. A different instrument, however, requires that the internal standard be completely removed from its shielded container; the

radioactive standard enters the laboratory, unshielded, before being manually repositioned in the instrument. This procedure is not recommended.

19.6.5 Physical Hazards to Personnel

Radiation counters, like most other instruments considered, require some protection of laboratory personnel from sharp corners and protruding knobs. In addition, the high mass contributed by the shielding presents possible hazards to personnel in the case of rapid accelerations of the vehicle. The instrument and its shielding must be mounted securely to a benchtop.

19.7 MODIFICATIONS

Although the operating principle of radiation counters is relatively simple and adaptable to space-flight use, many of the engineering solutions commonly employed in earth-based instruments are inappropriate for use in a zero-g environment. The following modifications may be noted particularly:

- The elevator used to move the sample vials into the counting chamber of liquid scintillation counters must be modified to give positive movement of the vial into and out of the chamber; gravity cannot be used. Clamps are recommended, since magnets cannot be used.
- A hold-down device must be developed for holding planchets in the planchet holder; a mechanical or vacuum hold-down would be appropriate here.
- A hold-down device must be provided to hold the sample tube in the detector cell in the gamma counter.

- Additional shielding may be needed to reduce background radiation for all types of counters used for analytical purposes.

- The lead shielding blocks must be held securely in place in and around the instruments; the mass of these blocks makes an unsecured block a particular hazard in case of unexpected accelerations.

- A blank detector and anti-coincidence circuitry must be provided for planchet and scintillation counters lacking this feature.

- Positive movement of the internal standard to and from the counting chamber must be provided; in some instruments, a compressed air and vacuum may be appropriate for this.

- Venting of the gas from the Geiger-Muller-type detectors must be provided to avoid accumulation of this gas in the laboratory.

- Shielding of the power supply may be needed to reduce EMI.

- A blower may be needed to remove the heat generated by the electronic components; normal convection cooling would be highly inefficient in a zero-g environment.

19.8 AVAILABLE INSTRUMENTS

Most commercially available radiation counters are sold only in highly automated configurations. Since automated sample-handling is not needed for expected Space Station applications and not feasible in a zero-g environment, these instruments have not been considered. Manual desk top instruments are manufactured by Beckman Instruments, Inc., and Intertechnique (French).

A portable radiation counting instrument has recently been introduced by General Electric (the NUCLE-EYE) which is of interest since it incorporates a silicon avalanche diode detector.

Section 20
RADIOMETERS

20.1 PRINCIPLES OF OPERATION

20.1.1 General

Radiometry (measurement of radiation) deals with the measurement of broad-spectrum radiation, covering wavelengths from ultraviolet through the infrared and including the visible region. Instruments to measure this radiation can be conveniently classified into two broad categories.

Instruments that measure radiation across wide areas of the spectrum are called pyranometers. These simple radiometers measure total sun and sky radiation. The category of pyranometers includes pyrheliographs or pyrheliometers which measure the direct solar energy component only. These devices normally use thermopiles or silicon solar cells as detectors.

Instruments that measure narrow or specific wavelengths in the spectrum are called filter radiometers or spectroradiometers. These measurements utilize narrow bandpass filters or optical monochromators to isolate and view a narrow portion of the spectrum from the ultraviolet to the infrared regions. A common variation is an optical drive mechanism which allows the instrument to become a scanning spectroradiometer.

When the specific category of only visible radiation is considered, the radiometer is called a photometer. The most significant difference in the two

instruments lies in the terminology used to define the radiation values. The terminology used to define light values in photometry are varied and can sometimes become confusing, whereas in radiometry measurements the terminology is simplified with the watt as the basic measurement unit. The common terminology for photometric systems is further defined in Tables 20-1, 20-2, and 20-3, along with other conversion factors for various terms. A summary of radiometric and photometric system terminology is shown in Table 20-4.

20.1.2 Standards

One of the very difficult areas of modern-day radiometry deals with calibration sources. Fortunately, the sun provides an excellent blackbody source for calibration of the radiometer if the radiometer requiring calibration operates in the infrared portion of the spectrum. The major problem in using the sun is in conveniently bringing the beam through the optics. When establishing a reference source for ultraviolet and visible radiometry, the reference source is a more difficult problem. In this instance, an NBS (National Bureau of Standards) reference source quartz-iodine lamp is required (Table 20-5). These lamps are specially calibrated and typically need recalibration after each 100 hours of operation. If the calibration sources are substantially brighter than the sources to be studied, neutral density filters should be available to approximate the illumination intensity of the radiation source being studied.

Name	Definition		Typical Unit
Luminous Flux (ϕ, F)	(Power radiated) x (Visibility)	$\sum P(\lambda) \cdot K\lambda\ (\lambda)$	Lumen
Luminous Intensity(I)	(Flux emitted)/(Unit solid angle)	ϕ/Ω	Candela
Illumination(E)	(Flux incident)/(Unit area)	ϕ/A	Foot-candle
Luminance(L)	(π) (Intensity)/(Unit area)	$\phi/\Omega A$	Stilb (Foot-Lambert)
Transmission Factor(τ)	(Flux transmitted)/(Flux incident)	$\phi_o/\ 1$	Dimensionless
Opacity	(Flux incident)/(Flux transmitted)	τ-1	Dimensionless
Density	Logarithm of opacity	$-\log_{10}\tau$	Dimensionless

NOTE: A = the area, and ϕ = the solid angle

Table 20-1. Definitions of Photometric Quantities

	Foot-Candles	Luxes	Phots	Milli-Phots
1 Ft. candle =	1	10.76	1.076×10^{-3}	1.076
1 Lux =	0.0929	1	10^{-4}	0.1
1 Phot =	929	10^4	1	1000
1 Milliphot =	0.929	10	10^{-3}	1

NOTE: Consider a light detector some distance from a light source. The amount of radiation it receives clearly depends on the concentration of the radiation in the direction from source to detector. This concentration, flux per unit solid angle, is called intensity.

The unit of intensity is the candela (formerly candle) and it corresponds to one lumen per steradian.

The luminous flux density (flux per unit area) incident on a surface is called illumination. The radiometric equivalent is irradiance.

It is usually measured in lux, phot or foot-candle. These correspond, respectively, to one lumen incident per square meter, square centimeter, and square foot.

Table 20-2. Illumination Conversion Factors

	Nits	Stilbs	Candles $in.^2$	Candles ft^2	Apostilbs	Lamberts	Milli-Lamberts	Foot Lamberts
1 Nit =	1	10^{-4}	0.6452×10^{-3}	0.0929	3.142	0.3142×10^{-3}	0.3142	0.2919
1 Stilb. =	10^4	1	6.452	929	31.420	3.142	3142	2919
1 Candle/$in.^2$ =	1550	0.155	1	144	4869	0.4869	485.9	452.4
1 Apostilb =	0.3183	31.83×10^{-6}	0.2054×10^{-3}	0.02957	1	10^{-4}	0.1	0.0929
1 Lambert =	3183	0.3183	2.054	295.7	10^{-4}	1	1000	929
1 Milli-Lambert =	3.183	0.3183×10^{-3}	2.054×10^{-3}	0.2957	10	10^{-3}	1	0.929
1 Foot-Lambert =	3.426	0.3426×10^{-3}	2.210×10^{-3}	0.3183	10.76	1.076×10^{-3}	1.076	1

NOTE: The intensity per unit area is a measure of the apparent brightness of a source and is called luminance. The radiometric equivalent is radiance. Its unit, the stilb, corresponds to one candela per square centimeter. The area referred to is the projection of the emitting surface on a plane normal to the direction of viewing. The total radiated flux density is π times the luminance, if the radiating surface is plane and perfectly diffuse. It is, therefore, convenient to have a unit π times as large as the stilb; this is called the Lambert and it is defined as the luminance of a perfectly diffuse plane surface radiating one lumen per square centimeter.

Table 20-3. Luminance Conversion Factors

Term	Definition	Radiometric	Photometric
Flux	Rate at which energy is emitted from source.	P - Watts	F - Lumens
Flux Density	Total flux divided by the surface area over which it is distributed. Normally considered as a distance r (cm) from the center of the source.	H - Irradiance H = P/Ap in watts/cm^2	E - Illuminance E = F/Ap in Lumen/cm^2
Intensity	Total flux divided by the total solid angle through which it is distributed.	J - Radiant Intensity J = P/ω in watt/ steradian	I - Luminous Intensity I = F/ω in Lumen/steradian 1 Lumen/steradian = 1 Candela
Emittance	Flux density at the source surface.	W - Radiant emittance W = P/A_s in watt/cm^2	L - Luminous emittance L = F/A_s in Lumen/cm^2
Angular Emittance	Intensity per unit area of the source.	N - Radiance N = $P/\omega A_s$ in watt/ cm^2 steradian	B - Luminance (Brightness) B = $F/\omega A_s$ in Lumen/ cm^2 steradian

NOTE: A_s = Area of Source

ω = Solid Angle

A_p = Projected Surface Area

Table 20-4. Radiometer and Photometer Terminology

Type	Power	Current & Voltage	Description
Quartz-iodine	1000 watt	8.3 A 115 V 60 cycle	G.E. Type DXW
Quartz-iodine	200 watt	No longer recognized as NBS standard lamp	
Tungsten	100 watt	0.75 A 115 V 60 cycle	100T 8-1/2, 120 V
Tungsten	125 Watt	35 A 3.5 V 60 cycle	G.E. 30A/T24/17
Tungsten	500 watt	3.60 A 115 V 60 cycle	500T 20, 115 - 120 V DNK
Tungsten	1000 watt	7.70 A 115 V 60 cycle	1000T 20, 115 - 120 V DPT

NOTE: The lower powered lamp may be the only practicable standard for space-flight purposes.

Table 20-5. Available NBS Certified Standard Lamps

20.1.3 Instrument Performance Characteristics

In discussing the performance of a radiometer, measurement and calibration are so interrelated that they are considered together. Since performance is a measure of an instrument's usefulness to avoid ambiguous quantitative comparisons, calibration must be able to isolate all parameters which significantly affect the performance of that instrument. When the measurements can be referred to standard values such as those from the U.S. National Bureau of Standards, then the calibration process is an absolute calibration.

The typical radiometric measurements of interest in remote sensing include the determination of radiant intensity and radiant emittance of some source which is not accessible for direct measurement. To adequately determine these unknowns, three instrument performance characteristics--response, sensitivity, and radiation reference--must be well defined. These three performance characteristics are defined as follows:

1. R = response--the output per unit input of incident radiation, either V/E or V/L.

2. D = sensitivity--the reciprocal of the noise-equivalent incident (input) radiation.

3. Radiation reference = the level of incident radiation corresponding to a zero reading on the output scale of the instrument.

Also, other more directly obtained characteristics such as the wavelength calibration of a scanning spectrometer and resolution are needed for interpretation of the output.

Of the three defined characteristics, the response is the most useful since it is always required in transforming an output reading into the corresponding value of incident radiation input. Sensitivity is of much lesser importance if the measurements are to be made far above the instrument noise level. Similarly, a rough measure of the reference radiation is always sufficient if a truly small amount of radiation is represented by an output reading of zero. Figure 20-1 is a block diagram of a comparison source radiometer which illustrates the typical components required or used for a radiometer. Several components are very basic to the radiometer:

1. Optics. These collect the radiation through an aperture of Area A and focus it on a field stop of a certain area.

2. Detector. This transduces the radiation which comes from the field stop to a signal, usually electrical of magnitude B.

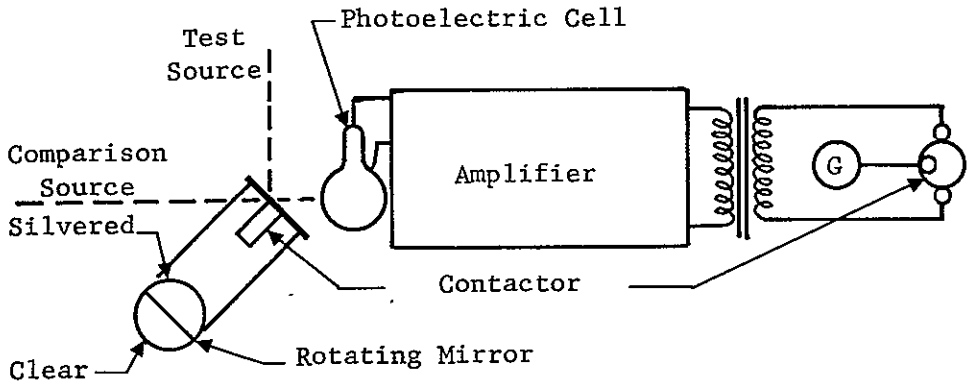


Figure 20-1. Comparison Source Radiometer--Block Diagram

3. Electronics and/or Recorder. These amplify or process the electrical signal and record it.

For practical radiometric instruments, the following are useful or desirable features for a multipurpose flexible instrument:

1. Direct readout in absolute energy;
2. A wide, dynamic range of input radiation intensity;
3. A wide overall wavelength capability;
4. Restricted use of visible light calibration source (to reduce power consumption).

5. Narrow wavelength resolution (to obtain specific data for restricted radiation wavelengths).

6. Scanning capability (to minimize manual measurements).

In practice, the simplest radiometers are the pyranometers that do not require optical systems for wavelength resolution. A spectroradiometer, which is a much more versatile and quantitative instrument for detailed source studies, is considerably more complex than the pyranometer--optically and electronically. Use of these two types in the Space Station would depend largely upon the experiments to be performed.

Radiometers have been routinely used in space for a wide variety and number of experiments. As a result, a substantial choice of radiometers developed for space applications already exists. Examples of this are: scanning radiometers for the NIMBUS Satellites and radiometers for the ITOS Satellite, MARINER MARS, JUPITER PIONEER, project scanner, and the Earth Resources Technological Satellite (ERTS). In view of this development work, selection of a specific radiometer should include both the commercial instrumentation available and those radiometers already developed for space applications.

20.2 APPLICATIONS

Radiometers have been used for a number of aerospace applications to date. Among the applications that radiometers have been routinely used for are reflected solar radiation from clouds, ultraviolet backscattering from the earth and its clouds, ocean and sea radiometry, spectral distribution of sunlight in space, temperature profiling, stellar research, planetary exploration,

planetary observation, and military defense for such items as missile surveillance, anti-missile defense, missile guidance, and other classified programs. This type of equipment has also been used for space navigation.

Radiometers are directly applicable to the following functional program elements (FPE's):

5.3A Solar Astronomy Module
5.11 Earth Surveys

20.3 LOGISTICS

20.3.1 Packing and Launch

The simple pyranometers used for wide-band radiation sensing are quite compact and resistant to shock and vibration. They can be launched with very minimal packing precautions. The true spectroradiometer is much more complex, containing shock-sensitive devices such as photomultiplier tubes and optical monochromators, filters, and readout meters. This instrument would usually be shipped in several medium-sized packages.

20.3.2 Installation

The radiometers of either type require electrical power only. No gases or other utilities are required. Electrical power requirements can be substantial, especially for operation of the NBS quartz-iodine Tungsten lamp. Assemblies can be readily handled, set up, or attached where necessary. The limiting factor in installation is in positioning the equipment such that the light energy of the subject to be studied, whether it be the earth, stars, etc., must pass through the entrance optics of the instrument.

20.3.3 Consumable Supplies

The calibration standard lamp is the only consumable supply. The standard lamp retains calibration to within ±1 percent for about 100 hours. It will operate much longer than this period of time with loss in calibration accuracy. No other consumable supplies are required.

20.3.4 Accessories and Spare Parts

A large number of accessories and spare parts are available for spectroradiometers. Among them are standard lamp power supplies, fiber optic source pickup assemblies, telephoto lenses, high-sensitivity detector heads, narrow beam adapters, photometric microscopes, and X-Y mechanisms. With this wide variety of available accessories, a reasonable survey of the intended application should be made to insure that only the necessary accessories are considered for flight. Certainly, an important accessory would be adaptable entrance optics to cover a wide variety of light sources. Spare parts that would be required would typically be such items as extra photomultiplier tube or light detector and batteries, if required. Spare calibration lamps might be considered, but could be handled by resupply as required.

20.3.5 Maintenance and Repair

Pyranometers are extremely simple and should require no maintenance at all, simply replacement if a failure occurs. The more complex radiometers, on the other hand, may require electronic, optical, or mechanical maintenance. This maintenance would have to be provided by a reasonably skilled person since such things as electronics and optics are closely interrelated functions, and reasonable troubleshooting skill would be required.

20.4 OPERATION

20.4.1 Warm-up and Speed-of-Operation

Pyranometers have literally no warm-up requirements. On the other hand, radiometers with their more complex electronic systems and calibration lamps do require approximately 15 minutes to one-half hour for warm-up. It is far preferable if the reference lamp is used for initial calibration and then turned off, and the instrument subsequently used in a single beam mode for unknown source analysis. Under these circumstances, power demands are substantially reduced since the quartz-iodine standard lamp requires up to 1000 watts. In the infrared region where the sun can be used as a source, this power consumption is not a problem. The speed-of-response of the radiometers is quite fast, typically being less than 2 or 3 seconds on the weakest sources. Electronically, response is normally in the millisecond region so that readings can be obtained very rapidly.

20.4.2 Operation Skills

Operating radiometers in space requires skilled technical personnel. Calibration and operation of the instrument require a person with only medium technical skills; however, general understanding of the measurement problems and troubleshooting would normally require those with superior technical skills in the opto-electronic area. Preflight training with the instrument would be mandatory for satisfactory Space Station usage.

20.4.3 Operation Procedure

For a pyranometer, simply using the data management or recording device connected to the instrument is all that is required. Data is immediately available on exposure to the source of interest.

A typical operating procedure for a spectroradiometer would be as follows:

Preparation:	Turn on and allow a 30-minute warm-up. During this period, align the optical system to view the source of interest.
Calibration:	Operate the calibration source whether it be the sun for an infrared standard or an NBS quartz-iodine lamp for the UV visible region so that the instrument outputs can be checked for direct energy readout.
Measurement:	After removal of the calibration source light or using the shutter if applicable, the radiometer will automatically give a continuous readout in the wavelength of interest. If a scanning radiometer is used, the drive mechanism must be actuated or in the instances where band-pass filters are required, they must be rotated or alternated as required.

20.4.4 Sample Preparation and Handling

Since only electromagnetic radiation being received by the instrument is analyzed, there is no requirement for sample preparation and handling other than the entrance optics of the instrument. Care must be taken that if outside sources are being studied that the windows or optics do not attenuate, distort, or otherwise compromise the radiation being received.

20.5 INTERFACE

20.5.1 Interface with Other Laboratory Instruments

The radiometer is a completely separate and independent instrument and does not require any special interface with other instruments. The only exception to this might be where a telescope or entrance optics are being used from some other experiment or on-board equipment. The output of the radiometer can routinely be transmitted to a potentiometric recorder or its millivolt output placed into the on-board data management system.

20.5.2 Interface with Vehicle System

Only two items are necessary for consideration with respect to interfacing with the vehicle. One of these is the power convection. Power requirements are substantial only when the UV visible NBS calibration light source is operation. Typically, the power requirements for a radiometer are on the order of 200 to 400 watts, not including the source. The second interface requirements are the entrance optics for providing the incident source of radiation to the monochromator of the radiometer.

20.6 SAFETY

20.6.1 Flame Hazards

Pyranometers present no flame hazards since only very low voltages and currents are generated. The radiometers do have high voltage supplies when photomultiplier tubes are used, and do carry large amounts of current "at low voltage" to the calibration lamp. As a result, these electrical connections must be carefully made to insure that no spark or arc source is present. The materials involved are typically metallic with very few gaskets or nonmetallic materials. For the electronic control sections, plastic control knobs may be removed and metal ones used instead. O-rings may be used for light-sealing some of the optical elements. These O-rings can be replaced with acceptable nonmetallic compounds.

20.6.2 Microbiological Hazard

Neither pyranometers or radiometers present any microbiological hazards to personnel.

20.6.3 Electromagnetic Interference

The power supplies operating from line voltages within the Space Station are the primary area where electromagnetic interference must be considered. In those instruments with battery packs, this problem is much simplified and interference radiation will be minimal. Radiated susceptibility should be very small when sufficient shielding is provided for the detector and associated electronics. This circuitry can be sensitive to radiated RF energy. Power-line filtering will reduce susceptibility to conducted RF and transient energy to acceptable levels in nonbattery-operated radiometers.

20.6.4 Ionizing Radiation

Ionizing radiation is not produced by radiometers, and radiometers are not affected by wavelengths short enough to cause ionizing radiation.

20.6.5 Physical Hazards to Personnel

The radiometers are very safe with the exception of the high-voltage supply associated with the photomultiplier detector tubes. Optical components are normally small and well encased so that the glass does not present any breakage problems with the exception of the glass dome housings on some models of pyrometers.

20.7 MODIFICATIONS

No special modifications are required for the radiometers with the exception that replacement of commercial transistors and semiconductors with flight-qualified counterparts may be desirable. Nonmetallic materials should be reviewed and replaced as mentioned in Paragraph 20.6.1

20.8 AVAILABLE INSTRUMENTS

A list of commercially available radiometers and pyranometers are listed in Tables 20-6 and 20-7.

Company	Model	Price	Wt	Output	Impedance	λ Range	Remarks
Eppley Lab.	8-48	$ 540	NL	7.5 mV per cal cm^{-2} min^{-1}	300 ohms	280 to 2800 nm	Measures total sun and sky radiation.
Eppley Lab.	8-48A	560	NL	2.5 mV per cal cm^{-2} min^{-1}	300 ohms	280 to 2800 nm	Low sensitivity version of 8-48.
Eppley Lab.	PS	990	NL	5.0 mV per cal cm^{-2} min^{-1}	300 ohms	285 to 2800 nm	Optical filter domes are available for limiting the spectral range.
Eppley Lab.	TUVR	1150	NL	0.2 mV per cal cm^{-2} min^{-1}	1000 ohms	295 to 385 nm	For solar UV measurement.
Yellow Springs Instrument Co.	68	300	3#	2 Langleys/min	Less than 5K	350 to 2000 nm	Silicon solar cell, portable.
Belfort Instrument Company	5-3850A	285	10#	3.0 Gram cals cm^{-2} min^{-1}	NL	Not given	Includes recorder. No power required.
Centralab Semiconductor	CSC-11	< 100	0.1#	0.1 mV cm^{-2}	NL	300 to 1150 nm	Calibrated silicon solar cell.
Kohl Scientific Instrument Co.	28AM150	365	1#	1.5 mV per cal cm^{-2} min^{-1}	5 ohms	300 to 3000 nm	32 junction thermopile.
Science Associates, Inc.	621	260	1#	3.5 mV cal cm^{-2} min^{-1}	NL	300 to 60,000 nm	22 junction manganin-constantan thermopile
Yellow Springs Instrument Co.	65	525	8#	0.25 to 250 mW cm^{-2}	NA	280 to 2600 nm	15 watts power--uses thermistor bolometer

Table 20-6. Pyranometer Instruments

Company	Model	Price $	Wavelength Range in Nanometers	Type	Size	Weight	Power (Not Including Calibration Lamp)	Ranges	Compensated for Direct Energy Readout	Comments
Beckman Instruments	W139652	40,000	250-2500	Grating or Prism	Large	500#			Yes	Weight, size & power too high for aerospace purposes.
Cary Inst.			250-2500	Prism	Large					"
Instrumentation Specialties Co. (ISCO)	SR	2,250	380-1050	Filter Wedge	11" x 9" x 7"	12#	Not specified - Line or Battery (50 Hrs)	Eight Ranges 0.3 to 1000 $\mu w\ cm^{-2}\ nm^{-1}$	Yes	
Cintra	101	3,200	200-1100	Filter					Yes	Numerous accessories available.
Gamma Scientific	3000R		380-700	Grating	Receptor - 19 x 7 x 12	9#	Not specified	Seven Ranges		Recorder accessory available as well as numerous other calibration and accessory devices.
					Control - 11 x 10 x 10	11#	115 V ac	$\cong$0.1 to 100 $\mu w\ cm^{-2}\ nm^{-1}$	Yes	
Block Engineering	E-98		700-35,000		7 x 10 x 8	14#	115 V ac, 20 W	10^5 Ranging		E-9B wavelength response determined by detector. Block has built & flown numerous other radiometers such as E6, E8, E8A, E12 and E21.
Instrument Development Labs (Kollmorgen)	TRIRAD		400-700	Filter	7 x 10 x 23	32#	115 V ac, 34 W	Three Ranges 0-10 to 0-100 lumen ft^{-2}	Reads out CIE chromaticity coordinates	Primary use is for color specifications.
International Light	IL 600 Series		200-700	Prism				Three Ranges	Yes	Rechargeable battery operation.
EG&G, Inc.	580/585	4,300	200-1200 (3 mono-chromators)	Grating	Medium	25# Est.	115 V ac or rechargeable batteries	Six decade + 3000.1 aperture attenuation	No	Large variety of accessories available. Wide dynamic range.
Barnes Engineering	PRT-5	7,500	8,000-14,000 (IR)		Electronics - 16" x 6" x 16" Optical Head - 5-1/2 D x /"	29#	Rechargeable batteries	4 to 17 $\mu w\ cm^{-2}$		Accessories Available.
	PRT-6		1,800-14,000 (IR)	Filters	Electronics - 7" x 19" x 12" Optical Head - 6" D x 7"	34#	115 V ac, 50 W			

*Data does not include calibration sources and power supplies.

Table 20-7. Radiometer Instruments

Section 21
RECORDERS

21.1 PRINCIPLES OF OPERATION

21.1.1 Chart Recorders

In the following discussion of chart recorders, only the lighter-weight (less that 35 pounds) units which could be labeled portable will be considered. Instruments which provide X-Y operation in addition to strip chart (Y vs. time) are preferred but not necessary.

Several types of marking systems and drive systems are in common use in various combinations. Marking systems include ink systems, pressure stylus systems, heat-sensitive systems, electro-sensitive systems, and light-beam systems. The pressure stylus system, if spring loaded, represents the system with the least drawbacks for the Space Station application.

- Ink systems include capillary flow, pressure flow, ball-point pen and fiber-tip pen systems. All types except the fiber-tip pen are capable of fine resolution recording. The capillary and pressure flow ink systems can lead to excess or spilled ink. For a zero-g environment, the felt-tip pen represents the only sure ink system. Chart paper is usually less expensive with ink systems as special surface preparations are not usually necessary.

- Pressure stylus systems make use of a stylus, using gravity or spring loading, to allow a metallic tool to make a continuous mark

on a special paper. Several less expensive recorders use a striker to periodically strike a sensitized paper to make a line of small dots. The continuous systems are capable of fine resolution lines. Paper costs are higher as a result of the special surface preparation. The most acceptable pressure stylus system for use in a zero-g environment is the spring-loaded, continuous type.

- Electro-sensitive systems utilize a special paper which will darken when a small electric current is passed through it from a small pen stylus to a conductive backing. The system will work in a zero-g environment if the pen is spring-loaded. However, a serious safety hazard exists as the pen is usually set to at least 100 volts above the paper backing. EMI generation may be a problem.

- The heated stylus system uses a special stylus pen heated by a low voltage of typically less than 5 volts. The heated stylus, when in contact with a special heat-sensitive paper, darkens the paper. The pen is spring-loaded and typically contacts the paper only at a square or acute-angle corner. The system is capable of high resolution and full operation in a zero-g environment. Safety hazards exist in operator electroshock or burns. A special flame-retardant paper would be required to reduce the hazard from fire in case of a pen heating-system failure. Another undesirable feature of the heated stylus system is the emission of an odor when the paper is sensitized in normal operation. This could possibly be corrected with the use of a special paper.

- Light-beam systems operate with a special photosensitive paper which darkens after exposure to the tiny beam of light. The beam is reflected from high-intensity light source by a mirror attached to a galvanometer. This type of system will not be considered, as a high-pressure xenon or mercury-xenon arc lamp used for the light source represents a very serious safety hazard.

Several types of pen-drive-systems are in common use. The types include mechanical output galvanometers, light-beam galvanometers, and potentiometric or null balance servo systems. Most galvanometer types, other than light beam, and most potentiometric types are applicable to the Space Station use. Some would require modification.

- Mechanical output galvanometers are typically an electrically-driven moving coil on a pivot immersed in a magnetic field. This is similar to the common D'Arsonval meter movement. The mechanical output of the coil is coupled to the marking device. Many strip-chart recorders using this type of pen drive result in a curvilinear readout where the pen traces a segment of a circle where the radius is the distance from the pivot to the pen. The trace can be made rectilinear or straight-line by special mechanical coupling or the use of a specific marking system. The heated stylus system, where the pen contacts the paper only along a square or acute-angle edge, results in rectilinear trace. A type of galvanometer which is driven in a linear mode in place of a rotational mode allows rectilinear recording with any type of mechanical marking system. The response of this type of pen drive

can be moderately fast and the upper -3 dB points (full scale deflection) can approach 50 Hz. At lesser deflections, responses of over 100 Hz can be obtained in some chart recorders. Increased speed-of-response and maximum sensitivity usually require heavier permanent magnets or additional amplification and power consumption. Mechanical output galvanometer types are seldom, if ever, used for X-Y recording. In the typical strip-chart usage (Y vs. time), a clock motor and a gear train or multiple clock motor paper drives are used. Many of the motors are synchronous 60 Hz types which would require replacement with 400 Hz or dc types, or special 28-V dc to 60 Hz converter circuits can also be used. The recorder types with dc drive motors or with integral dc-to-ac converters are preferred.

- Optical output galvanometer chart recorders (oscillograph recorders) employ a mechanically deflected light beam. The beam is focused on a light-sensitive chart paper which develops in 10 to 30 seconds. The deflection is accomplished in a miniature galvanometer with a very small mirror connected directly to the moving coil. A strong magnetic field is required so oscillographic instruments tend to be quite heavy. The frequency response (-3 dB point points) can easily exceed 2K Hz for nominal deflections and 10K Hz with special galvanometers and narrow deflection. The light source is typically a xenon or mercury-xenon high-pressure arc lamp. This represents an extremely great safety hazard in case of explosion. For this reason, oscillographic recorders with arc lamps will not be considered.

- The potentiometric type of chart recorder makes use of a servo system to position the pen. They are null-balancing types, as the servo motor is driven until the feedback voltage from the position feedback device (usually a multi-turn potentiometer or resistance slide wire), equals the recorder input voltage (appropriately scaled). This type of drive is used in a large number of strip-chart recorders (Y-Time) and most X-Y recorders. High sensitivity and high accuracy are the benefits of this system. However, speed-of-response is considerably less than the mechanical output galvanometer types and rarely exceeds 10 Hz (-3 dB and full-scale deflection). Many of the servo systems are ac operated, and synchronized to the 60 Hz power-line frequency. These would present problems in conversion to 400 Hz or 28-V dc operation. Types which use dc servo systems would more easily operate from other than 60 Hz power. A few commercially available recorders operate from dc power or batteries as an option, and would naturally be preferred. Potentiometric-type recorders typically use ink, pressure, or electro-sensitive paper-marking systems.

21.1.2 Magnetic Tape Recorders

Magnetic tape recorders provide a means of recording electrical phenomena for later analysis or usage. Repeated playback is possible. The electrical information is recorded on a thin plastic tape in the form of changes in the magnetic orientation of a very thin coating of a magnetic oxide material. Magnetic "heads" with extremely thin armature gaps are used in both recording and playback of information.

In many recorders, several different tracks of information can be recorded simultaneously by one multiple armature head. Seven-track heads are standard for some tape recorders. The heads are driven electrically by amplifiers or special signal conditioners for recording. The low-level outputs from the heads amplified are by high-gain amplifiers or special signal conditioners.

Analog tape recorders are capable of recording the range of information from low-frequency analog information such as electrocardiograms to high-frequency information such as video signals (6 MHz bandwidth). Higher frequency recording and playback requires a faster tape speed across the head. The amount of information which can be packed into each linear inch of tape determines the upper frequency limit at any one speed.

Applications requiring low-frequency, dc recording, or accurate amplitude information require the use of a technique where frequency-modulated subcarriers are used. Voltage-to-frequency converters (voltage-controlled oscillators) are used in the recording process. Information is retrieved upon playback by frequency-to-voltage converters (discriminators). Many different subcarriers can be frequency-multiplexed on one tape track. The total number of channels can be greatly increased with seven-track multiplexed FM techniques are applied.

Analog tape recorders designed for precision instrumentation purposes tend to be heavy (40 to 60 lbs), even when considered portable. For lesser applications such as annotation on tape and other audio-only applications, the portable battery-operated cartridge tape recorder is ideal. These recorders are housed in packages of less than 0.1 ft^3 in volume and 5 pounds in weight.

The small, self-contained tape cartridges (also called cassettes) have nearly replaced reel-to-reel tape handling in a majority of lower performance (low frequency) tape systems. This would be a definite advantage in a Space Station application as tape-handling is greatly minimized.

Digital tape recorders can be continuous or incremental. In the continuous process, binary (true-false) data is stored as magnetized spots or bits on the magnetic oxide. The tape is run nonstop across the head and the data stream is continuous. In an incremental recorder, the tape is started and halted as it passes over the head. During each movement, a character (usually 7 bits) is written or read. Data is written on the tape in blocks of characters called records. A number of records is then called a file.

Digital tape recorders, continuous and incremental, in the past have tended to be in the same weight-range as the "portable" higher-performance analog recorders. However, an entirely new field of digital recorders has recently appeared for use with small-size computers (minicomputers). The cassette tape cartridge is the standard of this new line. The weight and volume of the resulting recorders are more compatible with Space Station applications.

21.2 APPLICATIONS

Recorders are used to record the output of other laboratory instruments. An advanced data management system, such as that to be found on Space Station, could perform most of the recording functions. Recorders (especially chart recorders) are, however, familiar to most laboratory scientists, who may not be willing to do without them.

The application of other instruments in this survey has been considered with respect to functional program elements from the Blue Book. It is more appropriate to consider recorder application with respect to an instrument providing the input, as follows:

- Audiometer
- Atomic Absorption Spectrophotometer
- Blood Gas Analyzers
- Electrophysiological Equipment
- Gas Chromatograph
- Mass Spectrometer
- Oxygen Analyzers
- Radiometers
- Specific Ion Electrodes
- Spectrophotometers
- X-Ray Spectrometer

21.3 LOGISTICS

21.3.1 Packing and Launch

The packing procedures for recorders should present no problem. The pen assembly on chart recorders represents the most sensitive part in a recorder. Special "keepers" could be installed on pens, or they could be removed, to insure against damage during launch. In general, normal packing for commercial rail-type shipment should suffice for any recorder.

21.3.2 Installation

Unpacking and preparation for use is routine unless strip-chart recorder pens are removed during shipment. The pens would require installation and alignment prior to use. Some provision is needed to mount recorders for use.

21.3.3 Consumable Supplies

Consumable supplies for chart recorders include rolls of chart paper and ink, if an ink-type marking system is selected. Supplies for tape recorders include blank rolls of magnetic recording tape and provision for safe tape storage.

21.3.4 Accessories and Spare Parts

Accessories for a chart recorder include:

- The necessary interconnecting cables
- Paper take-up reel or chart rewinder
- Dust cover to protect the recorder when not in use
- Tools necessary for replacement and alignment of the pen assembly and calibration of the recorder.

Spare parts for a chart recorder include:

- Fuses
- Spare pen assemblies

Accessories for the tape recorder include:

- The necessary interconnecting cables
- Tape head cleaning supplies
- Dust cover to protect the recorder when not in use
- Tools and measuring devices to measure and adjust tape tension
- Tape leader material and reflective tape to signal tape start locations.

Spare parts for a tape recorder include:

- Fuses
- Spare pilot bulbs

21.3.5 Maintenance and Repair

Maintenance and repair of chart recorders will be limited to replacement and alignment of the pen assembly, replacement of fuses, readjustment of the span calibration of the recorder, and cleaning as required to remove accumulated dust from the chart paper.

Maintenance and repair of tape recorders will be limited to replacement of fuses, readjustment of tape tension, and cleaning as required to remove accumulated oxide.

21.4 OPERATION

21.4.1 Warm-up and Speed-of-Operation

No warm-up is required. The recorders are immediately ready for operation. In some cases the magnetic tape recorders require a tape rewind before usage.

21.4.2 Operation Skills

Space Station operation of recorders is possible for technical or professional personnel. A minor amount of training is necessary for proper replacement of chart paper in the chart recorders and installation of tape in the magnetic tape recorder.

21.4.3 Operating Procedures

A typical procedure for a chart recorder is as follows:

- Mount recorder and connect appropriate cables to test point or outputs of instrument to be monitored.
- Turn on recorder.
- Select proper scale factor for Y input.
- Adjust pen position.
- Adjust X input scale factor and pen position (X-Y operation) or time base and paper position under pen (Y-T operation).
- Enable pen. Start time base (Y-T only).
- Disable or lift pen when recording is complete. Halt time base (Y-T only).
- Turn off recorder.

A typical recording procedure for a magnetic tape recorder is as follows:

- Mount recorder and connect appropriate cables
- Turn on tape recorder
- Mount blank tape
- Select appropriate tape speed
- Adjust recording level without tape motion
- Start tape in record mode
- Stop tape when recording is completed
- Remove tape and document location where information was recorder
- Turn off recorder

A typical playback procedure for a magnetic tape recorder is as follows:

- Mount recorder and connect appropriate cables
- Turn on tape recorder
- Mount tape
- Advance tape to desired location
- Start tape in reproduce mode
- Adjust output level
- Stop tape when playback is completed
- Rewind tape to desired location for replay or to beginning of reel
- Restart tape (for replay) or remove tape from recorder
- Turn off recorder

21.4.4 Sample Preparation and Handling

Not applicable.

21.5 INTERFACE

21.5.1 Interface with Other Laboratory Instruments

The electrical interface between the recorder and the instrument to be monitored is, of course, required. The necessary cables will be provided along with the recorder.

21.5.2 Interface with the Vehicle System

Power from the vehicle will be required for operation of the recorder. Dependent upon the type of recorder and modifications necessary for operation on other than 60 Hz power, either 400 Hz or 28-V dc will be used. Vehicle power will be required for charging of batteries in portable magnetic tape recorders.

21.6 SAFETY

21.6.1 Flame Hazards

All chart papers or magnetic tape represent a flammability hazard unless treated to be nonflammable. The heat-sensitive paper used with the heated stylus marking system is especially hazardous. The stylus heat-control circuits could conceivably fail and ignite the paper.

21.6.2 Microbiological Hazards

Not applicable.

21.6.3 Electromagnetic Interference

The following are possible sources of interference in recorders:

- Dc-to-dc converters for conversion of power supplies to 28-volt dc input.
- Dc-to-ac inverter for conversion of 60 Hz capstan drive motors to 28-volt dc input.
- Ac servo drives.
- Dc servo motor with brushes.

All of the above sources can be easily controlled with proper filtering and shielding. A combination of LC filtering for lower frequencies and feedthrough bypass filtering for high frequencies is especially effective.

Electrosensitive marking systems can easily be a source of radiated interference if stylus currents are not maintained at low levels. Shielding of the pen would prove difficult. Therefore, this type of marking system should not be selected without a careful study of EMI.

Recorders should not be especially susceptible to radiated fields or RF or transient energy on the power input lines. The same filtering used to control interference provides sufficient rejection of conducted signals on the power lines. However, the magnetic tape itself can be susceptible to strong magnetic fields. Recorded information can be destroyed. The tape recorder should, therefore, be used away from strong magnetic fields. It may be advisable to store tape reels or cassettes in magnetically shielded storage devices.

21.6.4 Ionizing Radiation

No ionizing radiation is generated within the recorders considered for Space Station application.

21.6.5 Physical Hazards to Personnel

Electro-sensitive marking systems and heated stylus systems present electro-shock or burn hazards to operators. These types of systems should be carefully studied before use in this application. Oscillographic recorders should not be considered due to the hazard of possible high-pressure arc-lamp breakage. Sharp corners (if any) and protruding knobs on any type of recorder present some other physical hazards.

21.7 MODIFICATIONS

The following modifications may be necessary to operate chart recorders in the Space Station environment:

- Conversion of power supplies and chart drives to operate from 400 Hz or 28-V dc.
- Conversion of servo systems to 400 Hz or 28-V dc operation.

- Conversion of pen system to zero-g environment.
- Addition of chart paper take-up mechanism.
- Provision for mounting.
- Replacement of nonapproved plastic parts with metallic parts.

The major modification problem of Magnetic Tape Recorders is conversion of 60 Hz power operation to 400 Hz or 28-V dc power operation. The major problem is the capstan drive motor which directly determines the tape speed. Tape reel-drive motors can also be a problem. The degree of complication is dependent upon the precision of the recording and the capstan drive method used. Frequency multiplexing of precision analog data requires the most accurate tape speed and would naturally require the most critical modification. Digital recording usually requires less accurate speed regulation.

Recording of verbal annotated data in the audio spectrum would be accomplished by a portable battery-operated cassette recorder and would require no power modifications.

Other modifications would include:

- Power supplies (for circuitry)
- Provision for mounting
- Replacement of nonapproved plastic parts with metallic parts

21.8 AVAILABLE INSTRUMENTS

Chart recorders which are more directed toward possible portable operation and which would not be excluded for safety reasons are manufactured by the following:

Houston Instruments

Texas Instruments

Gulton Industries (including Techni-rite Electronics, Rustrak, and West Instruments)

Mechanics for Electronics

Hewlett-Packard Company (including Sanborn and Moseley)

Gould, Inc. (including Brush Instruments)

Beckman Instruments, Inc.

LH Electronics, Inc.

Bristol

Dohrmann Instruments Company

Esterline Angus

Tensitron

Simpson Electric Company

Honeywell

Leeds and Northrup Corp.

Varian

Manufacturers of high-performance magnetic tape recorders (not necessarily portable) include:

	Analog	Digital
Ampex Corp.	X	X
Bell and Howell	X	X
Digi-Data Corp.		X
Hewlett-Packard Co.	X	X
Honeywell	X	X
Kennedy Co.		X
3M Company	X	X
Mohawk Data Sciences Corp.		X

	Analog	Digital
Potter Instrument Co.		X
Precision Instrument Co.	X	X
Sony Corp.	X	

Manufacturers of cassette digital tape recorders include:

Compucord, Inc.
Telex
Dicom Industries
International Computer Products
Ampex Corp.
Mobark Instruments Corp.
Cipher Data Products
Auricord Division, Scovill

Section 22
SPECIFIC ION ELECTRODES

22.1 PRINCIPLES OF OPERATION

22.1.1 General

Ever since 1934 when Dr. A. O. Beckman developed the first commercial pH meter using potentiometric principles, attempts have been made to apply the same degree of operational ease of analysis of other ionic specifics. Ion selective electrodes, sometimes called Specific Ion Electrodes, are about ten years old. The first commercial specific ion electrode was marketed by Beckman in 1958. These electrodes measure directly the activity, rather than concentration, of an ion in solution. All ion selective electrodes operate on an ion-exchange mechanism whereby a Nernst potential is observed when an ion exchange membrane separates two solutions of a single salt concentration:

$$E = \frac{RT}{F} \log \frac{a'}{a_i''}$$

where a_A' and a_A'' represent the activities of the ion A^+ in the two solutions on either side of the membrane. R is the gas constant, T is the absolute temperature, and F is the faraday constant.

If an electrode is constructed by sealing an ion-exchange membrane, i.e., glass, at the end of a tube and filling the tube with a solution of a salt of constant composition, the electrode potential measured by using such a half cell depends only on the activity of A^+ in the external solution. This is true provided that an appropriate reference electrode is used to complete the circuit.

The two different concentration solutions of the same salt can be said to have different energy levels. Direct potentiometric measurement of ion activity is based on the different energy levels existing between two different states of the same matter; and that these differences are proportional to the relative population of the ions involved.

In electrolyte solutions these energy level differences are measured as an electrical potential. The Nernst equation of classical thermodynamics expresses this potential for a given activity of ion relative to a standard state, as follows:

$$E_{abs} = E^{o} - \frac{RT}{nF} \log (A^{+})$$

where E_{abs} = Potential observed for any given activity of A^{+}.

E^{o} = Potential of the standard state.

R = Universal gas constant in joules.

T = Absolute temperature in degrees Kelvin.

n = Number of electrons transferred in the reversible reaction.

F = Faraday constant

$\log (A^{+})$ = Natural log of the activity of A^{+}.

The Nernst equation predicts that at 25°C the potential at the glass electrode membrane will change approximately 59 millivolts for each decade change in activity of A^{+}.

Three basic types of electrodes exist--glass, solid, and liquid. In certain cases, especially when working with blood samples, a semi-permeable membrane

is used over the active membrane to protect the active membrane from interfering compounds such as proteins, enhance its specificity, or mechanically limit the loss of liquid membrane compounds.

The most important parameter in selecting an electrode is specificity. Given sufficient specificity, an electrode's sensitivity and stability are generally more than adequate for normal biochemical determinations. Unfortunately, only the hydrogen ion electrode can be considered to be relatively immune from interfering ions. All other electrodes are troubled to greater or lesser extents by interfering compounds. The importance of this interference is not always easy to determine in biological fluids. Since ion-selective electrodes respond only to ions, the total concentration of the individual species determined by alternate methods cannot be relied upon to correlate with the ionized values. Thus, a low reading for calcium that is induced by proteins may be due either to interference or an actual chelation of ionized calcium. The effects of interferences can be minimized by standardizing the concentrations of interferents. Thus, the effect of hydrogen ion interference can be minimized by adding a pH buffer to the sample. This type of standardization is not always possible or practical.

Ease of operation is second in importance to selectivity. While it is true that the use of electrodes eliminates the need for chemicals, spectrophotometers, polarographs, flame spectrophotometers, etc., electrodes do require a considerable degree of effort to achieve optimum results. Most of them benefit from being stored in concentrated solutions of the ions of interest. Semi-permeable membranes will require periodic replacement. Liquid ion-

exchangers will require periodic replenishment. All electrodes must be recalibrated frequently.

Sensitivity is of lesser importance. Generally, noise level is not a limiting factor, if sufficiently sophisticated electronic circuitry is used. Drift is the primary factor in limiting sensitivity. The zero reference position, the span, or the selectivity ratio of an electrode may change with time. Some of this drift may be overcome by frequent calibration, but this entails extra effort. Drift and selectivity shortcomings may be almost completely overcome by using electrodes only for monitoring relative changes during titrations rather than for direct potentiometric readings. Titration is, of course, very difficult for this application.

22.1.2 Glass Electrodes

Glass electrodes typically are used to monitor monovalent cations. The sensitivity order usually is $H^+ > Na^+ > Ag^+ > Li^+ > K^+ > NH_4$. By proper choice of glass, this order may be modified somewhat. For example, sodium may be slightly more sensitive than hydrogen ion, potassium may be slightly more sensitive than sodium, and potassium may be much more sensitive than lithium by properly choosing the glass composition. In any case, the amount of possible modification is severely limited, and glass electrodes are primarily used for hydrogen ion and sodium ion determinations. For these applications, they probably excel over other types of electrodes.

Beckman Instruments, Inc., and Corning Glass Works are the primary manufacturers of glass electrodes. A listing of specific ion electrode manufacturers is made in Table 22-1. This listing includes all types of electrodes even

Class	Ion of Interest	Principal Interferences	Principal Manufacturers
Glass	Hydrogen (pH)		B, C, CI, O
Glass	Sodium	Ag^+, H^+, Li^+, K^+	B, C, O
Solid	Bromide	CN^-, I^-, $S^=$	B, C, CI, N, O
Solid	Cadmium	Ag^+, Hg^{++}, Cu^{++}, Fe^{++}, Pb^{++}	O
Solid	Chloride	Br^-, I^-, $S^=$, CN^-, SCN^-, NH_3	B, C, CI, O
Solid	Cyanide	$S^=$, I^-	N, O
Solid	Cupric	Ag^+, Hg^{++}, Fe^{+++}	C, O
Solid	Fluoride	OH^-	B, C, CI, O
Solid	Iodide	$S^=$, CN^-	B, N, O
Solid	Lead	Ag^+, Hg^{++}, Cu^{++}, Cd^{++}, Fe^{++}	CI, O
Solid	Silver	Hg^{++}	C
Solid	Sulfide		B, C, N, O
Liquid	Calcium	Zn^{++}, Fe^{++}, Pb^{++}, Cu^{++}, Ni^{++}	C, O
Liquid	Nitrate	ClO_4^-, I^-, ClO_3^-, Br^-, $S^=$, NO_2^-	O
Liquid	Perchlorate	OH^-, I^-, NO_3^-, CN^-	O
Liquid	Potassium	H^+, Ag^+, Na^+, Li^+	B

B = Beckman Instruments, Inc.

C = Corning Glass Works

CI = Coleman Instruments

N = National Instrument Labs

O = Orion Research

Table 22-1. Commercially Available Ion Selective Electrodes

though not used for biochemical measurements, since they may be applicable to monitoring water or for other specialized applications.

22.1.3 Solid Electrodes

Solid-state electrodes use a low solubility precipitate usually having the same anion as the anion of interest. Orion is the foremost producer of electrodes using single-solid pellets, generally of silver halides or lanthanum fluoride. National Instrument Laboratories is the major producer of multi-particle precipitate electrodes in which silver halides are imbedded in an inert silicone rubber membrane. There is little to recommend one type over the other. The solid pellet type may be somewhat more durable, while the silicone rubber type may be less sensitive to surface poisoning. In either case, iodine has the best selectivity, chloride is second best, and bromide is poorest. The best selectivity for any one anion is achieved when the precipitate also includes this anion. The primary use of this type of electrode should be for determining chloride activity in serum or urine.

22.1.4 Liquid-Liquid Electrodes

This type of electrode uses liquid ion exchangers as the discriminatory membrane material. This technique greatly extends the range of possible materials that can be used to provide specificity. The greatest difficulty with this technique is in physically containing the ion exchanger. Beckman accomplishes this with a cellophane barrier or a series of capillary tubes containing the ion exchanger for their potassium electrode. Orion uses a flexible membrane, while Corning uses a porous glass frit for their calcium electrodes. All three types must be stored dry to prevent excessive dilution

of the liquid ion exchanger. Since glass electrodes and solid electrodes perform better if stored in concentrated buffer solutions, the liquid electrodes necessitate a separate handling procedure. Even when stored dry, liquid ion exchanger must be added to the electrodes periodically.

The potassium electrode marketed by Beckman provides excellent specificity and is relatively trouble-free, even when used with serum samples. The Orion calcium electrode provides equivalent performance, and the sensitivity appears adequate for the narrow physiological range of calcium activity in serum. Orion recommends restandardization between each serum sample. We have found that adequate results are obtained if a serum sample is followed by a large air bubble. If this is not done, however, following specimens will require extended times for stabilization. In any case, liquid ion exchanger electrodes require considerably more care than do other types.

22.1.5 Reference Electrodes

Most reference electrodes require a flow of electrolyte to maintain an electrical continuity between the measuring electrodes and the reference electrode. This flow occurs through a junction at the tip of the reference electrode and the rate of flow of the electrolyte is regulated by its hydrostatic pressure. This type of electrode would require that an external source of pressure be applied to initiate and maintain a flow. This would require a considerable amount of attention to prevent depletion of the electrolyte and subsequent drying of the junction.

The second approach is through the use of a salt bridge, which again would be difficult to adapt to a zero-g environment.

Beckman has developed a non-flowing reference electrode which should overcome the objectionable features found in the other two types, however, and make the measurement of ionic activities practical in space.

Unless an electrode pair is furnished for each measurement, the reference electrode will have to be switched between the reference electrodes. This will require special attention to shielding to avoid interference problems.

22.1.6 Specific Ion Amplifiers

Commercial pH meters capable of handling the signals from specific ion electrodes are quite simple. They consist of a high impedance amplifier (typically 1×10^{11} ohms) and a meter scale. Gain and zero offset controls are provided to calibrate the system.

The major functional problem in the measurement of blood gases in space will be the containment of liquid samples throughout the measurement and disposal process, coupled with a requirement that no air bubbles can be entrained in the specimen stream. A configuration to permit this can be designed, and a substantial amount of effort has already been expended in this direction.

An additional goal would be the simplification of the maintenance routine ordinarily associated with the measuring instrumentation, e.g., membrane replacement, cleaning and calibration.

22.2 APPLICATIONS

Specific ion electrodes can be used in the following functional program elements (FPE's):

5.9 Small Vertebrates (Bio D)
5.10 Plant Specimens (Bio E)
5.13 Biomedical and Behavioral Research
5.15 Life Support and Protective Systems
5.16 Materials Science and Processing
5.17 Contamination Measurements
5.23 Primates (Bio A)
5.25 Microbiology (Bio C)
5.26 Invertebrates (Bio F)
5.27 Physics and Chemistry Lab

They can also be used in General Purpose, Bioscience, Biomedical, Physics, and Chemistry laboratories.

Ion selective electrodes are used to monitor the activity of sodium, hydrogen, potassium, chloride and calcium in blood, serum, and urine. Another important use would be in detecting ionic contaminants that may be present in the water supply or in the fuel cell output. Ion selective electrodes also can be used to monitor spacecraft experiments, such as the growth of algae. The choice of electrode is a function of the ion to be measured rather than the sample to be tested. A listing of the various electrodes is made in Table 22-1. The "Class" column designates the preferred type of electrode; either glass, solid, or liquid. The "Principal Interferences" column lists the primary interfering ions that must be accounted for if maximum sensitivity and accuracy is required.

22.3 LOGISTICS

22.3.1 Packing and Launch

The electrode amplifier and readout system should require absolutely no precautions if reasonably rugged components are used. The electrodes can be transported either in individual wrappers or mounted in a flow cell assembly.

22.3.2 Installation

If the system is launched in a flow cell assembly, the system can simply be mounted at any convenient location. If the electrodes are individually wrapped, the procedure discussed in 22.3.5 must be followed.

22.3.3 Consumable Supplies

Membranes used to protect the electrodes from proteinaceous fluids such as blood must be replaced at one month intervals. Approximately 2 ml of a buffered solution should be used to rinse and soak each electrode after use. Calibrating solutions (approximately 5 ml/day) will be required for each electrode. Approximately 5 ml electrode filling solution will be required for each glass and liquid electrode at monthly intervals.

22.3.4 Accessories and Spare Parts

All the electrodes should be duplicated with a spare set.

22.3.5 Maintenance and Repair

Upon installation, or after a month's use, all electrodes should be refurbished. This requires membrane replacement, liquid ion exchanger replacement, and any salt bridge media additions. The electronic portions should not require maintenance. Zero-g sample handling techniques will be used. These procedures can be made available.

22.4 OPERATION

22.4.1 Warm-up and Speed-of-Operation

After the replacement of a membrane or after prolonged storage, the electrodes may require a 30 minute equilibration with a buffer containing an isotonic

concentration of the ion of interest. Also, most measurements will require closely controlled temperature conditions. An electrode block operated at 37°C typically requires one hour warm-up for good thermal equilibrium.

After warm-up, a set of standards and 12 samples generally can be analyzed within 30 minutes. With multiple electrodes in the same block, multiple ions may be analyzed with very little additional time.

22.4.2 Operation Skills

Good manipulative skills and an experienced operator are required to ensure continuing good performance.

22.4.3 Operating Procedure

Typical operation for electrodes enclosed in a block would be as follows:

Preparation:	Oven warm-up
Calibration:	Inject high standard Adjust gain to correct reading Inject low standard Adjust zero offset to correct reading Inject high standard Readjust gain
Measurement:	Inject sample Record after one minute Flush with buffer

22.4.4 Sample Preparation and Handling

Special sample preparation techniques are not usually required beyond those for sample handling in zero-g. In certain specialized cases, the sample may require dilution or a buffer may be added to a sample. This is done to standardize the sample pH or to swamp out interfering compounds. In a zero

gravity situation, specialized flow through cells must be devised. These must be designed to prevent the admission or generation of air bubbles.

22.5 INTERFACE

Critical interface problems should not be experienced with specific ion electrodes. Sample handling and data recording will be greatly simplified if an on-board computer is available. The nature of these electrodes makes them particularly appropriate as components in other laboratory instruments and vehicle systems.

The electrometer for specific ion electrodes normally operates from 115 volt, 60 Hz power; with some instrument modification, 400 Hz or 28 V dc power could be used.

22.6 SAFETY

22.6.1 Flame Hazards

Specific ion electrodes present no flame hazards.

22.6.2 Microbiological Hazards

Possible microbial growth in the buffer solutions used for specific ion electrodes can be effectively controlled with bactericidal agents.

22.6.3 Electromagnetic Interference

The only probable source of interference is from the power supplies, especially from dc-to-dc converter types operated from 28 V dc. LC filtering and feed-through bypass filtering and a small amount of shielding will effectively limit interference conditions or radiation to within acceptable limits. The extremely high impedance of most electrodes results in circuitry which can be

very susceptible to radiated RF energy. The electrodes should be as close as physically possible to the electronics, and cables should be double-shielded to avoid undesirable pickup. Design of the ground system is also critical. Extra shielding of the electronics may be necessary to provide complete and sufficient protection from radiated energy. The filtering used to control conducted interference will be equally effective in limiting circuit susceptibility to conducted RF and transient energy to within acceptable limits.

22.6.4 Ionizing Radiation

Specific ion electrodes neither produce nor are interfered with by ionizing radiation.

22.6.5 Physical Hazards to Personnel

Because breakage of the specific ion electrodes would present a particular hazard, shielding from mechanical damage of the electrodes is recommended.

22.7 MODIFICATIONS

1. The electronic components should be replaced by space-qualified components.
2. Any electrodes requiring liquid ion exchangers should be modified to have a bladder rather than a large air space.
3. Flow-through systems that eliminate air bubbles must be designed.
4. With fluid filled reference electrodes, a positive pressure must be applied or a non-flowing junction must be employed.
5. Fluid-filled electrodes must be equipped with an absorbant material to retain the fluids at the sensing surface.

6. Fragile glass electrodes should be shielded from mechanical hazards to prevent breakage.

22.8 AVAILABLE INSTRUMENTS

A list of commercially available electrodes and manufacturers is listed in Table 22-1.

Section 23
SPECTROPHOTOMETERS

23.1 PRINCIPLES OF OPERATION

Spectrophotometers are used primarily for the analysis of solutions. Instruments are available to cover the wavelength range from the ultraviolet through the infrared. In operation, a measurement is made of the relative intensity of the radiant energy transmitted at selected wavelengths through the sample. As the wavelength is varied, a plot of intensity (or absorptivity) is produced--this is called an absorption spectrum. Generally, the relative intensity of the energy transmitted by the solution is compared with that by a reference solution which does not contain the desired constituent. The absorption wavelengths determine the identity and/or molecular structure of the molecules, and the amount of absorption is proportional to the concentration. The types of absorption vary through the wavelength range. In the infrared region, which covers the wavelength range of 1 to 1000 microns, the absorption is caused by rotation and vibration of the molecules. In the near infrared and visible regions, the absorption is due primarily to vibration of the atoms; and in the ultraviolet, the absorption is caused by electronic transitions within the atoms. The amount of absorption is proportional to the thickness of the cell and independent of the intensity of the radiant energy. All spectrophotometric data, therefore, requires the measurement of the difference between the transmitted energy before and after the sample is placed in the light beam and/or a comparison with a known reference.

The optical path of a UV-visible spectrophotometer is shown in Figure 23-1, and that of an infrared spectrophotometer in Figure 23-2.

Different types of dispersion systems are used, depending upon the range of monochromatic light wavelengths needed. In the far ultraviolet range, gratings are required to provide resolution and sufficient energy. In the near ultraviolet range, either gratings or prisms are used to disperse the light. The visible region is usually displaced with a grating or prism; filters may be used when high resolution is not required. In the infrared region, both prisms and gratings are used to provide the required dispersion.

The materials used throughout these wavelength regions must be selected since no single material provides transparency over the entire region. Quartz is used in the ultraviolet for prisms and cells. The visible region accommodates glass cells and prisms, and in the infrared region NaCl, KBr, or specially synthesized crystals for windows and prism materials must be used. Table 23-1 lists the characteristics, advantages, and disadvantages of different window materials for infrared spectroscopy.

Similarly, sources of radiant energy vary for the three regions. The ultraviolet uses discharge lamps containing hydrogen, Xenon, or mercury, whereas a tungsten lamp is adequate for the visible and near infrared regions. For the longer wavelength infrared region, a Nernst glower or heated blackbody source provides the highest efficiency.

Detectors also must be selected for the wavelength region of interest. Photomultipliers and phototubes are useful through the UV and visible regions. The

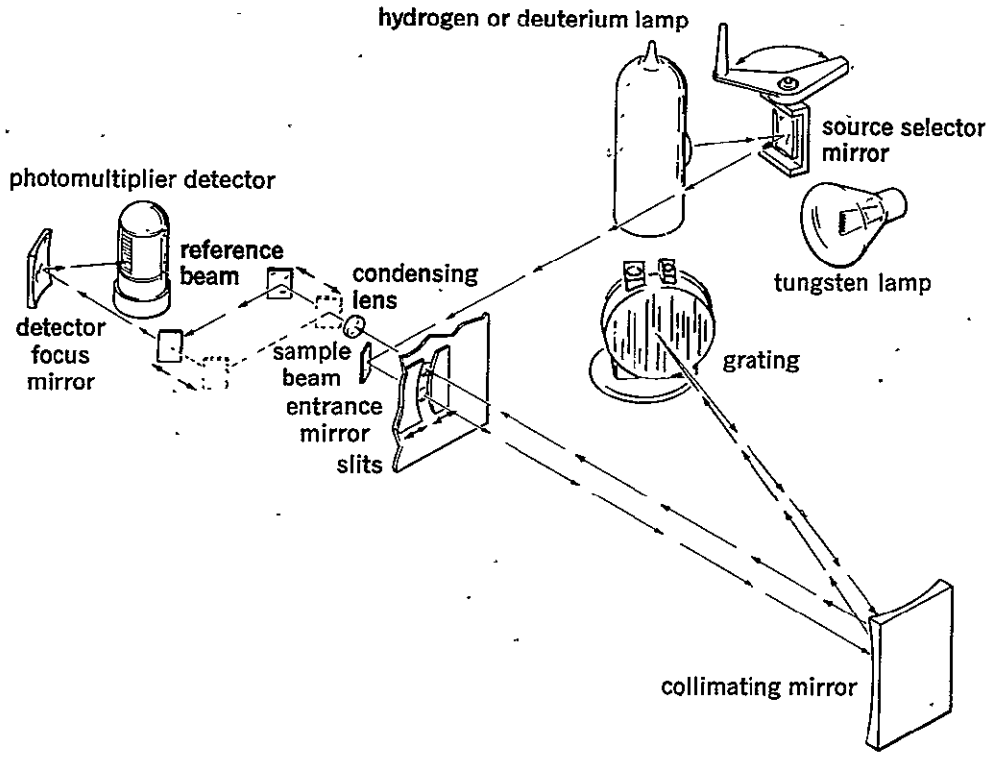


Figure 23-1. Ultraviolet-Visible Spectrophotometer Optical Path Diagram

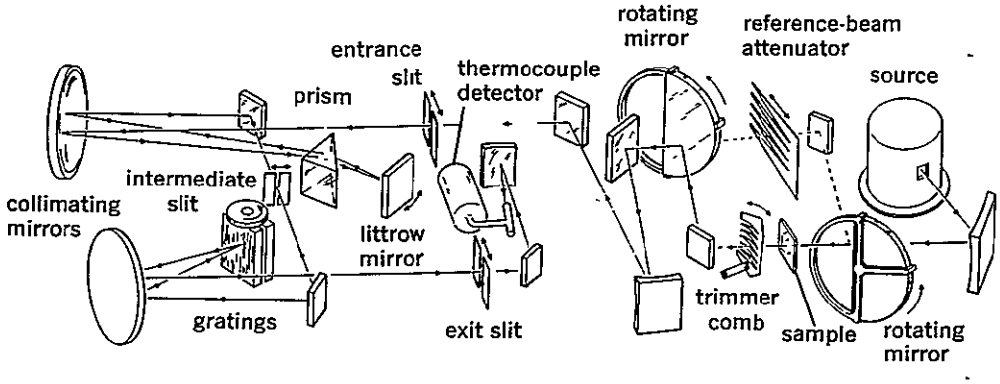


Figure 23-2. Infrared Spectrophotometer, Optical Path Diagram

Materials	Range	Advantages	Disadvantages
Sodium Chloride (NaCl)	0.2 to 16 microns	Inexpensive (Most often used)	Hygroscopic—can't be used with aqueous solutions or solutions of high water content
Potassium Bromide (KBr)	1.0 to 25 microns	Only slightly more expensive than NaCl (Most often used)	Hygroscopic—should not be used with aqueous solutions and solutions of high water content
Barium Fluoride (BaF_2)	0.2 to 10 microns	May be used with aqueous solutions	Limited wavelength range, relatively more expensive than NaCl
Calcium Fluoride (CaF_2)	0.21 to 8 microns	May be used with aqueous solutions	Limited wavelength range, relatively more expensive than NaCl
Cesium Bromide (CsBr)	0.5 to 35 microns	Wide wavelength range	Hygroscopic—should not be used with aqueous solutions and solutions of high water content
Cesium Iodide (CsI)	1.0 to 50 microns	Wide wavelength range	Hygroscopic—should not be used with aqueous solutions and solutions of high water content
Lithium Fluoride (LiF)	0.12 to 6 microns	Low wavelength range Low solubility in H_2O High transmission	Limited IR wavelength
Irtran-2	0.6 to 14 microns	Inert to strong inorganic acids, bases and other corrosive samples May be used with aqueous solutions	High index of refraction results in high reflection losses and produces fringe pattern effect even in spectra of filled cells Very expensive
(Silver Chloride) (AgCl)	2.5 to 22.5 microns	Insoluble in: water, Ammonium Hydroxide, Sodium Sulfate, Potassium Cyanide	Darkens with UV irradiation, fairly soft
Silver Bromide (AgBr)	0.45 to 35 microns	Insoluble in: water, Acetone Nitrobenzene, Methanol, Saturated alcohols Does not darken as much as AgCl with UV irradiation Less expensive than Irtran-2	More expensive than AgCl Darkens in time with UV irradiation
Polyethylene	From 20 microns on throughout the entire infrared region	Wide wavelength range, inexpensive, may be used with aqueous solutions	Has strong absorption bands in fundamental region, porous to certain substances
Thallium Bromide Iodide (KRS-5)	0.5 to 40 microns	Wide wavelength range Good for ATR work Not hygroscopic	Poisonous, high refractive index

Table 23-1. Window Materials for Infrared Spectroscopy Sample Cells

near infrared and infrared regions must use thermocouples, thermistor bolometers, or one of a variety of solid-state photo-voltaic cells.

Spectrophotometers may be single beam or double beam in operation. In the single beam design, only one light beam exists and it is necessary to interchange the sample cell with the reference cell to perform the absorption comparison. In double-beam operation, two light beams are formed from a single source and monochromator, and either the ratio of the two signals is measured or a difference signal provided.

To provide a wavelength scan, the prism or grating is normally rotated which causes the dispersed spectrum to sweep across the exit slit. In high resolution instruments, a double monochromator is supplied which produces twice the dispersion and results in a fraction of the scattered light which is important for many applications. The simple colorimeter type instruments are used in the visible spectrum; they generally vary wavelength by changing interference filters or by moving a filter wedge.

The resolution of a spectrophotometer is determined by the width of the entrance and exit slits, and since the energy transmitted through the system is proportional to the area of the slit, there is always a trade-off between energy (or noise) and desired resolution.

23.2 APPLICATIONS

Spectrophotometers are applicable to the following functional program elements (FPE's):

5.9 Small Vertebrates. (Bio D)
5.10 Plant Specimens (Bio E)
5.13 Biomedical and Behavioral Research
5.16 Materials Science and Processing
5.17 Contamination Measurements
5.18 Exposure Experiments
5.23 Primates (Bio A)
5.25 Microbiology (Bio C)
5.26 Invertebrates (Bio F)
5.27 Physics and Chemistry Lab

Almost all gases and liquids show absorption in the infrared region. The spectrum is relatively complex, but each constituent of the sample has a unique absorption spectra which can be readily identified. There are many overlapping bands, however, particularly since infrared absorption is dependent upon rotation of bonds in the molecule and, therefore, all molecules with similar bonds have similar absorption characteristics. Water vapor is a strong absorber throughout most of the infrared region, leaving very few windows where other samples can be accurately measured.

The visible region is particularly useful for the analysis of biochemical substances. A colorimeter is a standard instrument used by clinics for blood and urine analyses. Changes in color and absorptivity of prepared samples when reagents are added are quantitatively measured at selected wavelengths. This provides direct measurements of concentration for a large number of substances.

The ultraviolet region provides extremely high sensitivity to compounds which have ultraviolet absorption, generally resulting in much shorter path lengths and excellent sensitivity for trace contaminants. In the vacuum ultraviolet

region, oxygen and nitrogen have strong absorption bands in addition to water vapor, so that it is necessary to remove air from the entire instrument and sampling region during the analysis. Most organic compounds with double bonds exhibit characteristic absorption bands in the ultraviolet and visible regions while characteristic absorption bands of organic functional groups are found in the infrared region. Organic compounds containing double and triple bonds exhibit resonance in the ultraviolet region. The absorption maximum is shifted with different solvents and reagents. This is an important measure of the presence of many compounds. Aromatic hydrocarbons have high absorptivity in the ultraviolet.

Organic molecules possessing a dipole moment produce infrared absorption bands. Certain absorption bands correspond to specific linkages and groups, thereby providing good specificity in the infrared. A catalog of absorption spectra is necessary to identify specific compounds. Very high resolution is often required to determine the presence of trace quantities in multicomponent mixtures.

The preparation of the reference solution and solution containing the desired constituent is the most critical step in a spectrophotometric determination. This requires careful measurement of quantities of reagents and the use of solvents which have no absorptivity in the region of interest. Errors in the analysis can be caused by temperature changes or evaporation of a volatile constituent. The presence of bubbles or particulates can reduce the accuracy techniques as used in ground-based laboratories, and will not be adequate under conditions of zero-gravity due to bubble formation and unequal distribution of the liquid in partially-filled cells.

23.3 LOGISTICS

23.3.1 Packing and Launch

Spectrophotometers require no special packing procedures other than those normally provided for precision photo-optical instruments. Many components of spectrophotometers are often provided as subassemblies due to the requirement for changing sources, detectors, and sample cell compartments. These can easily be packaged separately to better withstand the launch environment. Components utilizing various salts, such as prisms and windows, should be carefully desiccated to prevent fogging in the presence of water vapor. Sample cells are necessarily fragile since they must be constructed of glass or other material consistent with the operating wavelength region and, therefore, are subject to transit damage unless properly packaged.

23.3.2 Installation

All spectrophotometers are table-mounted and require only proper clamping to be consistent with a zero gravity laboratory. Good temperature control of the instrument and samples is generally necessary to maintain good accuracy since many analyses are temperature-dependent. The relative humidity should be maintained below 40 percent, particularly for infrared instruments, to prevent fogging of the optical materials.

23.3.3 Consumable Supplies

Consumable supplies include additional sample cells, reagents, and calibration gases. It is desirable to provide the standard solutions and reagents pre-mixed wherever possible. Due to the absence of gravity, syringes will be necessary to transfer liquids, and either a large quantity of syringes must

be on hand or cleaning and rinsing steps must be performed aboard. The former approach is recommended.

23.3.4 Accessories and Spare Parts

The wide wavelength range included from the vacuum ultraviolet to the far infrared demands that a large variety of sources, monochromators, and detectors be available as accessories. Standard instruments which cover the ultraviolet, visible and infrared regions are supplied with several modular source and detector compartments. Similarly, both the gas and liquid cells used with these instruments must be of proper material so that high transmissivity is obtained for each specified wavelength range.

In addition to the standard accessories required to provide the normal versatility available with spectrophotometers, there are a number of accessories which can be added to convert the instrument to a reflectometer and/or a fluorometer. For reflectance work, an integrating sphere is normally used which provides the best accuracy for observing diffuse samples. The addition of a fluorometer accessory requires another ultraviolet source and either a monochromator or filter to select a specific excitation wavelength. The spectrophotometer is then set to the fluorescing wavelength for the analysis. In general, most accessories for spectrophotometers are easily added to standard instruments and present no problem for assembly and operation in the space-borne laboratory. Recent instrument designs use an optical bench-type of construction (Paragraph 3.19) which permits the interchange of accessories and monochromators so that a single set of modules can cover the desired wavelength range. This provides a much greater versatility for analysis and yet does not utilize additional work-bench space.

Other major accessories will include readout modules. A strip chart recorder is desirable when scanning the wavelength spectrum. For applications where a single wavelength is to be monitored, the output can be read by a digital voltmeter, meter, or printer. The majority of analyses to be performed in the spaceborne laboratory will be for specific routine tests and, therefore, will not require the scanning capability.

To prevent fogging of optical materials utilized for the infrared region, it is necessary to store cells, windows, and replaceable optical components in a desiccated container. This is particularly important for spaceborne laboratories where the relative humidity may exceed normal ambient conditions. Storing these components in an accessible space-vacuum container to provide the lower pressure is certainly worthy of consideration.

23.3.5 Maintenance and Repair

Because of modular construction, spectrophotometers are particularly suitable for major subassembly replacement in the event of malfunctions. An entire malfunctioning subassembly should be replaced rather than attempting to repair components. Certain optical components, such as sources, detectors, and prism or grating interchanges, can certainly be maintained aboard and will normally require replacement or refocussing on a routine basis.

23.4 OPERATION

23.4.1 Warm-up and Speed-of-Operation

The warm-up time required for a spectrophotometer is dependent upon the design of the specific instrument. In general, single-beam instruments require a longer stabilization time than double beam since the output is directly

affected by changes in source intensity and/or detector drift. Instruments of high wavelength accuracy are often temperature controlled to provide accurate readings of wavelength. In this case, it is necessary to maintain the operating temperature continuously to prevent excessive drift or inaccuracy of wavelength. Double-beam instruments stabilize much more quickly since changes in source and/or detector are compensated. Nevertheless, a 30-minute warm-up period is desirable for any spectrophotometer.

The set-up time of a new installation is relatively short, requiring only that suitable power be made available and connection to the readout device. The sample-handling system set-up time is dependent upon the type of analysis and requirements for calibration gases, etc.

The operating time is strictly dependent upon the application. Instruments are available which scan their entire wavelength range in less than one minute, while others require one or more hours. For trace analysis, or where high spectral resolution is necessary, it is required that the scanning speed be reduced to faithfully reproduce the sharp spectral bands. Visible spectrophotometers are generally set to one or more wavelengths and often no scanning is involved. The response time of most instruments at a single wavelength is usually a few seconds. UV instruments are similar in respect to the infrared spectrophotometers in terms of requirements for a slow scan. The UV spectral lines are generally quite sharp, although the length of the spectrum is limited and, therefore, the useful range of the instrument can generally be scanned in a few minutes. Routine sampling generally requires as much time to prepare and calibrate the sample as it does to perform the analysis.

23.4.2 Operation Skills

The spaceborne spectrophotometers can be operated by personnel with Earth-based experience on similar equipment. Some special skills will have to be developed in the transfer of liquid samples and reagent addition under zero-gravity conditions. A small centrifuge should be available to spin down samples in their liquid cells to eliminate bubbles and provide adequate mixing. The centrifuge can be relatively slow since it is only necessary to generate a one-g acceleration. The major item to be learned by the operator is the proper assembly of the various modules and accessories for each specific instrument. In some cases, this may be as simple as replacing a filter. In other cases, the addition of a major accessory such as a reflectance attachment will require relatively complex calibration and realignment procedures. As a minimum, spectrophotometers should be operated by technical level personnel with considerable previous experience. Extensive pre-flight training of personnel unfamiliar with spectrophotometric equipment would be essential.

The interpretation of the spectra often requires considerable experience. In cases where only a single wavelength is to be monitored, an analysis procedure can easily be provided.

23.4.3 Operating Procedures

It is difficult to specify a typical operating procedure for a spectrophotometer, since there is a wide variety of instruments covering a large wavelength range. All spectrophotometers, however, use a source. This may be a discharge lamp in the case of the UV, a tungsten lamp for the visible and near IR, and a lower for the IR region. Typically, the source is turned on first

and permitted to stabilize for several minutes. An electric check for zero and 100 percent transmission is then normally made. For double-beam operation, the reference cell is placed in the reference beam and the sample cell is filled with the desired gas to be measured and placed in the sample beam. For operation where a wavelength scan is to be made, the slit is adjusted to provide the desired energy and the pertinent switch selected for proper time constant. The scanning speed is then chosen commensurate with the preset resolution and time constant. For scanning type instruments, a spectrum is generally traced on a recorder chart. The tracing is either in percent transmission or absorbance, also selectable by a switch. For wide wavelength range scans, it is often necessary to substitute sources and/or detectors to provide adequate energy and signal-to-noise ratio over the entire band.

23.4.4 Sample Preparation and Handling

The majority of samples used for the ultraviolet, visible, and near infrared instruments are liquids. The use of sample cells in a space environment will require special cell designs which can be filled by means of a syringe through a serum cap. Where necessary, centrifugation will provide the necessary mixing and proper displacement of the liquid within the cells. For gas samples, which are analyzed primarily in the infrared region, a sample-handling system must be designed which can introduce both calibration and test samples. A connection to space vacuum would provide the necessary pressure reduction and sample flow. If solid samples are to be analyzed, a pellet-making operation is required. This is generally a press capable of compressing the solid sample mixed with KBr salt into a thin pellet which can then be placed in a special sample holder. These types of operations are not recommended for operation in the spaceborne

laboratory due to contamination of the atmosphere by particulates if the sample crushes or disintegrates. It should be possible to return any desired solid samples to Earth where they can be prepared in proper pellet form for analysis on the ground or returned to the space vehicle where the analysis requires this step.

Samples must be disposed of after the analysis is completed. Since rinsing of sample cells is not easily accomplished under zero-gravity conditions, a large number of sample cells must be made available so that the used samples can either be returned to Earth for cleaning or discarded.

One unique method of handling liquid samples and reagent mixing has been developed by Beckman Instruments, Inc., for space applications which utilizes plastic bags into which the sample and reagents are injected. The bag is then kneaded by hand until proper mixing is achieved and then placed in a special holder which compresses the bag and holds it in position for inserting into the light beam. A flick of the wrist moves the fluid to one end of the bag and removes bubbles. This technique is particularly amenable to the measurement of body fluids where the prepackaged reagents can be contained within the bag requiring only that the urine or blood sample be introduced.

23.5 INTERFACE

23.5.1 Interface with Other Laboratory Instruments

Spectrophotometers are valuable additions to other laboratory instruments, such as the gas chromatograph, since they can provide a more positive identification of constituents in a multicomponent mixture. The samples are not consumed or

destroyed and, therefore, can be transferred from the optical measurement to other analytical instruments such as the mass spectrometer. The interface normally is by manual transfer of sample rather than direct connection since the quantity of sample required for spectrophotometric analysis is generally quite a bit larger than needed for the gas chromatograph or mass spectrometer. With the wide variety of accessories available for standard spectrophotometers, the instruments can easily be converted from an ultraviolet spectrophotometer to a visible colorimeter and/or a fluorometer or reflectometer.

A computer is often used in conjunction with a spectrophotometer to provide the constituent concentration analysis where multi-component mixtures are being analyzed. The computer interface consists of encoders mounted on the wavelength and recorder drives so that the proper digital signals are provided to the computer with simplified interface circuitry.

When used with liquid samples, the spectrophotometer interface with other instruments generally involves only the sample itself. This may be in the form of a pH measurement to insure the proper reaction or an aliquot of the sample for introduction into a gas chromatograph.

23.5.2 Interface with Vehicle System

Spectrophotometers will require power from the vehicle system. Instruments using water cooling for temperature control should be avoided. A vacuum line will be required for flushing samples, particularly for the infrared region where gas samples are being analyzed. It is desirable to maintain fairly constant temperature for most spectrophotometers to avoid frequent

recalibration. The relative humidity should remain below 40 percent or special means provided for desiccating the instrument.

For an advanced mode of operation, the output spectra could be accepted and serviced by the data management system. Programmed analyses could be made and comparisons made with known spectra. However, less automated operation would probably be preferred by many investigators. Most laboratory workers expect to see the spectra drawn on a strip-chart recorder. The data management system could be used to store, retrieve, and display sample spectra stored on microfilm.

Additional interfaces are primarily in the sample preparation area where it will be necessary to supply centrifugation and means for storing and disposing of samples.

23.6 SAFETY

23.6.1 Electromagnetic Interference

Spectrophotometers are dc or low frequency ac instruments, and in most cases should not radiate unwanted RF radiation. They are susceptible to external radiation, however, because of the extremely low signal levels associated with the measurement. Most commercial instruments are sufficiently well shielded. However, the close proximity of transmitting equipment and the possibility of high levels of ionizing radiation from space could result in higher noise levels when operated in a spaceborne laboratory. In general, presently required EMI specifications for spaceborne equipment must be significantly reduced in order to utilize any standard commercial instrumentation. The requirements should be reviewed for each space mission and relaxed in those frequency bands where

some interference would be of no consequence. This would be a less costly approach than to redesign the commercial equipment to minimize radiation.

23.6.2 Physical Hazards to Personnel

Probably the major hazard to personnel during the operation of spectrophotometric equipment is the sample handling of materials and cells. It is necessary to use glass cells of fragile material in order to provide the transmissivity in specific wavelength regions. Shattering of cells or optical materials could produce a serious hazard to the space environment since no settling of the fragments would occur under zero-gravity conditions. This may require that all sample-handling steps be performed in a special enclosure or hood so that accidental breakage would not contaminate the entire spacecraft.

When working with gases, mixing and purging are necessary to prepare samples of the proper operating pressure. All stainless steel sampling systems can be fabricated and are preferable over glass systems for space applications. Vacuum lines to space will probably be utilized and this could produce a hazard in the event of breakage or leakage.

Ultraviolet sources produce a serious eye hazard and should be shielded during operation. The sharp corners and protruding knobs of most spectrophotometers may present a hazard when operated in space.

23.7 MODIFICATIONS

The following modifications are necessary for spectrophotometers to be used in Space Stations:

1. A method must be provided for tying down the instruments to the work bench and either magnetic or Velcro strips must be supplied for holding the various components for the sample-handling system to the working surface.

2. The liquid sample cells must be modified so that they are sealed and contain a septum cap for penetration by a syringe. A centrifuge and mating fixture must be designed to accommodate the cells so that they can be properly filled without bubbles.

3. A gas sample-handling system, probably of stainless steel, must be designed to admit sample gases to the spectrophotometer.

4. Many accessories for spectrophotometric equipment utilize alignment pins and depend on gravity for proper positioning in the optical train. These must be modified to insure positive clamping of the interchanges.

5. Sampling compartments generally depend on gravity for placement of sample cells and movement of cells in and out of the beam. Positive locking devices must be designed to incorporate the sealed cells, and cell placement mechanisms must be reviewed for compatibility with the zero-gravity laboratory.

6. Spectrophotometers with built-in recorders must be modified to insure proper operation of the pen and paper supply in the absence of gravity.

7. The mechanical configuration of each spectrophotometer must be reviewed for launch environments and special tie-downs and shock mounts provided to withstand the launch environment.

8. Switches and knobs should be reviewed to insure ease of operation under conditions of zero gravity. Projections and sharp corners should be protected or eliminated.

9. Spectrophotometers generally include a wide variety of accessories and separate power supplies for sources and readout equipment. These should be integrated into a console with interconnections simplified where feasible.

10. All materials should be reviewed to minimize out-gassing possibilities which could produce toxic compounds within the closed atmosphere.

23.8 AVAILABLE INSTRUMENTS

Major instruments and their specifications are shown in Table 23-2 for UV-visible spectrophotometers, Table 23-3 for infrared spectrophotometers, and Table 23-4 for colorimeters.

Company	Model	Range (nm)	Readout Type	Adaptable To
American Instrument Co.	4-8202	200 to 800	Meter, Recorder	FLP, RM
	SPF-1000	200 to 1,000	Meter	FLP, RM
Bausch & Lomb, Inc.	Spectronic 600	200 to 800	Meter	FP, FLP, RM
	Spectronic 505	200 to 800	Recorder	FP, FLP, RM
Beckman Instruments, Inc.	DB-G	190 to 800	Meter, recorder, and DCC	AA, RM
	Std Direct Reading DU-2	190 to 700 (opt to 800)	Meter, recorder (opt)	FP, FLP RM, SEM
	DK-2A	Ratio recording: 185 to 3,500; energy recording: 185 to 3,500	Recorder, opt DCC	FP, FLP RM, SEM
	Kintrac VII	190 to 800	Meter plus recorder, opt DCC	None
	ACTA III	160 to 1,000	Digital Recorder BCD	FLP, RM, SEM

AA Atomic Absorption
FP Flame Photometry
FLP Fluorophotometry
RM Reflectance Measurement
SEM Spectral Energy Measurement

Table 23-2 (Sheet 1 of 2). UV-Visible Spectrophotometers

Company	Model	Range (nm)	Readout Type	Adaptable To
Cary Instruments	14	186 to 2,650	Recorder	FP, FLP, RM, SEM
	15	185 to 800 (170 to 600 opt)	Recorder	FLP, RM
	16	Std: 186 to 800, low UV, 170 to 600	Meter Recorder	RM
Coleman Instruments	14	325 to 825	Galvanometer	FP, FLP
	Junior IIA	325 to 825	Galvanometer	FP, FLP
	44	325 to 825	Meter	FP, FLP
DuPont Co.	400 Photometric Analyzer	200 to 1,000	Meter, Recorder	RM
General Electric Co.	7015E30	380 to 700 (380 to 1,000 opt)	Recorder	RM
Perkin-Elmer Corp.	202	190 to 750	Recorder	RM
	356	185 to 850 (opt to 1,200)	Recorder	FLP, SEM
	402	190 to 850	Recorder	RM

AA Atomic Absorption
FP Flame Photometry
FLP Fluorophotometry
RM Reflectance Measurement
SEM Spectral Energy Measurement

Table 23-2 (Sheet 2 of 2). UV-Visible Spectrophotometers

Company	Model	Optical System	Monochromator	Wavelength Range (cm^{-1})
Bausch & Lomb, Inc.	Spectronic 270 IR	DB: OPN	Double grating with slide interchange 4 stray light filters	4,000 to 400
Beckman Instruments, Inc.	IR-9	DB	D-KBr prism, 2 gratings	4,000 to 400
	IR-12	DB	S-filter, 4 gratings	4,000 to 200
	IR-20A	DB	S-filter grating	4,000 to 250
	Microspec	DB	S circular var filter	4,000 to 690
Perkin-Elmer Corp.	221	DB: OPN	NaCl prism (others opt)	2.0 to 15.5 microns
	621	DB: OPN	Filter grating	4,000 to 200
	137G	DB: OPN	Filter grating	12,000 to 3,900; 4,100 to 1,300
	257	DB: OPN	Filter grating	4,000 to 625
	457	DB: OPN	Filter grating	4,000 to 250

DB Double Beam
OPN Optical Null

Table 23-3. Infrared Spectrophotometers

Company	Model	Optics	Monochromator	Readout	Wavelength
American Instrument Co.	4-7102	SB	Filter	F, T, OD	250 to 700
Bausch & Lomb, Inc.	Spectronic 20	SB	Filter	F, T, OD	250 to 700
Beckman Instruments, Inc.	B	SB	Prism	T, A	325 to 1,000
Coleman Instruments	B	Vis filter	Color filters	T, A	400 to 700
Dow Chemical Co.	Computer Colorimeter	SB	Filter	A	420 to 660
DuPont Co.	400	SPB, SB DB	Filter	A	200 to 1,000
Honeywell Inc.	4600, 4605 4610, 4615 4620	SB	Filter	T	375 to 765 (20 filters)
Photovolt Corp.	401	SB	Filter	T, A	420 to 650
	402E	SPB	Filters	T	Vis
	402EF	SPB	Filters	T, F	UV

DB Dual Beam
SB Single Beam
SPB Split Beam
vis Visible

Table 23-4. Colorimeters

Other manufacturers of UV-visible spectrophotometers include:

Ace Scientific
Agricultural Specialty Company
Boehringh Manheim Corp.
Calbiochem
Canalco
Cary Instruments
Cosmos Scientific
Durrum Instrument
EG & G
Engis Equipment
General Electric
Gilford Instrument Laboratories
Heath
Hughes Aircraft
Jarrell-Ash
Kollmorgen Color Systems
Laboratory Data Control
McKee-Pederson Instrument
Perkin-Elmer
Philips Electronic
Phoenix Precision Instruments
Process & Instruments
Pye/Unicam Ltd.
Radiation Equipment
Rudolf Instruments Engineering Co., Inc.
Scheer Instrument Company
Schoeffel Instrument Company
Shimadzu Seisakusho, Ltd.
Space Instruments Research, Inc.
Spectrex Corp.
Spex Industries
G.K. Turner Associates
Varian
Carl Zeiss

Other manufacturers of infrared spectrophotometers include:

Agricultural Specialty Company
Balder Cryogenic
Block Engineering
Cary Instruments
Engis Equipment
General Electric
Hughes Aircraft
Jarrell-Ash
Philips Electronic
Pye/Unicam, Ltd.
Spex Industries, Inc.
Systems Research & Development Co.
Varian
Wilks Scientific

Other manufacturers of colorimeters include:

Brinkmann Instruments
Calbio Research
Central Scientific Co.
Clay-Adams
Delta Scientific
Evans International
Fisher Scientific
A. GallenKamp & Co.
Gilford Instruments
Golden Instruments
H. H. Chemicals
Hach Chemicals
Hellige
Hollywood Instruments,Inc.
Hunter Associates
Joyce, Loebl
Klett Manufacturing
Kollmorgen Color Systems
LKB Instruments
E. Leitz
Loribond of America, Inc.
Magnuson Engineering,Inc.
Manufacturers Eng. & Equipment
Particle Data
Phoenix Precision Instrument Company
Photobell
Photoronic
Pye/Unicam, Ltd.
Schoeffel Instrument Co.
Searle/BMI
Martin Sweets
Technical Operations,Inc.
Waters Associates
Carl Zeiss

Section 24
X-RAY SPECTROMETERS

X-rays occupy the portion of the electromagnetic spectrum between 0.01 and 100 Å in wavelength. Their range of quantum energy is from 2×10^{-6} to 2×10^{-10} erg (10^6 to 100 electron volts). X-ray analytical methods are based on the following:

- Fluorescence
- Absorption
- Emission
- Diffraction

Qualitatively and quantitatively, X-ray techniques are used to determine the elemental composition of mixtures and also to determine atomic arrangement and spacings in crystalline materials. Fluorescence and Absorption are the most common X-ray methods for quantitative chemical analysis. These two techniques account for the majority of the chemical analyses routinely handled in quantitative analysis. Only these two methods are discussed in detail in this survey.

24.1 PRINCIPLES OF OPERATION

In conventional X-ray spectrography or spectrometry using an X-ray tube as the excitation source, the sample to be analyzed is subjected to a very intense X-ray flux of approximately 10^{13} photons per second. The resultant characteristic X-rays are then received or dispersed and counted by a wide variety of detectors such as Geiger-Müller tubes, ionization chambers, scintillation counters, proportional counters, electron multiplier tubes or photoconductive

cadmium sulfide crystals. The left side of Figure 24-1 shows the X-ray diffraction system. Wavelength dispersion results in excellent spectral resolution, but the geometrical losses and low diffraction efficiency reduce the X-ray intensity by a factor of more than one million. These wavelength dispersive systems and direct absorption methods require a stable 2 to 3 kilowatt power supply, a precision goniometer, and relatively complex electronic readouts. The spectrographs are bulky and expensive, costing approximately $20,000.

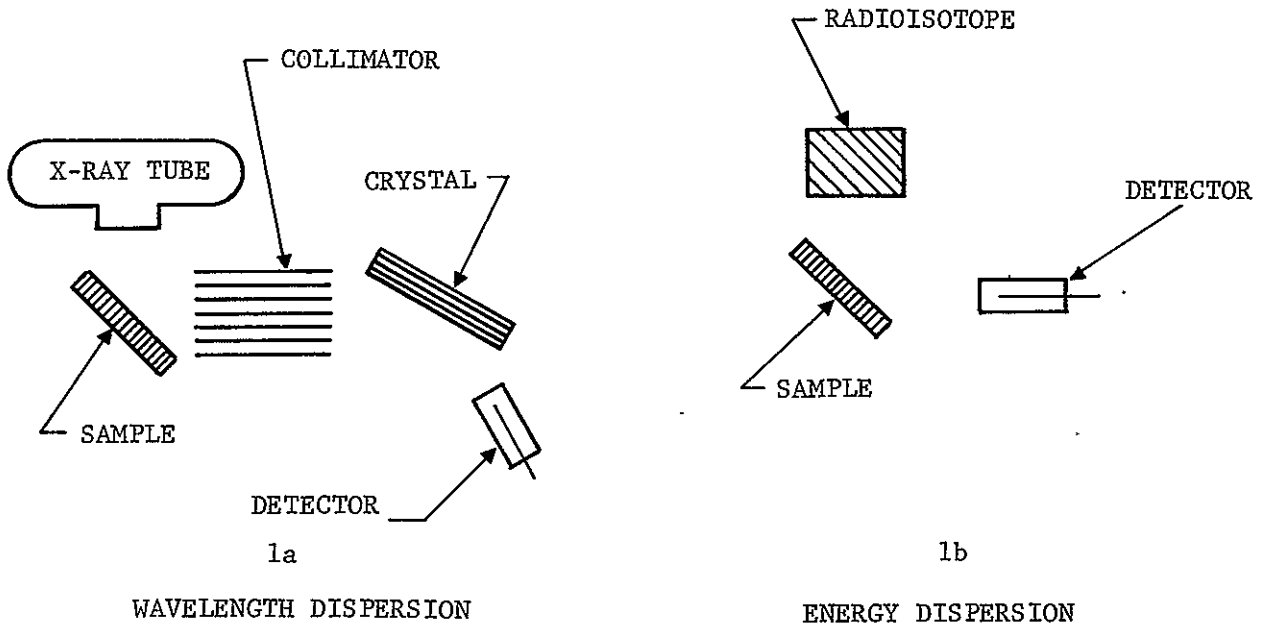


Figure 24-1. Comparison of Conventional X-ray Spectrographic Optics and Energy Dispersion

In more recent years, the X-ray source has been replaced by a radioisotopic X-ray source. The resulting system is relatively simple. (Figure 24-1, left side). Recently developed lithium drifted silicon or germanium detectors can

be used in this system. In this form, the X-ray spectrometer costs substantially less, has large resulting power savings, and is smaller. The nondispersive radioactive X-ray source system is the only one to be seriously considered for spacecraft applications. The following technical discussion is directed specifically to the radioactive isotope X-ray system since almost all factors relating to its flyability are substantially better than the older, more conventional, X-ray tube systems.

24.1.1 X-ray Fluorescence Principles

When X-rays of sufficient energies strike an atom and cause photoelectric ejection of electrons from an inner atomic shell, electrons in an outer shell will move in to fill the vacancy and thus will emit a secondary X-ray. This process of emitting the secondary X-ray is known as X-ray fluorescence. X-ray photons corresponding to a wavelength just slightly shorter than the critical absorption edge are strongly absorbed. Such X-rays must have sufficient energy to eject an electron from the atom. This energy is slightly greater than that corresponding to electron transfer from one of the outer shells to the inner shell to fill the vacancy. This energy change is equivalent to the energy of the X-ray photon emitted in this fluorescing process. The important result is that the wavelength of the emitted or fluorescent X-ray is slightly longer than the wavelength of the incident or absorbed X-ray photon. For maximum fluorescence yield, the wavelength of the excitation X-ray should be approximately 0.2 to 0.6 Å shorter than that of the fluorescent line. The wavelength of fluorescent energy for each element is exactly equal to the emission line of that element as generated by electron bombardment. The characteristic

X-ray line spectra is the same whether it is initiated by bombardment with electrons or with X-ray photons.

For the heavier elements, the absorption of X-ray photons at wavelengths shorter than the K edge* gives a high probability of emission of the K alpha or K beta photon. X-ray fluorescence in light elements is not nearly so efficient; only about 1 out of every 10 absorbed photons more energetic than the K alpha line will result in the emission of that radiation. As a result, the X-ray techniques are not nearly so useful for light elements as those elements above atomic number 20. X-ray fluorescence may be used for the elemental analysis of gases, liquids, or solids; however, it is most commonly employed for the analysis of solid samples. Although X-ray fluorescence methods are independent of the state of the element in the sample, i.e., it can be free, combined, or in liquid, gas, or solid state, they are subject to interference from absorption effects. A common example to illustrate this is as follows: Small quantities of chlorine can be readily detected and measured in organic samples while detection of this same chlorine in samples containing large amounts of lead is extremely difficult. This is the result of two absorption effects. The presence of large quantities of heavy elements strongly absorb the incident radiation, thus preventing reasonable penetration of the X-rays. Second, the lead absorbs the fluorescent K alpha radiation emitted from lighter elements very strongly. The primary reason

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* The K edge refers to the energies required to dislodge electrons from the innermost electron shell in the atom.

that the detection of light elements is difficult is that their characteristic fluorescence line at long wavelengths is limited primarily by the strong absorption of the radiation by air in the instrument. The range of the instrument to the analysis of light elements can be extended if the X-ray source, sample, and detector are placed in a vacuum or helium atmosphere. It is interesting to note that, in contrast to the absorption technique, the fluorescent technique provides essentially a surface analysis. With the heavy elements, the penetration may extend up to 1/8 to 1/4 of an inch. This allows analysis of solid samples without the necessity of chemical treatment, dissolution and other chemical techniques that would be extremely difficult to perform in a zero-g environment.

24.1.2 Radioisotope X-ray Spectrometry

The basic system for a radioisotopic X-ray spectrometer is shown in Figure 24-2. From this diagram, it can be seen that the radioisotopic source, the detector and the electronics are key components of this system. These individual areas will be discussed in some detail.

24.1.2.1 Excitation Sources

There are a wide number of radioisotope X-ray fluorescence sources. As a result, there is a need for careful choice of source for the particular analyses to be performed. Table 24-1 lists some of the commonly used low-energy X-ray and gamma ray sources. The source activities range from about 1 mCi to 1 Ci. In general, the X-ray flux from these sources is on the order of 10^7 photons/sec, and the cost varies with the isotope and its activity. Typically, the source cost is in the range of $50 to $300 per mCi. In actual practice, these

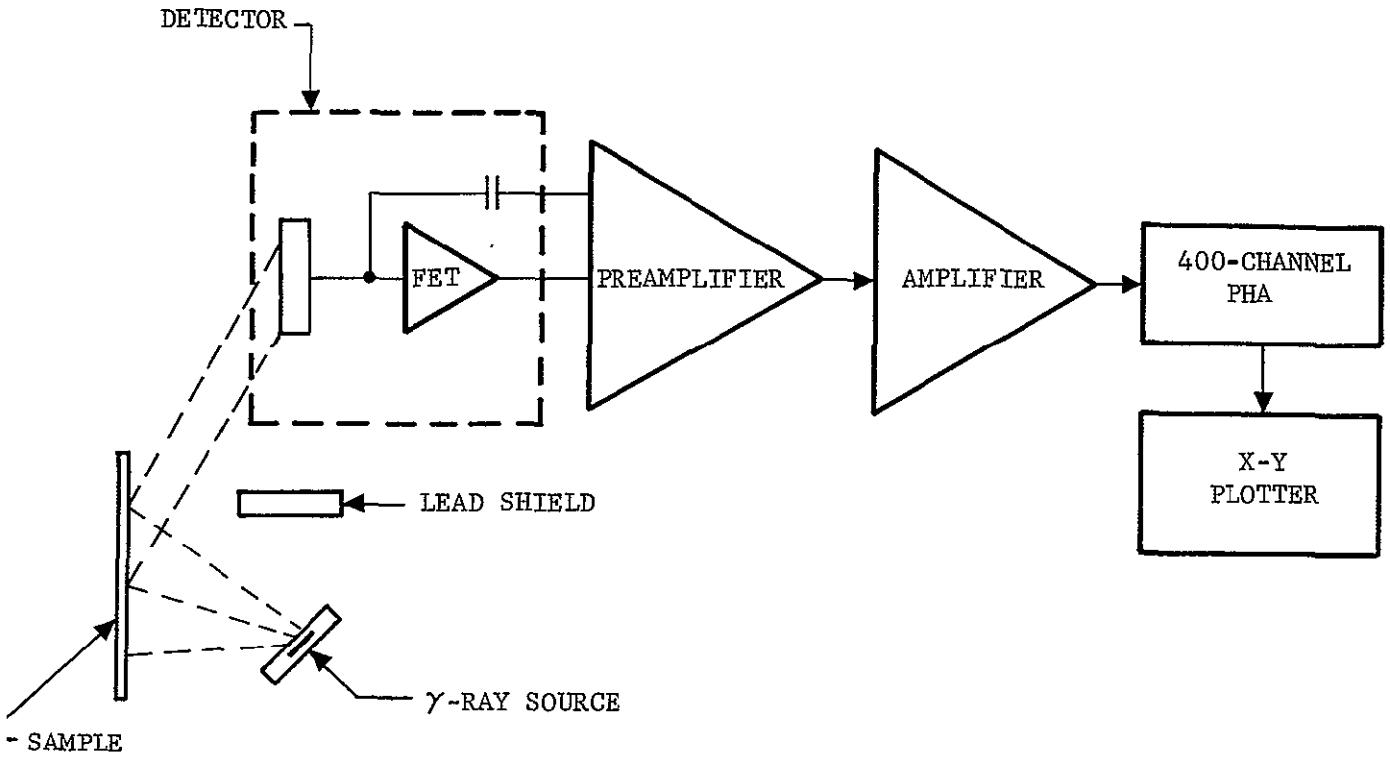

Figure 24-2. Schematic of Energy Dispersion Analyzer Using Lithium-Drifted Silicon or Germanium Detector

sources are very small in size and their relatively low output (10^7 photons/sec) are an advantage since only very nominal shielding of the source is required. In fact, adequate shielding is usually afforded by the normal mechanical structure around the measuring head. As a result, these sources pose little safety hazard. Certainly the major consideration in their usage in a space cabin would be to insure that the sources are not broken and spread throughout the cabin. This can be controlled readily by the proper mechanical mounting of the system. It is also desirable in case of breakage to eliminate alpha emitters from the sources used, to prevent the usual alpha emission health hazards. The sources have been selected by using a criteria of a

Source	Half-life	Useful Radiations	Practical emission efficiency, (photons/ disintegration)	Typical Activity	Highest Atomic Number usefully excited, K X-rays
Iron-55	2.7 yr	manganese K X-rays, 5.9 keV	0.15	2 mCi	24
Tritium-zirconium	12.3 yr	bremsstrahlung, 2 to 12 keV zirconium L X-rays, 2 keV	4×10^{-5} 10^{-5} to 10^{-4}	units of 1 to 3 cI	30
Cadmium-109	1.3 yr	silver K X-rays, 22 keV γ-ray, 88 keV	0.8 0.04	1 mCi	43
Promethium-147-aluminum	2.6 yr	bremsstrahlung, 10 to 100 keV	2×10^{-3}	0.5 Ci	60
Americium-241	470 yr	γ-ray, 59.6 keV γ-ray, 26 keV neptunium L X-rays, 11 to 22 keV	0.35 0.02 0 to 0.2	1 mCi	69
Gadolinium-153	236 days	γ-ray, 103 keV γ-ray, 97 keV europium K X-rays, 42 keV	0.2 0.2	1 mCi	88
Cobalt-57	270 days	γ-ray, 136 keV γ-ray, 122 keV γ-ray, 14 keV iron K X-rays, 6.4 keV	0.10 0.88 0 to 0.06	0.5 mCi	98

Table 24-1. Commonly Used Low-Energy X-Ray and γ-Ray Sources

long half-life, low cost, and ready availability with high specific activity. The sources emit monochromatic X-rays or gamma rays, a continuous spectrum of X-rays or alpha or beta particles. The sources as noted in Table 24-1 are usually available as disc-shaped pellets with overall dimensions from about 0.5 to 1.5 cm diameter. They are typically 2 to 5 mm thick. The back and sides of these sources are shielded so that emission takes place from one face only. Stainless steel windows are used on sources emitting energies greater than about 30 keV, and aluminum or beryllium is used when the required output is of lower energy. In some cases, such as iron-55, a thin, plastic window protects the electroplated iron layer.

Alpha sources such as Po^{210} and Cm^{242} are used to excite very low-energy X-rays. The principle advantage of an alpha source is the high peak-to-background ratio since the alpha-produced Bremsstrahlung radiation is reduced by a factor of the square of the mass of the electron to that of the alpha particle. Alpha sources are a potential health hazard as mentioned above, and must be handled with greater care than beta or gamma sources.

Beta emitters such as Pm^{147} are used to generate Bremsstrahlung and characteristic X-rays that serve as the source of excitation for the sample. These sources are normally prepared by depositing a thin layer of the beta emitter on a suitable target material or by mechanically mixing the isotope and the target material.

Sources such as Fe^{55} which decay by K-electron capture are essentially monoenergetic. That is, iron-55 emits manganese K X-rays. These sources are used to excite specific X-ray lines.

24.1.2.2 Source-Sample-Detector Assemblies

Optimum excitation of the sample can be achieved by using a spectrally pure characteristic X-ray from a secondary target excited by a primary source. The target material is chosen to have its characteristic energy just above that of the absorption edge of the element to be determined. This provides high analytical sensitivity. Using this type of a system, the targets can be changed easily and can be made of mixtures emitting more than one X-ray energy, thus giving a very flexible arrangement. Figure 24-3 shows several geometrical arrangements that have been routinely used for X-ray fluorescence analysis. All three of these geometries have been used in commercially available radioisotope X-ray analyzers. The important parameters are the sample-source-detector distance and the relative sizes of the three components. If X-ray filters are used, they are usually placed between the source and the detector window. With the central source geometry, overall efficiency is typically 10^{-2} to 10^{-4} for pure elements, and counting rates from 10^{3} to 10^{5} counts per second are routinely obtained. This relatively low output from the isotopic source makes shielding a minor problem since radiation propagation is very low. Adequate shielding is normally provided by a shutter and also by the sample being analyzed. At a distance of 1 foot from an unshielded source, the radiation is typically only 1 mr per hour.

24.1.2.3 Detectors

Also a major component of the X-ray analyzer is the detector. As mentioned earlier, numerous detectors have been routinely applied and used for X-ray analysis. In very recent years, solid-state technology has researched,

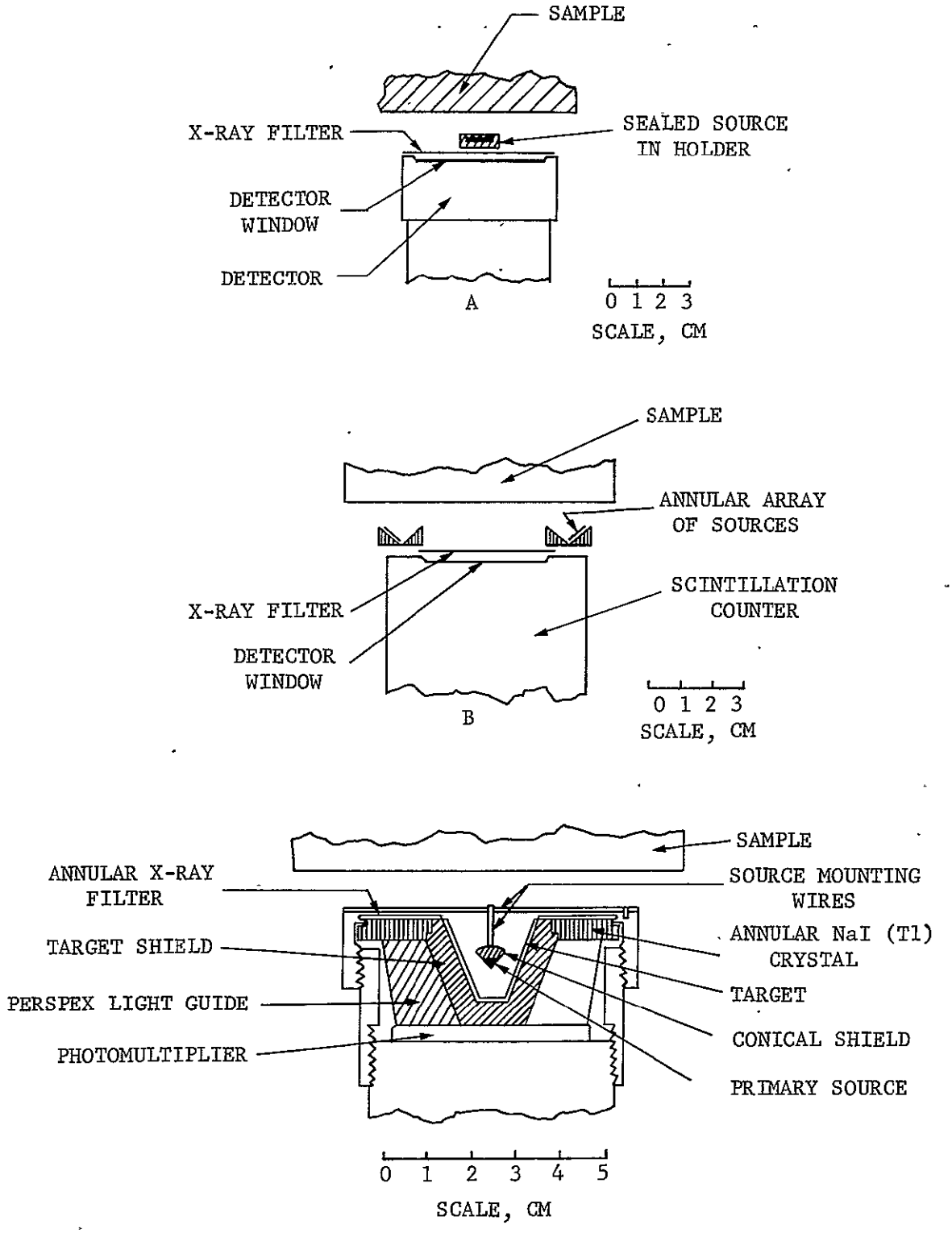

Figure 24-3. Source-Target Geometry

developed and placed on the commercial market lithium drifted silicon and germanium detectors. These solid-state detectors are now being routinely used in the X-ray region, i.e., less than 100 keV. For silver K alpha (22 keV) and gold K alpha (68 keV), the lithium drifted silicon detector has pulse width 2 to 3 times sharper than a proportional detector and 6 to 7 times sharper than the scintillation detector. The proportional detector and the scintillation detectors have been the most often used detectors prior to the advent of the solid-state detector. Both advantages and disadvantages occur in using solid-state detectors. The most substantial advantage is that a medium-voltage power supply at low current is required. For proportional and scintillation detectors, a well regulated high-voltage supply is required. The lithium drifted silicon and germanium detectors require relatively low applied voltages, 50 to 500 V dc, but to obtain maximum resolution do require liquid nitrogen or helium cooling. Since liquified gases may be available on board spacecraft for this purpose, this may not be a serious deficiency; however, it will cause some additional systems interfacing to make the coolant readily available. Solid-state detectors are operable at room temperatures, but suffer a substantial loss in resolution. Substantial technical progress has been made in recent years in improving the room temperature operation of these devices. Although the lithium silicon detector provides excellent resolution, only moderate detection efficiency is provided in the high energy X-ray region.

24.1.2.4 Readout or Data Handling Techniques

With a proportional counter, photomultiplier scintillation detector, or solid-state detectors, a preamplifier and amplifier are needed to supply a count

rate meter for total count, or a single-channel-multichannel pulse height analyzer. The output of the pulse height analyzer can be plotted on a normal X-Y plotter or recorder, if desired. The most versatile system involves using a multichannel pulse height analyzer.

Numerous companies manufacture multichannel pulse height analysis equipment usable for a nondispersive X-ray system. Much of this equipment was originally developed for pulse height analysis in the nuclear industry. These systems are compact, completely transistorized, have high density core storage capabilities, and are relatively low powered. This type of equipment is much more versatile than the single-channel scanning systems.

24.2 APPLICATIONS

Table 24-2 lists a number of current applications of the nondispersive X-ray spectrometer. The multichannel pulse height analyzer used with the X-ray spectrometer can also be used for other specific radiation counting applications (see Section 19). This type of equipment could serve multipurpose capabilities in equipping a Space Station laboratory. The nondispersive X-ray spectrometer with pulse height analyzer simultaneously depicts the fluorescent X-ray spectrum lines at one time. This type of assay can be performed in real time without altering the sample or specimen in any way. Some 80 elements with a dynamic range of a few parts per million up to 100 percent can be analyzed by this type of instrument. For such a wide analysis capability, the instrument is quite compact, uses ordinary power outlets, and is substantially more rapid in its analysis capabilities than the older X-ray spectrometers. Its resolution is not as good as the conventional X-ray equipment, and only elements above atomic numbers 12 can be resolved at this time.

Art	Nonharmful analyses of art objects, including: 1. Pigment analysis in oil paintings 2. Clay or metal composition determination in ancient sculpture
Chemistry	Nondestructive analyses of solids or liquids, including: 1. Quantitative analysis of elements to less than 10 ppm. 2. Adaptation to electron beam microprobes and electron scanning microscopes 3. Rapid qualitative analysis
Field Studies	Investigations of chemical compositions with portable field instruments, including: 1. Archeological studies 2. Meteorite identification and analysis 3. Ocean sediment studies 4. Identification and quantitative determination of ore enrichments
Industry	Quality control and testing, including: 1. Composition determination of steels, alloys, and impurities 2. Quality control in the manufacture of film, paper, glass, cloth, metals, and machine components 3. Engine wear determination (metal content in oil)
Medicine	Nonsurgical and quantitative analyses, including: 1. Plutonium studies of wounds 2. Iodine uptake measurement in thyroid-nonradioactive tagging 3. Trace element determination in blood
Physics	Applications requiring high resolution and high sensitivity in the spectroscopy of low energy charged particles and electromagnetic quanta, including: 1. Atomic and fluorescent X-ray investigations 2. Nuclear-energy level investigations 3. Beta-ray studies 4. Mossbauer effect studies 5. Intrinsic semiconductor property determination
Security	Many possibilities, including: 1. Identification and tracing of currency, passports, art, security documents, jewelry, and credit cards

Table 24-2. General Applications of the Nondispersive X-ray Spectrometer

X-ray spectrometers are applicable to the following functional program elements (FPE's):

5.10	Plant Specimens (Bio E)
5.17	Contamination Measurements
5.18	Exposure Experiments
5.26	Invertebrates (Bio F)
5.27	Physics and Chemistry Lab
	General Purpose Laboratory

24.3 LOGISTICS

24.3.1 Packing and Launch

The radioactive source X-ray spectrometer can be conveniently divided into two separate sections. One of these is the sample holder, source, and detector section. The remaining package is the amplifier, multichannel pulse height analyzer, and readout package. The latter package can be bulkhead-mounted and is capable of withstanding typical launch and vibration requirements. The source, detector, and sample-head assembly should be packaged so that no possibility of damage to the radioactive source or the detector can occur. None of these components should be overly sensitive to shock and vibration.

24.3.2 Installation

Using the radioactive source spectrometer, the only requirements for installation are a power plug-in and, in the case of a cooled solid-state detector, a source of liquid nitrogen or helium coolant. Obviously, some plumbing or filling method must be interfaced with the vehicle itself. In the event a room temperature solid-state detector is used, this interface for coolant would not be required. The analytical resolution of the instrument for all elements is impaired without an available coolant.

24.3.3 Consumable Supplies

The only consumable supplies required for the X-ray spectrometer are necessary matrix mixing powders and chemicals for sample preparation, if required. Additionally, small capsules for holding metal samples, liquids, or powders would be required. These are small plastic capsules and they are typically inexpensive and expendable. The coolant required for the solid-state detector could also be considered a consumable. Only one or two leters of liquid nitrogen per day are required.

24.3.4 Accessories and Spare Parts

The following accessories may be desirable and necessary for operation of the X-ray system. The first of these are density filters, usable for selecting and enhancing the particular element of interest on the X-ray spectrometer. In addition, there may be a need to use two or three types of radioactive sources to cover the entire range of elemental interest. These would typically be combined with the target assembly. None of these accessories requires large amounts of space. The solid-state electronics system utilized in this type of system is normally quite rugged and reliable. It is not considered necessary nor desirable to maintain spare parts stocks for repair of the electronics system. This would normally be handled on a resupply basis.

24.3.5 Repair and Maintenance

Because of the relative complexity of a solid-state pulse height analyzer, on-board repair and maintenance is considered undesirable. The component of greatest concern is the solid-state detector which can fail if not specifically designed for both room temperature and cooled operation. Under circumstances

where the unit might fail after long-term exposure to room temperature, it would be considered helpful to maintain an on-board spare.

24.4 OPERATION

24.4.1 Warm-up and Speed-of-Operation

Approximately 15 minutes is required to place the instrument into operation. After this time, multiple analyses could be obtained in a very short period of time. During operation, the unit can be operated continuously since only the amplifier/pulse height analyzer amplifier system draws current. Although some X-ray spectrometers are battery-operated, line operation of the multichannel pulse height analyzer is not recommended since its power consumption is nominally higher than that desired for battery systems of reasonable operating life. Analysis duration time is often determined by the sensitivity desired in the analysis. For high-sensitivity measurements, 5 to 10-minute counting times may be necessary. For analyses where percentage amounts of elements are present, this analysis can often be made in a matter of seconds.

24.4.2 Operation Skills

Space Station operation of the radioactive source X-ray spectrometer is possible for personnel of medium-skill levels. It is preferable if the individual operating this equipment has had prior experience with this type of equipment. For special analyses, a reasonable knowledge of matrix effects and techniques for avoiding analysis errors are important. With the exception of sample preparation, the remaining instrument operation and calibration procedures are relatively simple. Preflight training for this equipment is considered necessary.

24.4.3 Operating Procedure

The typical operating procedure for a radioactive source X-ray spectrometer would be as follows:

Preparation:	Check liquid nitrogen coolant supply for detector Turn on. Allow a 15-minute warm-up
Calibration:	Introduce calibration samples and count for preset time to check calibration curve
Measurement:	After removal of calibration samples, insert unknown sample to be analyzed and count for preset period of time Make analysis computation by comparing with calibration curve

24.4.4 Sample Preparation and Handling

The end results obtained from X-ray spectrometry are in many cases directly dependent upon the skill and technique used for sample preparation. Where liquids are analyzed, this problem is minimal. However, the area of greatest potential interest that of powder and solids analysis, the problem of matrix effects and particle size are important. In these areas, the skill of the operator is very important in that he must recognize the limitations and the problems in the sampling process. Solid samples such as metals can be placed directly into the analyzer providing they cover a sufficient surface area to give good beam geometry. Powder samples can be contained by a small plastic film in plastic cups such that they can then be inserted into the analyzer. Here again, liquid samples can be handled with the same arrangement as are the powders. Gas sampling would only be used as a very special technique for X-ray spectrometry since most of the lighter inorganic gases cannot be readily analyzed by this technique. As a result, if gas analysis of heavier gases is required by X-ray methods, a gas flow cell would be used.

24.5 INTERFACE

24.5.1 Interface with Other Laboratory Instruments

The X-ray spectrometer is a completely separate and independent instrument, and does not require any special interface with other instruments. The pulse height analyzer portion of the spectrometer can be used for other radiation monitoring work, if necessary. The output of the pulse height analyzer can be routinely placed into the on-board data management system or it can be read into a standard X-Y recorder, if available.

24.5.2 Interface with Vehicle System

There are two vehicle interfaces for the X-ray analyzer. One of these is for approximately 100 to 150 watts of power which, in most of the commercial instruments, is 115 V, 60-Hz power. In addition, connection must be made for a liquid nitrogen or helium supply for the solid-state detector cooling. These requirements are minimal since a typical 10-liter dewar with an enclosed solid-state detector might require approximately 1 fill every 5 days. This means that only a very small connection line would be required and only occasional filling needed. It would be possible, but not necessarily desirable, to replace most of the functions of the pulse height analyzer with the data management system.

24.6 SAFETY

24.6.1 Flame Hazards

The nondispersive X-ray spectrometer presents no flame hazard. The only area of concern is in the medium-voltage dc, low current that may be supplied to

the detector. The remaining electronics are typically low voltage solid-state equipment which is considered safe for space applications.

24.6.2 Microbiological Hazards

The analyzer does not present any microbiological hazards to personnel.

24.6.3 Electromagnetic Interference

The analyzer will normally require 115 V, 60-Hz power. Under these circumstances, it will not require a special converter. As a result, only minimal problems could occur in meeting EMI requirements. Elimination of an X-ray tube in the nondispersive method not only avoids the very high power requirements of an X-ray tube but also an attendant and substantial generation of EMI.

24.6.4 Ionizing Radiation

Ionizing radiation is neither produced by nor does it interfere with the operation of the X-ray spectrometer.

24.6.5 Physical Hazards to Personnel

The radioactive sources used for nondispersive X-ray spectrometers are relatively small from a radiation dosage standpoint. Typically, the radiation hazard is small, even with completely unshielded sources. A typical localized dose rate at 1 foot is 1 mr per hour. This does rate should be very acceptable. Of more concern is the breakage or fracturing of the source assembly such that radioactive material might actually be spilled into the cabin atmosphere. In normal source construction procedures, this should be a minimal problem since the source is mounted in a rigid metal holder and would not be amenable to external damage.

24.7 MODIFICATION

Electronic modification of commercial equipment is not considered economically justified. The only minor modifications recommended would be replacement of unapproved nonmetallic materials with approved materials, as necessary. This should be a very minimal effort.

24.8 AVAILABLE INSTRUMENTS

A list of commercially available nondispersive X-ray spectrometers and their manufacturers is listed in Table 24-3.

Manufacturer	Model	Price ($)	Detector	Electronics	Power	Weight	Size		Remarks
							Electronics	Detector	
Nuclear-Chicago	9200		Scintillation	Single channel pulse height analyzer, scaler, timer, display and HV supply	Rechargeable battery or 110 V ac; battery life 50 hours	15 lb	12"x6"x8"	9"x2-1/2" D	Ruggedized for field work
Columbia Scientific Industries	700	4000	Scintillation or proportional	Single channel pulse height analyzer, scaler, timer, display, dc-to-dc converter and HV supply	Rechargeable battery or 110 V ac; battery life 50 hours	13 lb	12"x8"x10"	1"x2-1/2"D	
Pinametrics	4000	4000	Scintillation or proportional	Single channel pulse height analyzer, scaler, timer, display and HV supply	Rechargeable battery or 110 V ac, battery life 50 hours	18-1/2 lb	15-1/2x8-1/2x6-1/2	8-1/2x4 D	
Jarrell-Ash	PMA 80-702		Proportional	Count rate meter, high voltage supply, sample and hold circuit, timer	110 V ac		8"x8"x5"	10"x10"x4"	Analyzes any six of thirty-five elements
Hilger & Watts	Lab Model		Scintillation	Count rate meter, high voltage supply and amplifier	110 V ac or battery pack	23-1/2 lb	12-1/2x15x8-1/2	8"x3-1/2"D	
Princeton Gamma-Tech Inc		10-15,000	Silicon (Li) & Cryostat	Linear amplifier, cooled FET preamplifier, 512-channel analyzer, bias voltage, & CRT display	110 V ac	50 lb			Many electronic & cryostat variations available
Nuclear Equipment Corp		10-15,000	Si or Ge (Li) & Cryostat	Linear amplifier, bias voltage, 400-channel analyzer & CRT display	110 V ac 100 W	85 lb			
Ortec, Inc		10-15,000	Si or Ge (Li) & Cryostat	Bias supply, preamplifier, amplifier, baseline restorer & multichannel analyzer	110 V ac				Many electronics & cryostat variations available
Nuclear Diodes, Inc.	DS&CF Series	10,000							
Nuclear Equipment Corp	NX-31	8,000							

Table 24-3. X-Ray Spectrometer

Section 25
ZERO-GRAVITY SAMPLE HANDLING

The basic problems of sample handling in a zero-g laboratory have been discussed in Volume 1 (paragraph 3.4.2). These problems include the transfer of liquids, the containment of liquids and particulates, and the avoidance of unwanted bubbles in liquid samples. These, and indeed most, sample-handling problems are those of wet chemistry applications. Gas-handling techniques are essentially the same for an earth-based or the Space Station laboratory.

25.1 General Sample-Handling Techniques

Many, perhaps most, zero-g sample-handling problems can be solved by use of some combination of flexible tubing, syringes, needles, resealable diaphragms, valves, and collapsible bags.

25.1.1 Tubing-Syringe-Valve Sample-Handling Systems

A sample-handling system of flexible tubing, syringes, needles, and valves is used in earth-based laboratories for administering drugs and collecting samples from venous canulas in experimental animals. In clinical medicine, similar techniques are used for procedures involving extracorporeal circulation (for example, heart-lung machines and dialysis devices). A tubing-syringe-valve system can be completely modular, highly diverse, and available from commercial sources. Syringes can be used to move fluids from one part of the system to another, and several types of tubing pumps are available which can move

solutions through the system without intervening in the system. Several types of electrochemical sensors can be incorporated in a tubing-syringe-valve sample-handling system. (See Section 3 and Section 22, for example.) Tubing-syringe-valve systems are ideally adaptable for introducing samples into zonal or continuous-flow centrifuge rotors (see Section 5), or continuous-flow sample tubes for liquid scintillation counters (Section 19). With hypodermic needles and resealable membranes or plugs (silicone rubber, for example) samples can be withdrawn or introduced into an otherwise closed system of tubing, syringes, and valves. In one form or another, tubing-syringe-valve sample-handling systems are almost certain to be used in the Space Station laboratories.

25.1.2 Collapsible Bags

Collapsible bags are used in containers dispensing photographic chemicals and in some types of baby bottles. The unique feature of collapsible bags is their ability to accept or dispense a volume of liquid without exchanging an equal volume of air. This feature makes them ideal for containing, receiving, dispensing, mixing, or transporting liquids in the Space Station laboratory.

Collapsible bags are easily made from a variety of types of plastic, and can be easily fitted with resealable plugs or with valves and connectors necessary to interface with a tubing-syringe-valve sample-handling system. A variety of sizes and shapes are possible to adapt these bags to many highly specific applications.

A collapsible bag designed by Beckman for use as a sample cell for colorimetric determinations is shown in Figure 25-1. In this particular example, a reagent

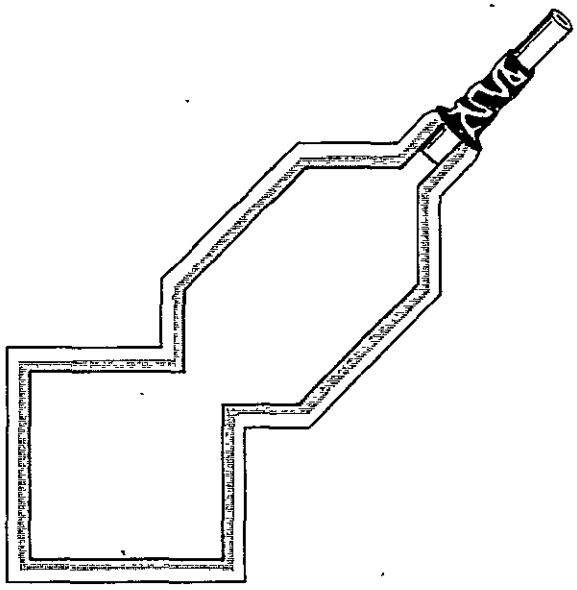

Figure 25-1. Sample Cell Collapsible Bag

(solid tablet) is prepackaged in the bag. A sample, usually diluted, is injected through the resealable silicone rubber plug in the neck. Mixing is achieved by manually pressing the fluid between the two compartments. A simple flick of the wrist while holding the neck of the bag creates enough centrifugal force to drive the liquid sample to the square end, leaving bubbles and excess sample in the other compartment. The bubble separation achieved by this action is sufficient to allow optical analysis of the sample in a colorimeter. The square end of the bag fits into the sample cell of a space-qualified colorimeter. This is only a single example of a specific application of a collapsible bag to zero-g sample-handling problems.

Although most sizes and shapes of collapsible bags which will be needed can be anticipated in advance, it would be possible to fabricate a limited number of bags of unanticipated size or shape during a Space Station mission. The shape of the bags is determined by the sealing pattern (Figure 25-1). Sealing patterns can be made from standard printed circuit boards etched to leave a strip of copper in the pattern desired. Passing current through the copper strip heats it and seals the plastic sheets against which it is pressed. The supplies needed to prepare and use this type of bag-making device could be provided in the laboratory to solve special sample-handling problems.

25.1.3 Centrifugation for Zero-g Sample Handling

The use of a flick of the wrist to produce a momentary centrifugal force for separation of bubbles from a liquid sample was mentioned in paragraph 25.1.2. This manual technique should be rather widely applicable in a zero-g laboratory, and could, perhaps, be extended slightly by use of short tethers to spin sample containers manually. There are obvious limitations to manual spinning procedures. A low-speed centrifuge could be adapted to accommodate sample-handling devices. This would be useful, perhaps indispensible, for separating precipitates from liquids, liquids from gases, or for establishing interfaces between immiscible liquids. Several of the specific sample-handling devices discussed in paragraph 25.2 include provisions for spinning the device in a centrifuge. High speed and ultracentrifugation is incompatible with the mechanical properties of most sample-handling devices.

25.1.4 Automated Diluting Device

Automated diluting devices are commercially available. These allow mixing of liquid samples and reagents in predetermined, yet adjustable, proportions.

With little or no modification, these devices can be used to accept samples and reagents from closed containers and deliver the mixture to closed containers. Two-pump (diluent and sample) and three-pump (diluent, reagent, and sample) models are available. Diagrams of the two types are shown in Figures 25-2 and 25-3.

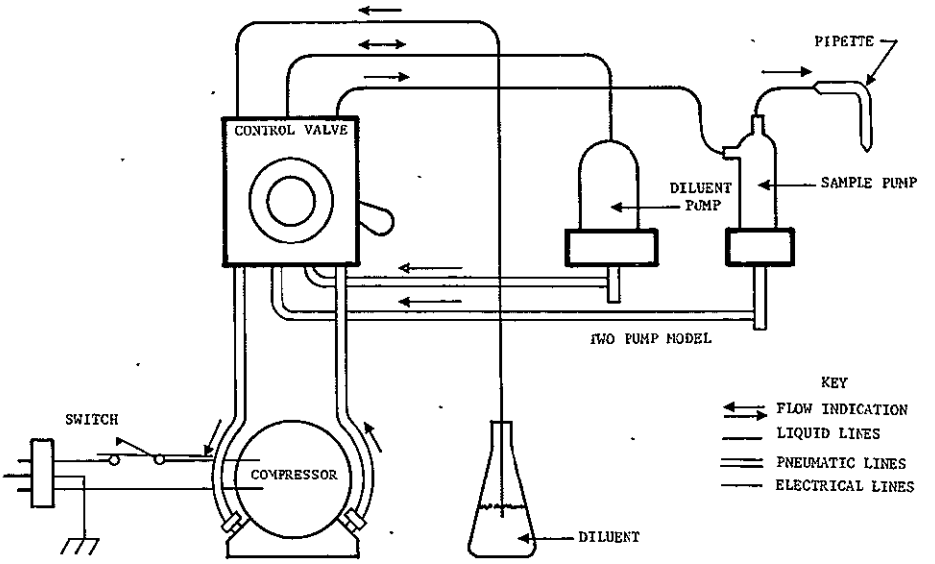

Figure 25-2. Auto-Dilutor--Two-Pump Model

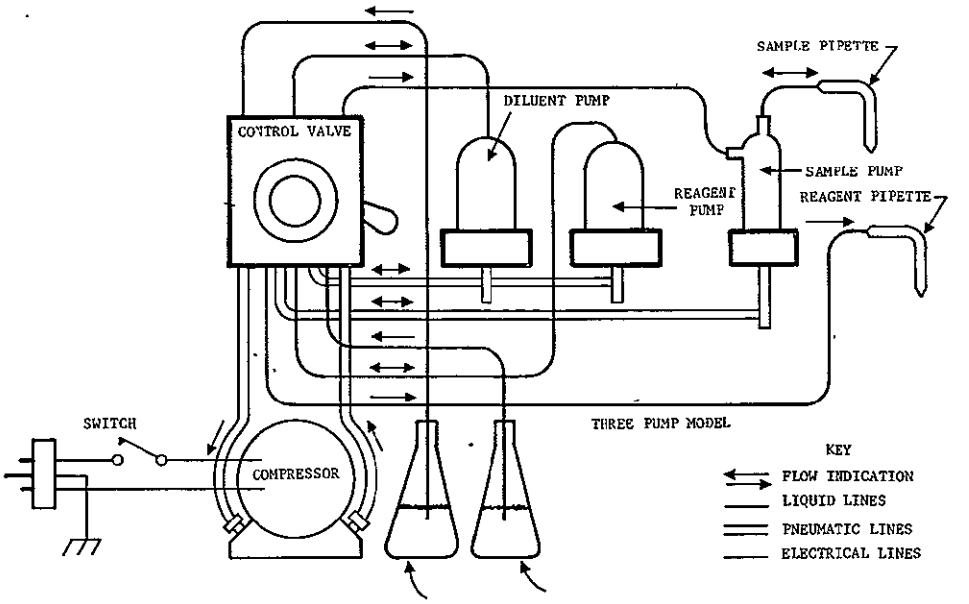

Figure 25-3. Auto-Dilutor--Three-Pump Model

Diluting devices are manufactured by the American Optical Corp. (Auto-Dilutor) and Research Specialties, Inc. (Dilumat). Commercially available syringe pumps and tubing pumps will perform similar diluting and mixing functions.

25.2 Specific Sample-Handling Devices

The following section considers several design concepts for individual sample-handling devices. Some of these devices can be assembled from standard, commercially available parts.

25.2.1 Specific Ion Electrode Syringe (Figure 25-4)

The pH electrode syringe has a standard barrel with the specific ion electrode, the reference electrode, and the temperature-compensator incorporated into the plunger. A sonic mixer, attached to the cable, is clamped to the exterior wall

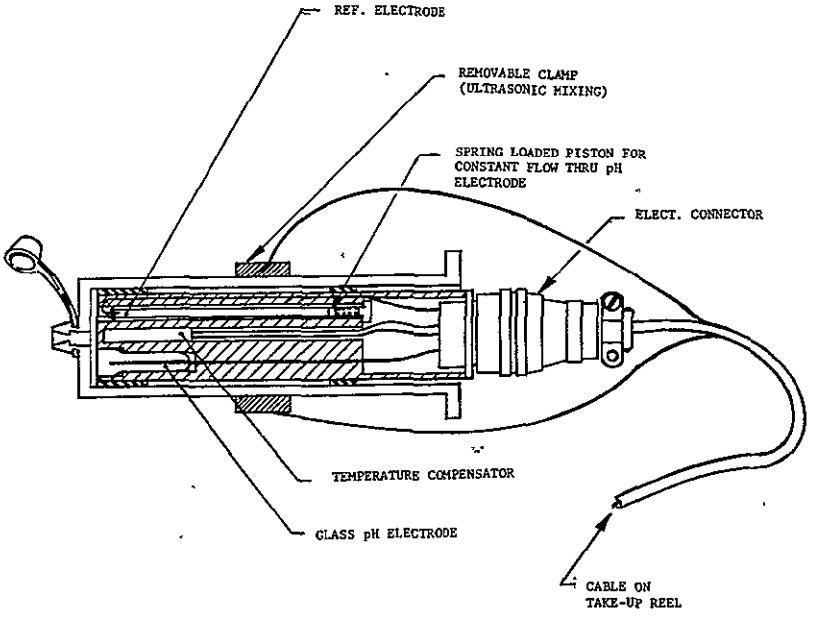

Figure 25-4. Specific Ion Electrode Syringes

of the syringe barrel. The electrodes and temperature-compensator are incorporated into the syringe plunger, insuring contact between the electrodes and the sample regardless of the sample volume. The mixer in the syringe permits simultaneous mixing of reagents.

25.2.2 Type B Micro Sampler (Figure 25-5)

The Type B micro sampler was designed because of the necessity for transferring micro volumes accurately. Use of the micro sampler removes the responsibility of measuring minute samples in a syringe.

The Type B liquid micro sampler is a 4-port rotary chromatographic type with a sample input port (female syringe connector), a water input port (male syringe connector), a sample output port (female syringe connector), and a waste output port (male syringe connector). In operation, a syringe containing a liquid is

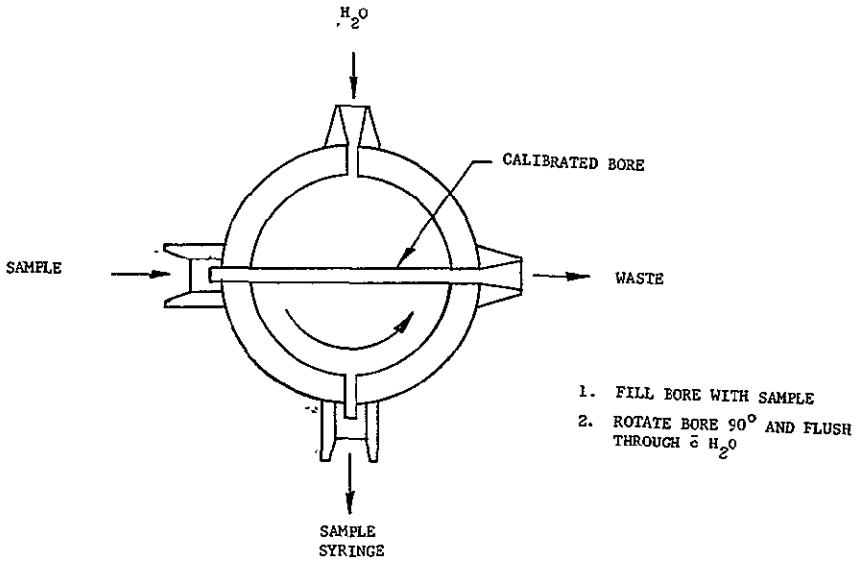

Figure 25-5. Type B Micro Sampler

connected to the sample input port, and the rotating center is rotated to line up with the sample input and waste output. The sample is injected with the overflow going to waste. The rotary center is then rotated 90 degrees to line up with the water input and the sample output. A syringe is connected to the sample output port, and the sample is flushed into it for analysis.

25.2.3 Capillary Blood Sample Collector

A capillary blood sample collector might include a lancet on one end and a female syringe connector on the other. The bore and the length are calibrated to permit sampling of a very small known volume of whole blood from a pricked fingertip. The micro sampler can then be transferred to a syringe and the sample drawn in for analysis. After use, the micro sampler should be discarded into the solid waste container.

25.2.4 Adsorption Column (Figure 25-6)

An adsorption column can be used to achieve chromatographic fractionation, of various compounds in urine, in a zero-g environment.

The column is packed with either a granular or powdered solid adsorbent material. Both ends of the column are equipped with female syringe fittings, to accept syringes, and a detachable coupling enabling insertion of the column into the waste and/or reagent ports. Check valves are incorporated at each end of the column to allow only one-way flow, and glass frits are used at each end to keep the packing from fouling the inlet and outlet ports.

In operation, a sample is transferred with hydrostatic pressure applied from one syringe through the column to another syringe. During the operation,

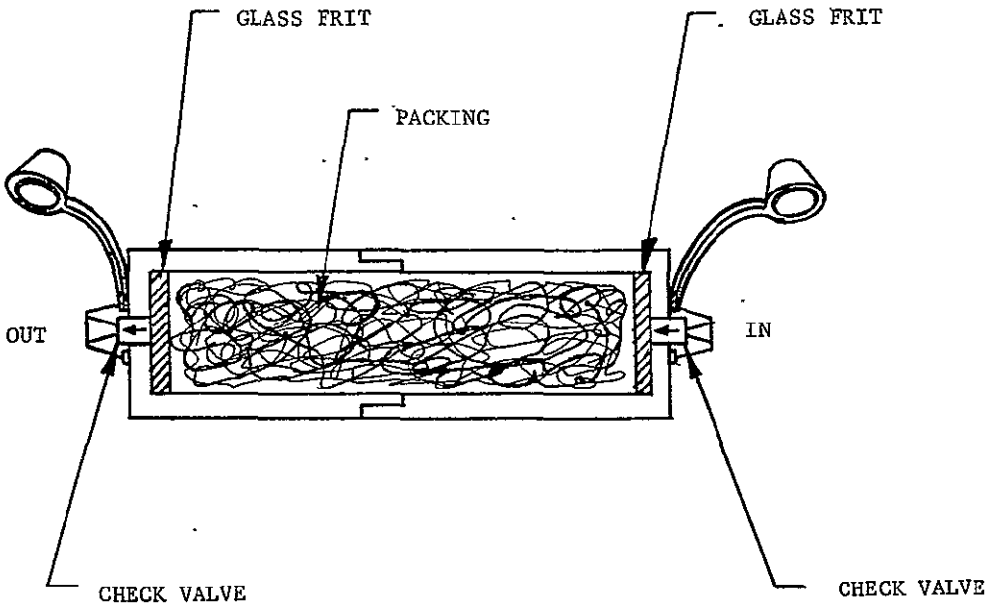

Figure 25-6. Adsorption Column

specific compounds are adsorbed from the sample as it passes through the adsorbent material. After elution, with appropriate solvent, the column should be returned to the individual case from which it originated and will not be used again.

25.2.5 Mixing Syringe

The syringe like the one shown in Figure 25-7 can be used to maintain the separation of two liquids until mixing is required. The mixing syringe contains two reagent chambers separated by the piston or plunger. The two reagent chambers are interconnected by one-way check valves leading from the rear reagent chamber to the front of the piston. A check valve in the needle hub prevents the introduction of air into the front chamber when the plunger is being pulled to the rear, accomplishing the mixing of the two liquids.

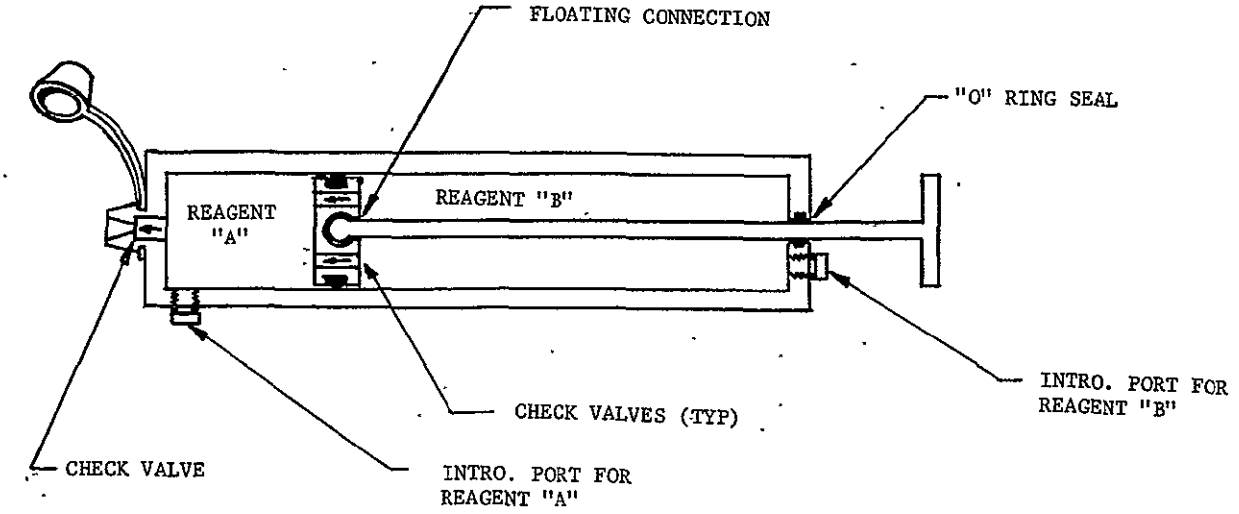

Figure 25-7. Mixing Syringe

25.2.6 Zero Dead-Space Syringe

The zero dead-space syringe is a standard type syringe with a standard barrel and piston (Figure 25-8). The piston is modified to displace the dead volume of the needle hub. As shown, the plunger is threaded to permit micro-manipulations and provides a position-lock for centrifugation.

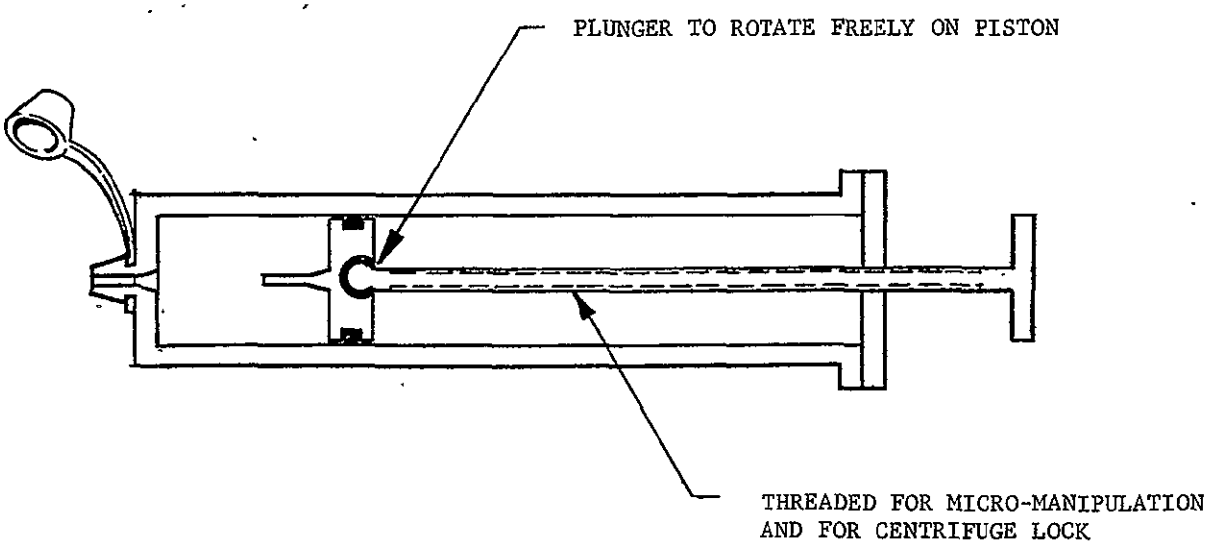

Figure 25-8. Zero Dead-Space Syringe

25.2.7 Precipitate Retention Syringe

The precipitate retention syringe, as shown in Figure 25-9, permits retention of a precipitate while discharging the supernatant liquid. The precipitate retention syringe is identical to the zero dead-space syringe, with the exception of the concave piston head which can be designed to retain the precipitate as the liquid is being discharged.

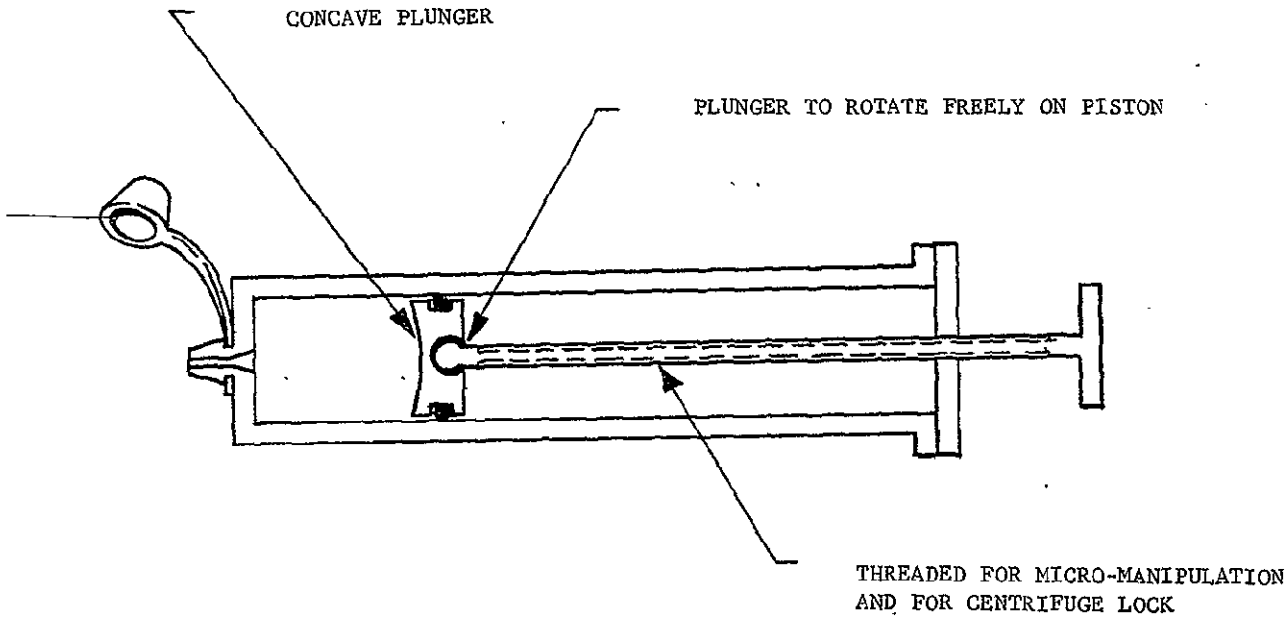

Figure 25-9. Precipitate Retention Syringe

25.2.8 Filter Syringe

The Filter syringe (Figure 25-10) can filter unwanted particulate matter from a liquid sample as the sample is discharged from the syringe. The syringe is equipped with a side-sample induction port, with a check valve that allows flow into the syringe only. The head of the syringe contains a filtering wafer composed of a filter paper pad sandwiched between two glass frits. The head of the piston is flat to match the surface of the filtering wafer.

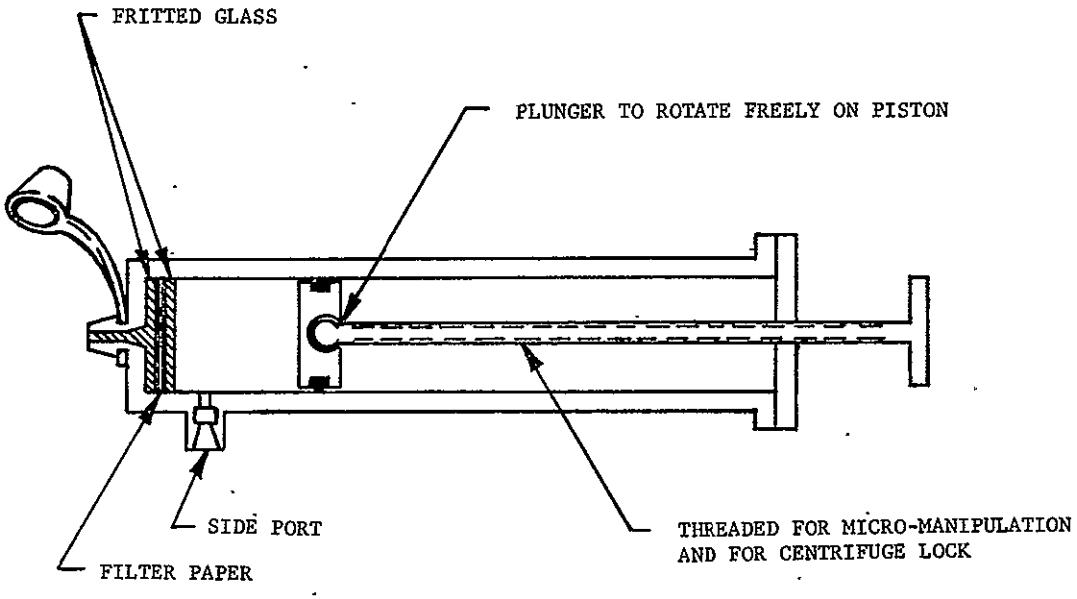

Figure 25-10. Filter Syringe

25.2.9 Nested Syringes

A particularly difficult task in a zero-g environment is to obtain blood plasma without hemolysis and within a closed chamber. This problem was recently solved by Beckman with the nested set of syringes as shown in Figure 25-11.

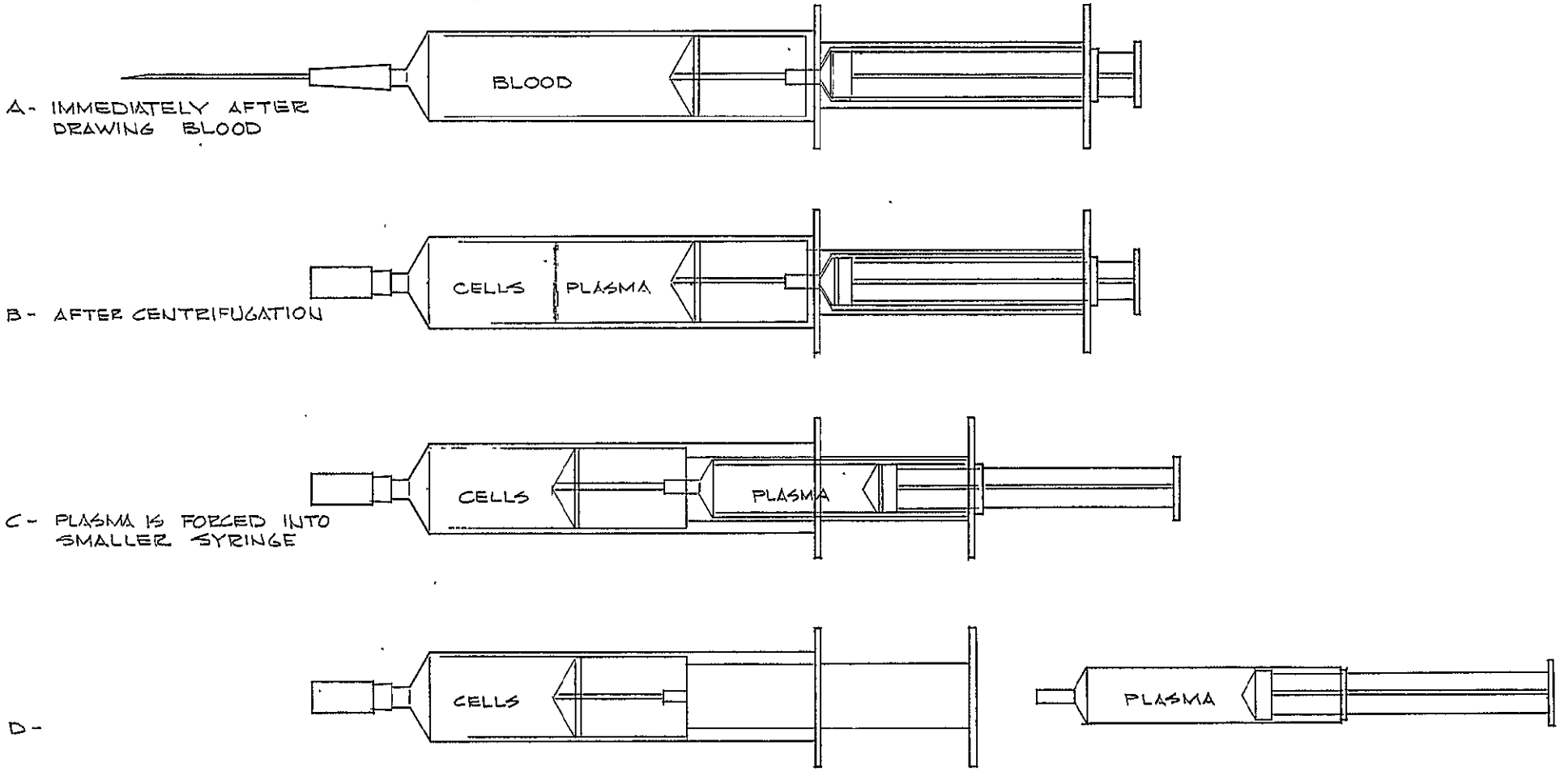

Figure 25-11. Centrifuge Syringe Assembly

A 5-ml syringe is contained within the plunger of a 20-ml syringe. Both plungers are initially in the closed position. Blood is conventionally drawn into the larger syringe. The needle is removed (B) and replaced by a cap. The assembly is then placed in a centrifuge with the capped end facing outward. After centrifugation (B), the blood cells migrate towards the capped end, leaving clear plasma towards the plunger end. The plunger is then moved towards the capped end of the large syringe (C) until the blood cells are reached by the inlet end of the small syringe. The hydraulic pressure created by this action forces the plasma into the smaller syringe and, in turn, forces the smaller syringe plunger out. The smaller syringe can then be removed from the assembly and the large syringe discarded. This technique has the following advantages: minimum amounts of apparatus and manipulations are needed; the blood does not need to be drawn precisely; all transfer is within a sealed assembly; the inner syringe is never contaminated by blood cells; and the inner syringe serves as a handy dispenser. The system is easy to use and provides a good, clear separation.

25.2.10 Reagent Bottles (Figure 25-12)

The reagent bottles vary in size depending upon the amount required for the particular determinations programmed for the mission. Each bottle contains a bladder which is filled with reagent. The bottle and the bladder may be pressurized by either gas or a piston driven by a spring.

25.2.11 Reagent Ports (Figure 25-13)

The reagent ports are female syringe connectors with a check valve that allows only one-way flow through the port to the outside. A syringe, when inserted

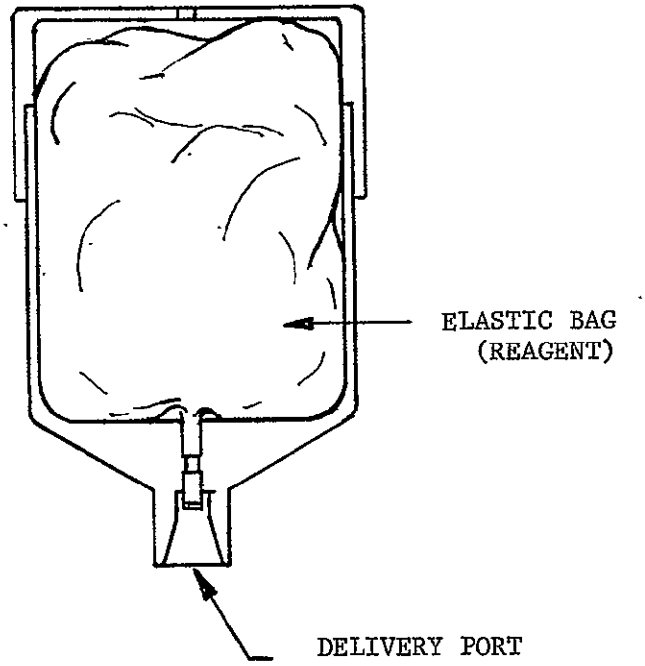

Figure 25-12. Reagent Bottle

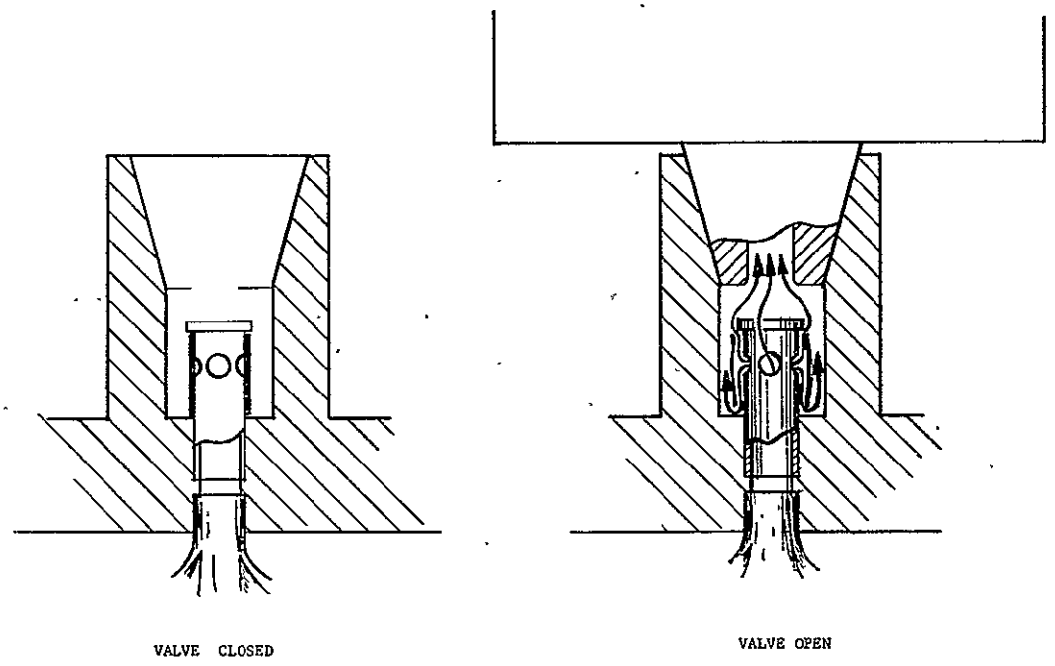

Figure 25-13. Reagent Ports

into the port, activates the valve, allowing the reagent to be dispensed from the reagent bottle through the reagent port.

25.2.12 Gaseous Reagent Addition Device (Figure 25-14)

The addition of very small amounts of reagents and the solubilization of gases into the reaction mixture can be readily accomplished by the gaseous reagent addition device shown in Figure 25-14. This device could add a measured volume of gas into the reaction vessel and perform this quantitatively in a zero-g environment.

The gas is stored in a glass vial surrounded by a Teflon envelope with the tip of the vial protruding into a rotating piston. Rotation of the piston breaks the tip of the vial and the bore opens the passage to the syringe coupling. A check valve prevents backflow into the glass envelope. The rotating piston is then returned to the closed position.

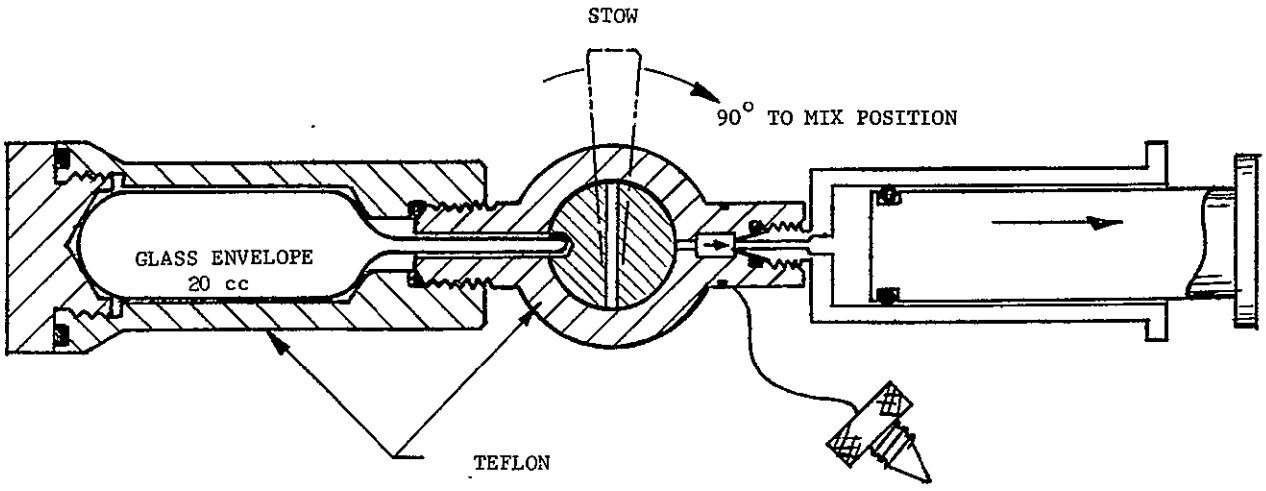

Figure 25-14. Gaseous Reagent Addition Device

25.2.13 Staining Apparatus (Figure 25-15)

A staining apparatus designed for successful staining of blood smears in zero-g environment is shown in Figure 25-15. The staining apparatus consists of a waste receptacle, a slide holder, and a septum with retainer and T-spreader. The waste receptacle with a permanent septum connects to the back of the slide holder by means of an O-ring seal and snap lock. The needle on the back of the slide holder penetrates the septum of the waste receptacle to interconnect the two compartments. A check valve in the slide holder prevents waste from returning into the slide holder.

The septum with the T-spreader is affixed to the front of the slide holder (T portion of the spreader in compartment) by means of the threaded retainer.

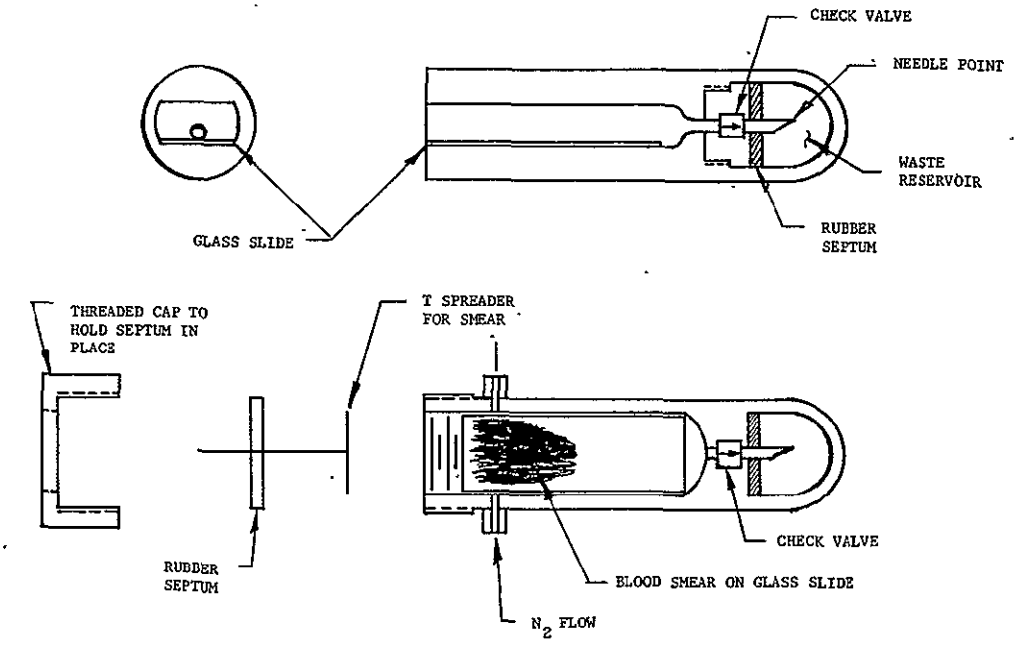

Figure 25-15. Staining Apparatus

Blood and stains and reagents are injected into the glass or plastic slide through the septum, spread by the T-spreader, and transferred into the waste receptacle by centrifugation. The drying operations are carried out by passing dry, inert gas through the slide holder by means of the inlet and outlet ports on the side of the slide holder. At the end of the operation, the waste receptacle is transferred to the solid-waste container and the slide compartment transferred to the storage area to be reused with a new waste receptacle.

25.2.14 Culture Chamber for Microorganisms (Figures 25-16 and 25-17)

Two types of culture chambers are suggested: one for liquid and the other for particulate samples. The two chambers are identical except for the sample induction ports. The liquid sample is transferred into the chamber through a septum at the induction port. The particulate or semi-solid samples are administered with a cotton swab through a sealable port. For purposes of providing a controlled atmosphere to the culture, gas induction and exhaust ports are provided on the sides of the chamber.

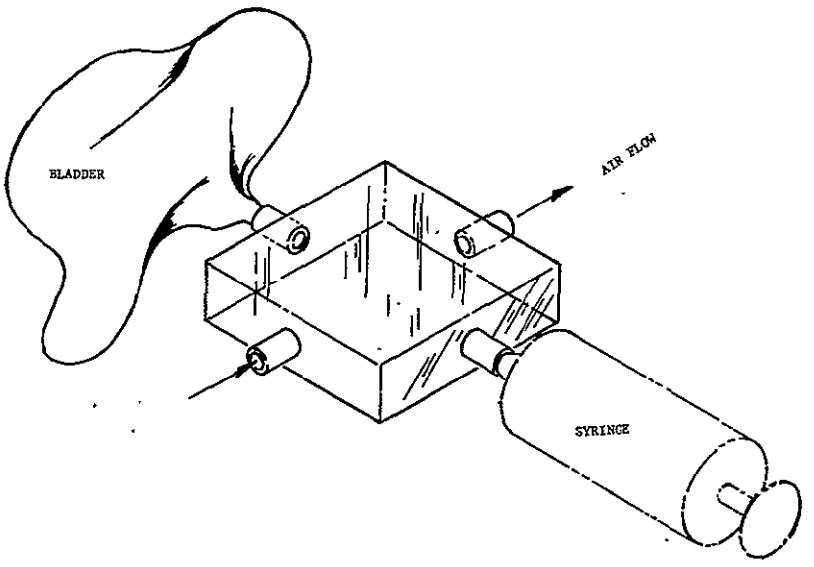

Figure 25-16. Microorganisms Culture Chamber

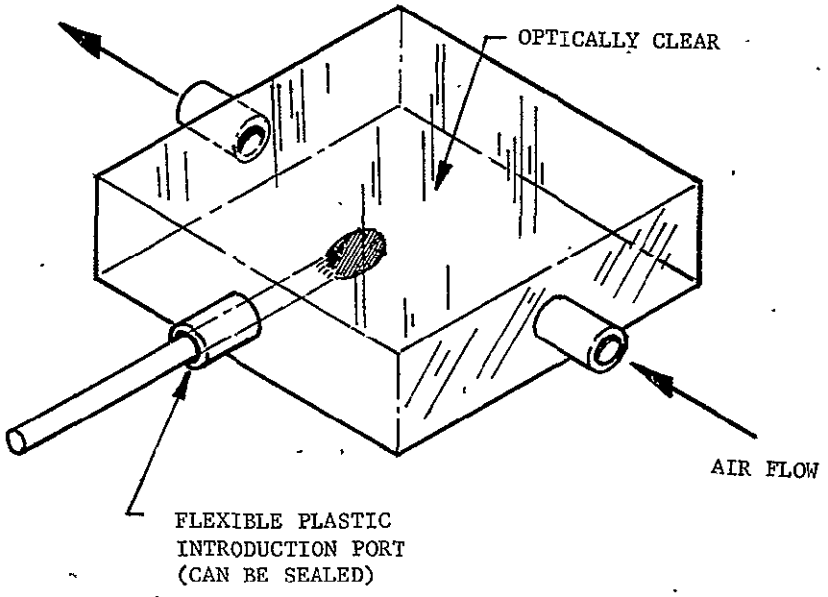

Figure 25-17. Particulate Microorganisms Culture Chamber

25.2.15 Electromagnetic Trap (Figure 25-18)

Leukocyte phagocytic activity is determined when a sample of leukocytes (white blood cells) containing ingested iron particles is passed through an electromagnetic trap. The leukocytes are trapped and the remainder of the sample passed through the field to the particle counter. For purposes of counting the trapped cells, the field is deactivated and the cells flushed into the particle counter.

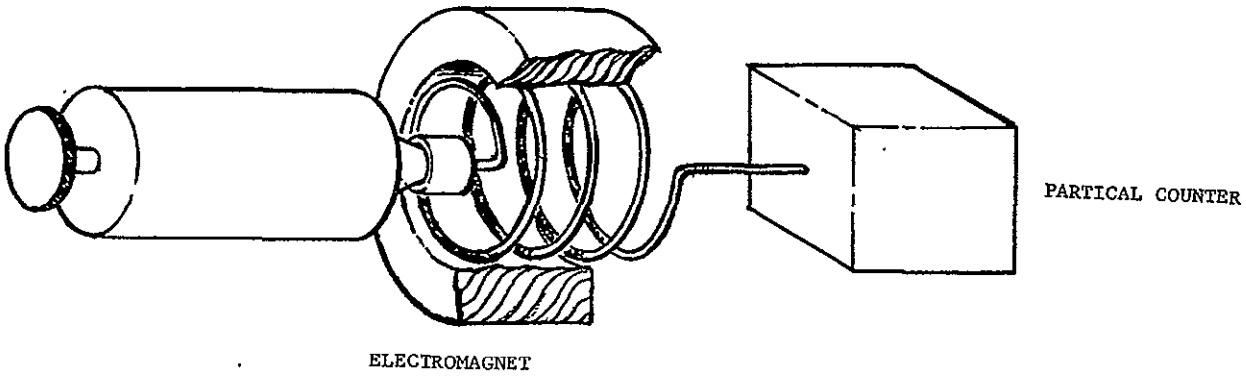

Figure 25-18. Electromagnetic Trap

25.3 LYOPHILIZATION

Although facilities of the Space Station will be available for many analytical procedures, some biological samples will need to be returned to earth for additional analysis. Freeze-dry (lyophilization) equipment will undoubtedly be used for sample preservation.

In earth-borne laboratories, liquid samples to be lyophilized are placed in large-mouth glass bottles or in shallow pans and then prefrozen before being subjected to the vacuum treatment. In the space laboratory, it will be highly desirable to collect and handle the fluids (plasma and urine) in variable volume containers which are kept free of undissolved gases. In the case of plasma, the handling is complicated by the necessity for centrifuging whole blood to remove the cells before lyophilization. If serum is to be preserved, it will have to be separated from the fibrin clot. This can be accomplished in the same centrifuge as used for removing the blood cells from plasma.

Syringes of the kind used for collecting the initial blood specimen can be used for transferring plasma and serum between processes. For freeze-drying, it is expected that 10 to 15 cc aliquots will be injected into collapsed lengths of sealed sections of cellophane dialysis tubing, perhaps fitted with rubber septa. When the tube of fluid is subjected to a vacuum, evaporation of water vapor from the surface will chill the specimen to the freezing point. Following freezing, vapor will continue to pass through the cellophane from the subliming frozen sample. When completely dry, the solids will be conveniently contained within the cellophane bag. Laboratory experiments conducted at Beckman have shown that 10 cc samples of water prepared in this fashion can be vacuum-frozen, and

that lyophilization proceeds at a very acceptable rate. The manner in which the specimen freezes is ideal for proper preservation. The entire tube of liquid (approximately 1.5 cm in diameter and 5-6 cm long) becomes supercooled so that when freezing starts, it proceeds as a wave along the length of the cylinder, producing a solid chunk of ice in less than one second.

Insofar as urine is concerned, the method of sampling and the volume to be handled are somewhat different from those of plasma. It appears that for some tests it will be necessary to obtain pooled, 24-hour urine specimens. Thus, from 1000 to 1500 cc of urine per. day will be collected. However, all of the desired tests can be accomplished with a volume of less than 100 cc. It is, therefore, recommended that the 24-hour collection be pooled and thoroughly mixed, and that only 100 cc be lyophilized, the balance being returned to the water recovery system. This approach will not only result in a requirement for a more compact and less complicated lyophilizer, but will result in a significant reduction in the required amount of stored water. The sample of urine to be processed will thus be only 7 to 10 times that of plasma. The freeze drier can easily be designed to accommodate this range in sample size. It is contemplated that the same technique (freezing and drying in dialysis tubing) can be used for urine as for plasma.

Some method will be required for identifying the initial volume when both plasma and urine specimens are reconstituted. One suggested method is to dissolve a nonvolatile dye which will not interfere with subsequent determinations, to measure the optical density of the specimen before drying, and to reconstitute to the same optical density. Such an approach may not be required if the

experimental protocol includes handling the samples with syringes, because these can be calibrated and even the volume of gas bubbles can be estimated when the samples are in syringes. It may also be possible to control the diameter of the dialysis tubing with sufficient accuracy so that the sample volume can be calculated by simply measuring the length of the filled tube.

The hygenic collection and handling of feces and the drying of the specimen without contamination of the apparatus may require some development effort. No difficulty is envisioned in applying the principle of vacuum-freezing to fecal matter and it seems likely that the material can be frozen and dried in a cellophane wrap. The ground-based analysis will probably be based on dry-weight standards so that reconstitution, per se, will not be required, nor will an initial estimate of specimen volume be required.

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