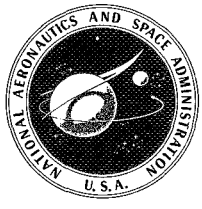
NASA SP-5974 (01)

TECHNOLOGY UTILIZATION

ANALYTICAL AND APPLIED CHEMISTRY

A COMPILATION



NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Foreword

The National Aeronautics and Space Administration has established a Technology Utilization Program for the dissemination of information on technological developments which have potential utility outside the aerospace community. By encouraging multiple application of the results of its research and development, NASA earns for the public an increased return on the investment in aerospace research and development programs.

This document is one in a series intended to furnish such technological information. Divided into two sections, this Compilation presents a number of new or modified analytical instruments and techniques, followed by a number of articles describing chemical processes or chemical techniques, useful in various industries.

Additional technical information can be requested by circling the appropriate number on the Reader Service Card included in this Compilation.

The latest patent information available at the final preparation of this Compilation is presented on the page following the last article in the text. For those innovations on which NASA has decided not to apply for a patent, a Patent Statement is not included. Potential users of items described herein should consult the cognizant organization for updated patent information at that time.

We appreciate comment by readers and welcome hearing about the relevance and utility of the information in this Compilation.

Jeffrey T. Hamilton, *Director*
Technology Utilization Office
National Aeronautics and Space Administration

For sale by the National Technical Information Service, Springfield, Virginia 22151. $1.00

Contents

Section 1. Analytical Chemistry

CHEMICAL AND PHYSICAL PROPERTIES OF HUMAN URINE CONCENTRATES

A study has been made of the chemical and physical properties of urine samples whose concentrations fall in the range from 4 to 90 percent solute. The following list of properties was determined over a temperature range of from 21° to 60° C (70° to 140° F):

solute weight fraction	pH
vapor pressure	refractive index
density	viscosity
solute concentration	specific conductivity
water concentration	surface tension
solute-to-water ratio	differential heat of solution
osmolality	differential heat of vaporization
osmotic pressure	

One of the goals of this study has been to gain an understanding of concentrated urine, to aid in the design of devices for the recovery of water from urine. For this reason, many of these properties are determined as a function of the weight fraction of extracted water and of precipitated solids in evaporated urine.

The report includes a discussion of each of the investigated properties and a description of the experimental methods.

The following documentation may be obtained from:
National Technical Information Service
Springfield, Virginia 22151
Single document price $3.00
(or microfiche $1.45)
Reference: NASA-CR-1802 (N71-32520), Composition and Concentration Properties of Human Urine

Source: D. F. Putnam of
McDonnell Douglas Corp.
under contract to
Langley Research Center
(LAR-10430)

PRETREATMENT OF IRON CATALYST FOR CO_2 REDUCTION

Exhaled CO_2 can be reacted with hydrogen in the presence of an iron catalyst to give water and solid carbon. The water formed by this reaction (known as the Bosh reaction) can be electrolytically decomposed to hydrogen and breathable oxygen. The H_2 may then be reused in the Bosh reaction. This cycle can serve as the basis of an air recycling system.

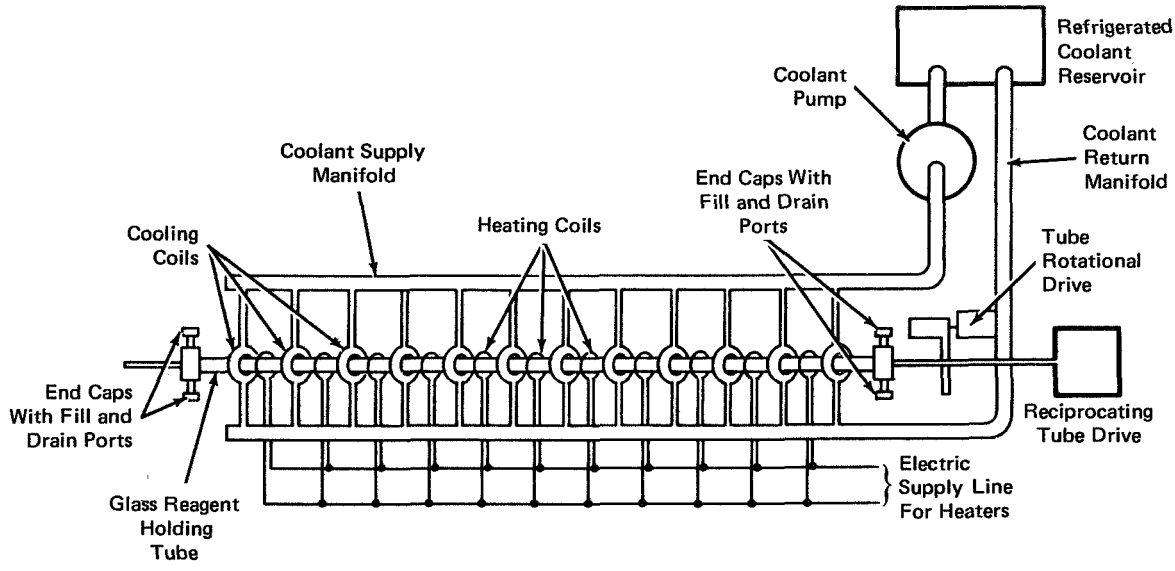
Normally, new iron catalyst requires 7 to 10 hours of heating in a hydrogen atmosphere at around 676° C (1250° F) before the reaction will begin. A new method of pretreating the catalyst eliminates the delay and the high temperatures that were necessary to begin air recycling.

In the new pretreatment process, a steel wool catalyst is immersed in a 2% HCl solution for 2 minutes, rinsed in distilled water, and rapidly dried at 110° C (230° F). Reactor cartridges made from this steel wool will reduce CO_2 as soon as reaction temperatures are reached (about 1.5 hrs).

Source: General Dynamics Corp.
under contract to
Langley Research Center
(LAR-10669)

No further documentation is available.

ZONE PURIFICATION OF ORGANIC LIQUIDS



Zone Purification Apparatus

Zone purification is a well-known procedure for purifying inorganic solids. But the technique is not as well developed, nor the apparatus generally available, for the purification of organic materials. A zone purification apparatus (see figure) developed for the purification of hydrazine may be used with other organic liquids or moderately low-melting-point solids.

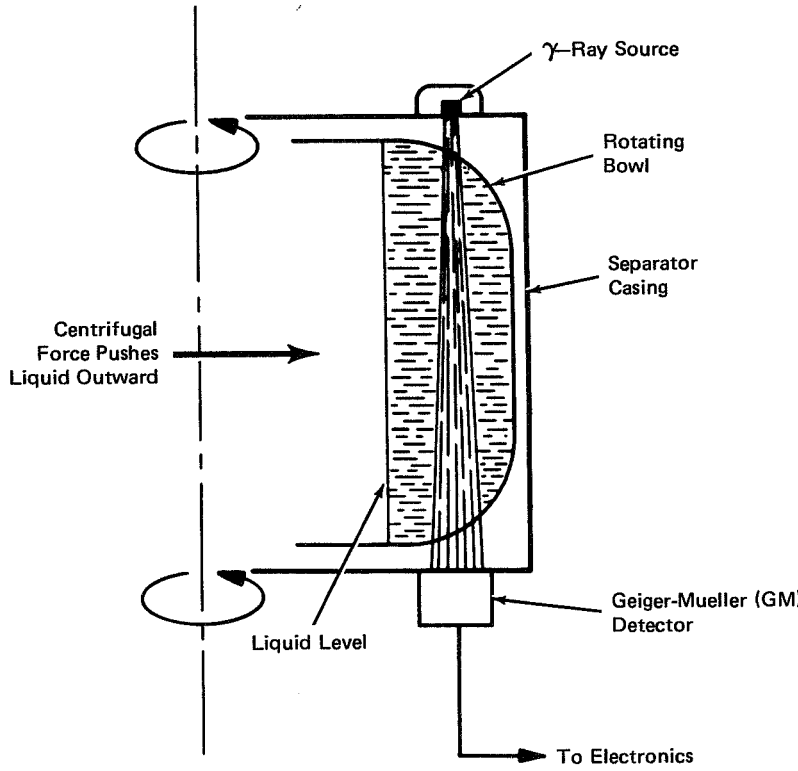
Zone purification takes advantage of the different solubilities of impurities in the liquid and the solid phases. A melted zone is passed through a solid rod of the material to be purified. If the impurity is more soluble in the melt than in the solid, the solidified portion behind the melted zone is purer than the melt. In this manner impurities tend to travel up the rod in the direction of heat flow, or if the impurity is more soluble in the solid, in the opposite direction. This method can reduce contaminants to the parts-per-billion range, a feat beyond the capability of chemical purification.

The apparatus consists of a reagent tube with ports through which the material to be purified is introduced. Cooling is provided by coils attached to a coolant pump and refrigerator. The material in the tube is solidified into a series of solid plugs while the tube is rotating. Next, the tube is slowly advanced so that the solid zone is melted and refrozen. When one full zone-length has been melted, the tube is returned quickly to the starting position, and the process is repeated until the desired purity is reached. At the end of the process, the liquid at each end, containing the impurities, is drained off; and the purified solid material is melted and removed. During the purification of hydrazine, the concentration of one impurity, aniline, was reduced by a factor of 10 on each pass.

Source: L. O. Williams of
Martin Marietta Corp.
under contract to
Langley Research Center
(LAR-10809)

Circle 1 on Reader Service Card.

GAMMA-RAY SPECTROMETRY IN A WATER RECOVERY CONTROL SYSTEM



Location of Source and Detector on Separator

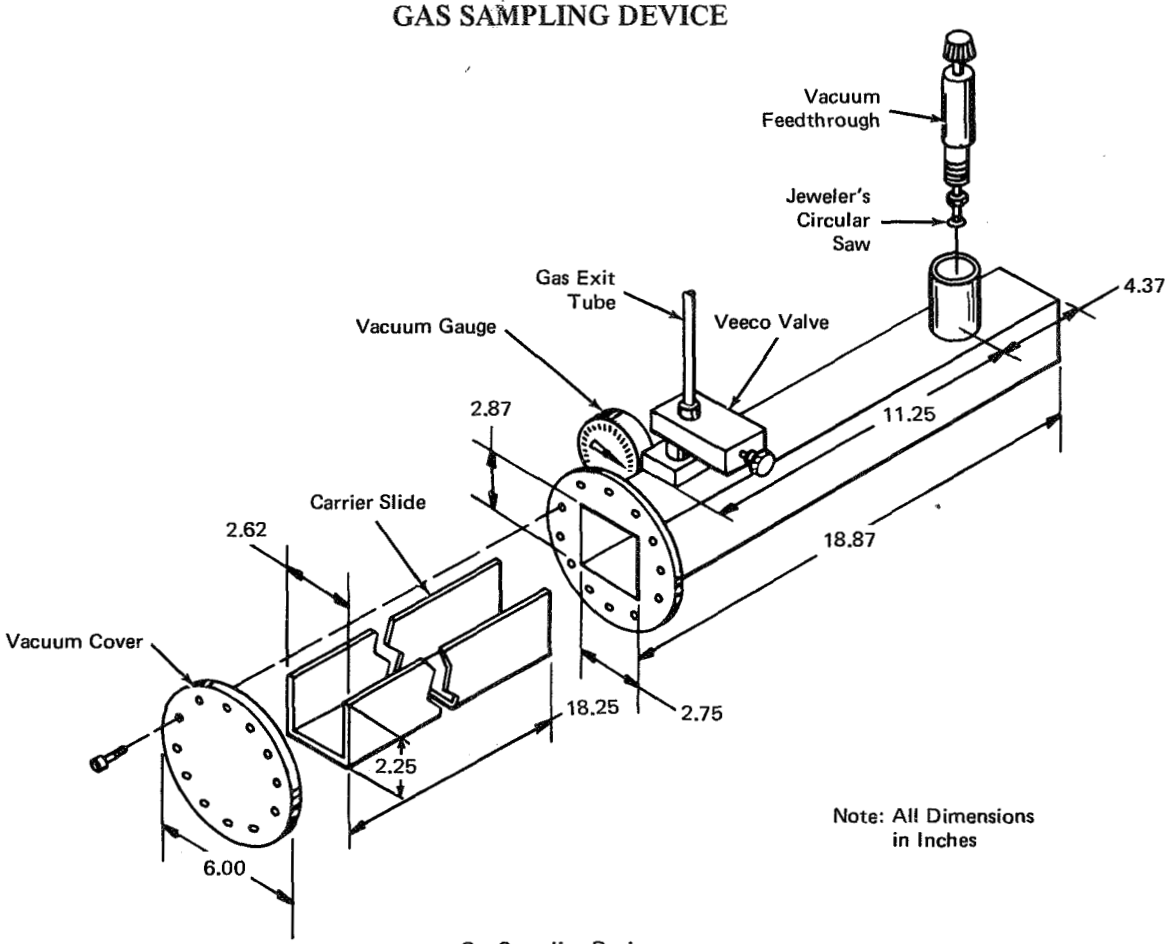
A low-energy gamma-ray source (americium 241) and a Geiger-Mueller tube are used to detect the level and concentration of the brine in the centrifugal separator for a water recovery system. The level and concentration are detected independently. Both detection systems use a low-energy americium-241 source mounted on one side of the phase separator (see figure). The amount of energy passing through the separator depends on the amount of liquid present and on the concentration. A Geiger-Mueller tube on the opposite side detects the radiation, which is converted by an electronic system into a measurement of the liquid density.

In the case of the level detector (not shown), the source and the sensor are placed at an angle to the surface of the liquid, so that exposure of the gamma-ray source to the liquid increases as the liquid level drops.

Source: A. H. Bauer of
Garrett Corp.
under contract to
Johnson Space Center
(MSC-14000)

Circle 2 on Reader Service Card.

GAS SAMPLING DEVICE



Gas Sampling Device

A dependable and convenient system takes samples of gas from a closed envelope and feeds them to analytical equipment. Although designed for use with an SAS counter and a mass spectrometer, the system can be modified to transfer samples of gases from any closed system to other analytical tools such as a gas chromatograph.

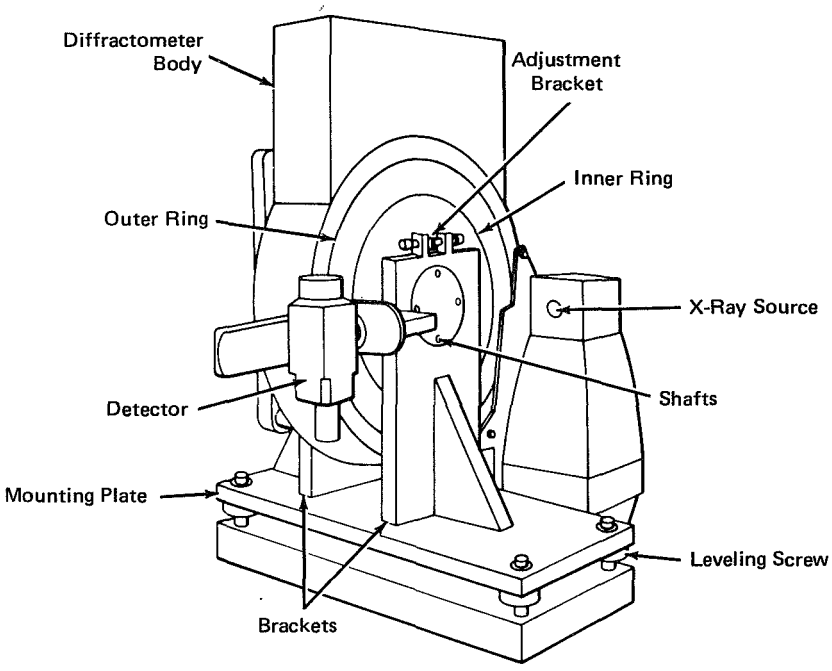
The sampler is shown in the illustration. It is about 50 cm long, 8 cm wide, and 8 cm deep. Its major components include a vacuum gauge and connecting copper tubing, a vacuum feedthrough, a circular saw, and a channel-shaped slide. The SAS counter is attached inside of the slide, which is free to move along the length of the sampler. The copper gas exit tube is attached to the mass spectrometer.

To take a sample, the SAS counter is fixed inside the slide and the sample box is seated. After the sample is pumped down and checked for leaks, the sampling valve is opened, and a background reading is taken in the spectrometer. Next, the system is tilted, and the SAS counter slides under the circular saw. The saw is rotated until it pierces the gas envelope at the site of the fill tube. The envelope is opened at the fill tube so as to make the envelope reusable. Penetration is indicated by an increased pressure on the vacuum gauge. At this point, the sampling valve is reopened, and a mass spectrometer reading is taken. By subtracting the background reading, an accurate measure of the gas is obtained.

Source: Alfred J. Hobbs and
Benjamin Seidenberg
Goddard Space Flight Center
(GSC-10903)

Circle 3 on Reader Service Card.

X-RAY DIFFRACTOMETER ADAPTATION FOR LIQUID STUDIES



θ–θ Diffractometer Front View

Standard θ-2θ X-ray diffractometers can be converted to θ-θ diffractometers that are suitable for liquid studies. With a θ-2θ diffractometer, the sample is rotated relative to the X-ray source, and the detector is rotated at a rate twice that of the sample. To determine the diffraction pattern of a liquid, the sample should remain stationary, while the X-ray source and the detector rotate about it at the same speed, but in opposite directions (a θ-θ system).

A θ-2θ diffractometer can be converted to a θ-θ machine by mounting it on a rigid support through a set of bearings whose axis coincides with the axis of rotation on the diffractometer. The inner ring supports the sample and is attached to a rigid support. The converted diffractometer shown in the figure is a specific model, but the principle applies to other models as well.

In the converted model shown, the diffractometer body is attached to an outer ring that is concentric with a smaller inner ring. The rings are supported by shafts through their centers. The shafts are supported by two aligned ball bearings (not shown) that are mounted in two support brackets. The brackets are affixed to a mounting plate equipped with leveling screws. A bracket mounted on the inner ring holds that ring stationary, but permits small angular adjustments.

The detector is attached to the outer ring. The sample holder, although not shown in the figure, can be mounted on the side of the bracket opposite the detector, in line with the source and the axis of the detector. To obtain a diffraction pattern, the inner ring remains stationary while the outer ring and the diffractometer body rotate about it in opposite directions.

Source: J. R. Guadagno of
Denver Research Institute
under contract to
NASA Headquarters
(HQN-10228)

Circle 4 on Reader Service Card.

COUNTERFLOW ELECTROPHORESIS SYSTEM: IMPROVED RESOLUTION

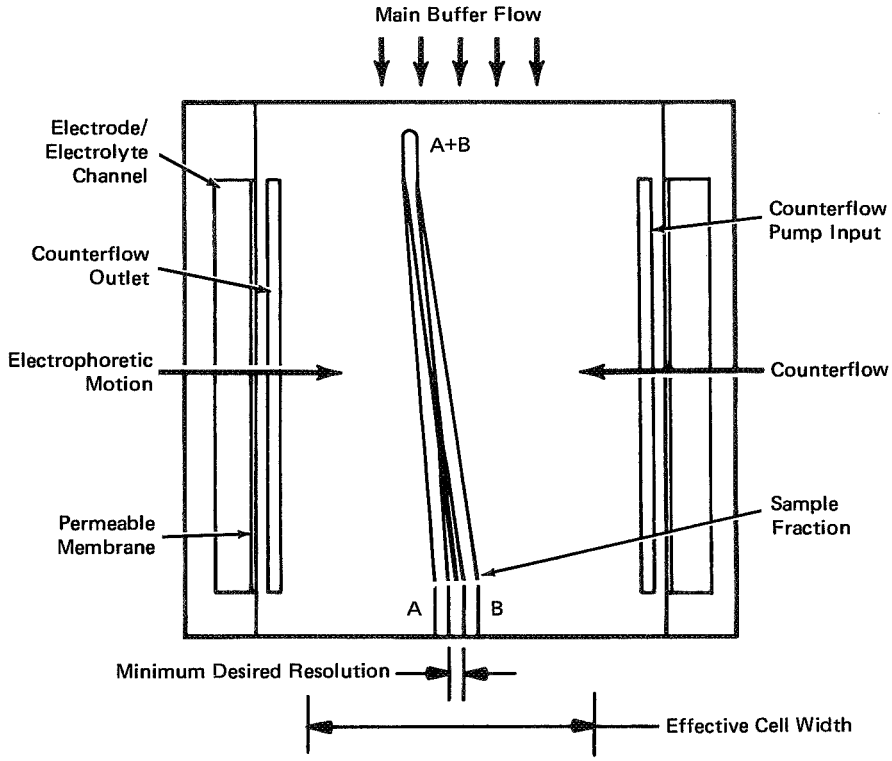


Figure 1. Sample Fraction Paths

Electrophoresis is a widely used technique for separating biochemical and microbiological species. The technique is based on the fact that different species will migrate across a buffer solution at different rates when they are under the influence of an electric potential difference. When the species are very similar, they must travel a greater distance and spend a longer time in the electrophoresis cell to obtain satisfactory resolution. As a result, large cells requiring considerable power are needed to separate similar species; the resolution is subject to degradation from closed-circulation osmotic flow, and the amount of sample throughput is restricted.

An order of magnitude or greater improvement in resolution can be obtained by applying a buffer flow, counter to the direction of migration of the separated species. This increases the effective "distance" the species travel without long exposure times or wide cells. Figure 1 shows the general arrangement of the cell and how the buffer flow opposes the electrophoretic motion. The species move down the cell with the main buffer flow. They migrate horizontally at different rates and should be completely separated by the time they reach the sample collection points. By applying a counterflow of buffer solution as shown, the horizontal distance the sample stream moves can be kept to a minimum, and the cell width kept narrow, despite the need for extensive migration to separate similar species.

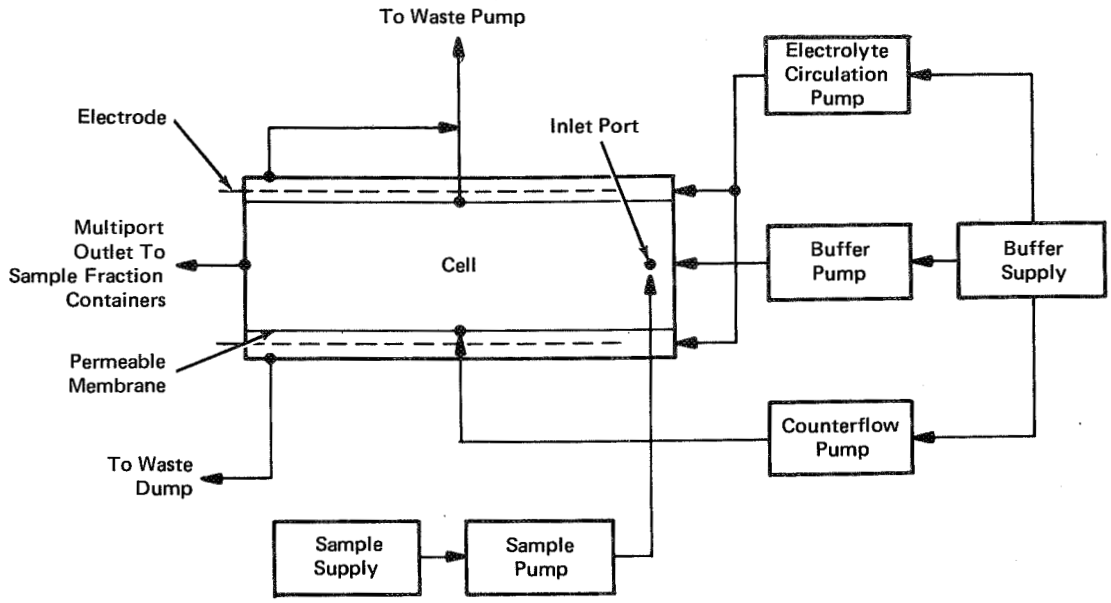


Figure 2. Fluid Flow Diagram

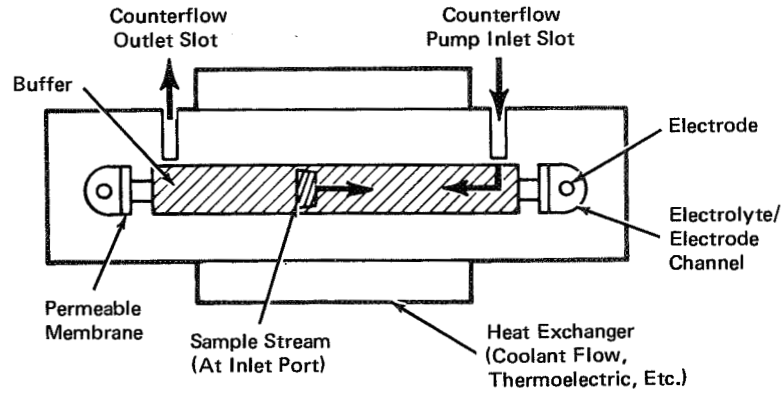


Figure 3. Cell Cross Section

Figure 2 shows the fluid flow in the system, and Figure 3 is a cross section of the cell. The electrolyte circulation pump circulates the buffer solution through the two electrode channels, to remove the products of electrolysis generated by the cell. The counterflow pump injects buffer at right angles to the main buffer flow. The counterflow acts uniformly along the length of the electric field and is adjusted to keep the sample stream in the center of the cell.

Source: G. L. Fogal of General Electric Co. under contract to Marshall Space Flight Center (MFS-22334)

Circle 5 on Reader Service Card.

ELECTROPHORESIS CELL CONTROL SYSTEM

In another article in this section (page 6) an electrophoresis cell is described. This and similar cells separate biochemical species as they migrate through a buffer solution under the effect of a voltage gradient. When separating large amounts of sample, the process can take several hours. During this time, the position of the sample stream can move, making it difficult to continuously collect the sample. Several factors can cause this motion: cell temperature variations, voltage gradient, zeta potential, and sample, buffer and counterflow rates. However, these variables are independent; if a single one of them can be varied, the position of the sample stream can be controlled, provided the remainder are kept within reasonable limits. For example, here a new optical feedback system is used to control the counterflow rate. Optical sensors detect the position of the sample stream; the sensor outputs are then used to control the counterflow rate and keep the sample stream stationary. The system is shown in Figure 1.

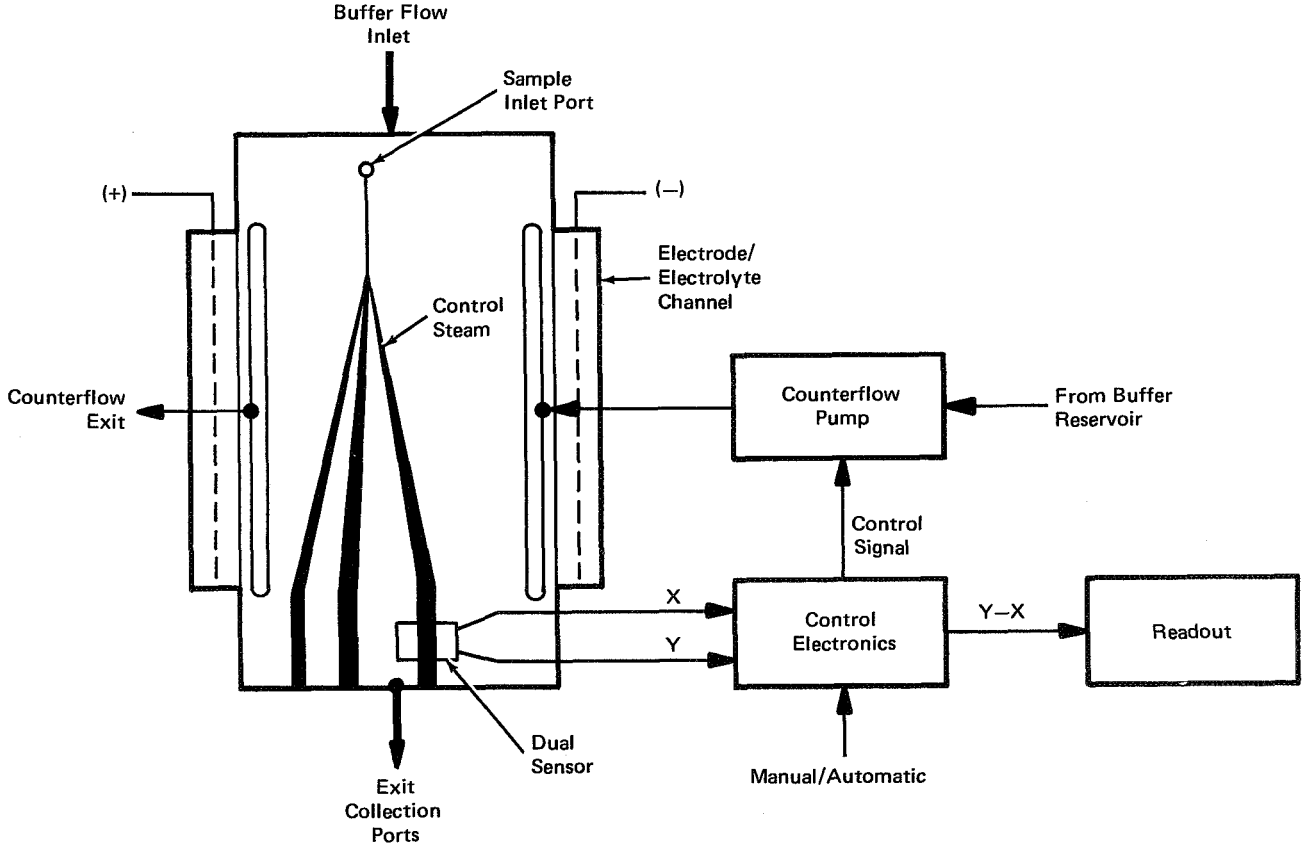


Figure 1. Control System Schematic

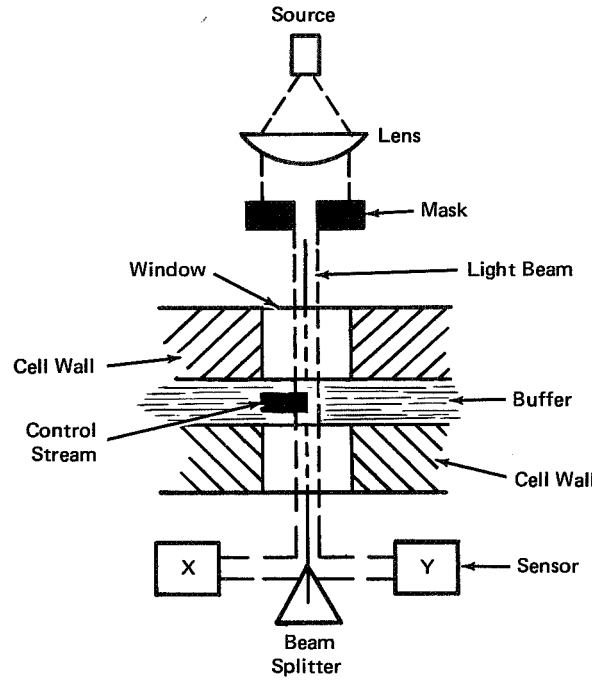


Figure 2. Optical System

The most concentrated sample stream is selected as the control. The sensors are manually positioned across the stream and then adjusted to give a maximum readout at this point. The positioning of the optical system is shown in Figure 2.

If the stream drifts to the left, the output of sensor Y remains constant and the output of sensor X increases; the counterflow rate is then decreased. If the stream drifts to the right, the counterflow is similarly increased.

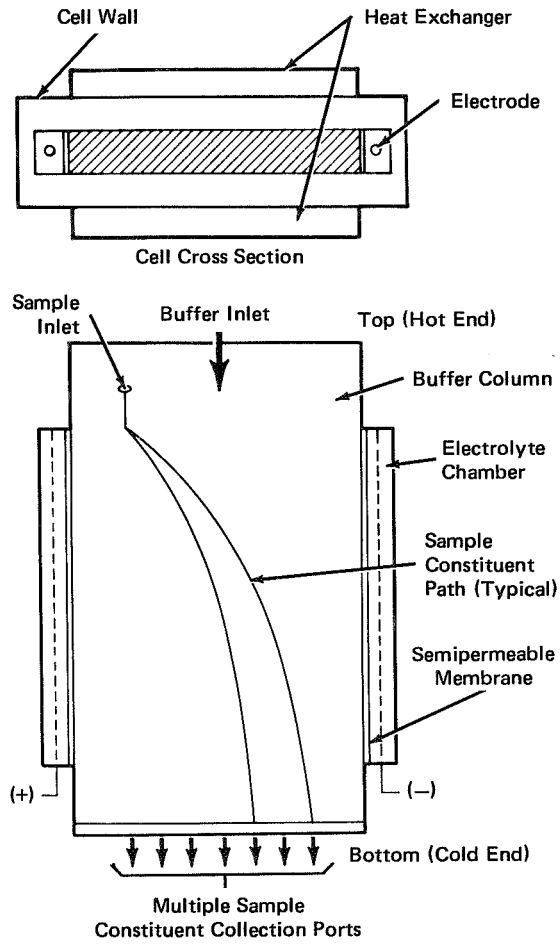
In cells without counterflow, another variable (the buffer flow, for example) may be altered to control the position of the sample stream.

Source: G. L. Fogal of
General Electric Co.
under contract to
Marshall Space Flight Center
(MFS-22379)

Circle 6 on Reader Service Card.

STABILIZED FLOW CELL FOR ELECTROPHORESIS

The separation resolution of existing flow-type electrophoretic cells is limited by local convective flow within the cells. This convective flow spreads and remixes separated sample constituents. The resolution in an electrophoresis cell may be improved by removing the internally generated heat from the cell, which sets up a temperature-dependent, vertical density gradient. The density gradient decreases uniformly, from the bottom (lowest temperature) to the top of the cell (highest temperature). This gradient prevents convective flow disturbances in the cell buffer stream, allowing higher separation resolution.



Stabilized Flow Cell Schematic Representation

The cell is shown in the figure; with the exception of the cell temperature-control means, the cell construction is similar to that used in commercial equipment. The rate of heat removal is controlled locally, so that the buffer temperature increases uniformly with increasing distance from the bottom of the cell. Correspondingly, the buffer density is greatest at the bottom of the cell decreasing uniformly from bottom to top. This density gradient stabilizes the cell buffer column against convective disturbances.

The temperature gradient (and thus the corresponding density gradient) can be provided by a number of heat-removal means. For example:

a. By the use of thermoelectric (TE) elements at appropriate spacings along the length (column dimension) of the cell with constant power input,
b. By the use of TE elements evenly spaced with power input depending upon element position, or
c. By the use of a coolant coil with cold coolant entry at the coil end on the lower end of the cell.

Temperature control can be referenced to either single or multiple locations, the latter providing control on a horizontal section basis.

For aqueous buffer solutions, the cell should be operated at an average temperature exceeding about 20° C. At lower temperatures, the rate of change of buffer density with temperature is relatively low, decreasing to zero at about 4° C. Assuming a buffer temperature at the bottom (exit) of the cell of 20° C and an inlet temperature of 25° C, a density gradient of about 0.0013 gm/cm^3 per °C can be generated. A higher gradient will be generated as the temperature range is increased.

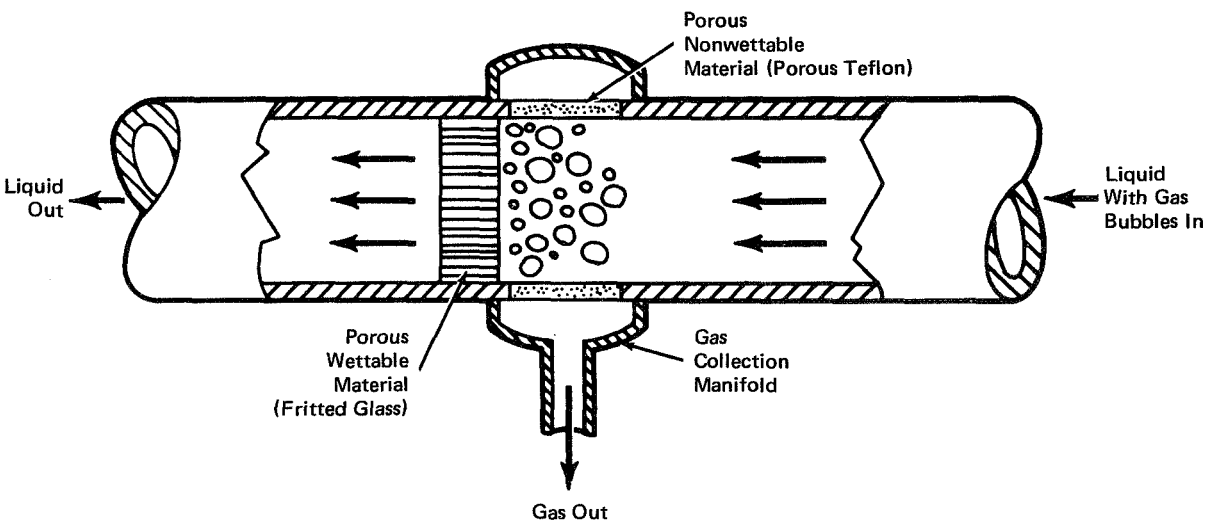
The significant new feature is the incorporation of a temperature-control means for generating a controlled density gradient, with a uniform decrease in buffer density occurring from the bottom (exit) to the top (inlet) of a flow-type electrophoresis cell.

The use of a density gradient for cell stabilization is not new. Generating the density gradient without the addition of other materials (such as sucrose) and the application to a flow-type cell are considered the novel aspects of this device.

Source: G. L. Fogal of
General Electric Co.
under contract to
Marshall Space Flight Center
(MFS-22335)

Circle 7 on Reader Service Card.

GAS/LIQUID SEPARATOR: A CONCEPT



Gas/Liquid Separator

A design for a gas/liquid separator has been conceived, which provides a means of continuously removing entrained gas bubbles from a flowing stream. The separator consists of two porous membranes: (1) a wettable membrane through which the liquid only can pass and (2) a nonwettable membrane through which the gas only is removed. As shown in the figure, the wettable porous membrane placed across the stream allows the liquid to pass through, but prohibits passage of the gas. The nonwettable membrane, located immediately upstream, allows the collected gas to be removed into the collection manifold, but prohibits passage of the liquid.

The materials of which the membrane are made, their porosities, sizes and operating pressure ratios are selected according to the type and characteristics of the gas and liquid being separated and the operating conditions of the system. For example, an experimental wettable porous membrane for the separation of air and water is constructed of fritted glass having a pore size of 10 to 15 micrometers and a grit size of 0.114 to 0.06 mm (0.0025 to 0.0045 inch). This construction has been tested and works satisfactorily. A companion nonwettable porous membrane can be made of suitably perforated Teflon.

Source: P. F. Berg of
United Aircraft Corp.
under contract to
Lewis Research Center
(LEW-11792)

No further documentation is available.

DIFFERENTIAL SPECTROSCOPY OF ATMOSPHERIC POLLUTANTS

Absorption spectroscopy is one of the fastest and most convenient methods of measuring atmospheric pollutants such as carbon monoxide. This method allows concentrations to be found quickly, accurately, at a distance from the measured location, and requires no wet-chemical analysis. However, there are several factors that currently prohibit the effective use of spectroscopy to detect trace gases in the atmosphere. The absorption of electromagnetic radiation by gases depends upon pressure and temperature. Thus, it is necessary to know the thermodynamic state of the atmosphere at the point of measurement, in order to interpret the absorption spectrum. In addition, the absorption signals are usually weak and are interfered with by background radiation (ground clutter) and atmospheric propagation effects.

It has been suggested that these problems can be overcome if the absorption measurement is taken at a crossover wavelength and if differential spectroscopy is used; i.e., if a second reference measurement is taken at the nearest wavelength outside the absorption spectrum of the gas of interest. At the crossover the wavelength, temperature and pressure do not affect absorption by the gas. Normally, these factors may increase or decrease the absorption, depending upon the particular wavelength at which the measurement is taken. If one scans the absorption spectrum, there will be a wavelength at which temperature and pressure increases no longer increase the absorption, but decrease it. At this crossover point, there will be a zero increase or decrease; or, in other words, the absorption will be independent of temperature and pressure.

The effects of ground clutter and atmospheric propagation are compensated for by differential spectroscopy. The absorption at the reference wavelength includes all the interfering effects, but since it is outside the absorption of the trace gas, it contains no information about trace gas concentration. The reference spectrum is then used to subtract all absorption, other than that of the gas being measured, from the crossover-wavelength spectrum.

Source: F. R. Krause
Marshall Space Flight Center
(MFS-22632)

Circle 8 on Reader Service Card.

STABILITY OF Fe(II) IN FERRIOXALATE SOLUTIONS

Radiation dosages can be measured by the photoreduction of Fe(III) to Fe(II) oxalates through the reaction:

$$2\ Fe(Ox)_3^{-3} \xrightarrow{h\nu} 2\ Fe(Ox)_2^{-2} + 2\ CO_2 + Ox^-$$

This reduction is sensitive and has a wide range of response. But no data existed to allow the quantitative determination of the effects of long-term disappearance of Fe(II). Such information is needed for Fe(III) – Fe(II) actinometers that are analyzed several days after exposure.

In a study of the stability of Fe(II), it has been found that Fe(II) undergoes significant decomposition after several days. The rate of decomposition is dependent upon pH, temperature, and the concentrations of Fe(II) and $Fe(Ox)_3^{-3}$. Minimum decomposition occurs at low temperatures, high acidity, and low $Fe(Ox)_3^{-3}$ concentrations. Quantitative data have been developed that allow correction factors to be calculated for solutions stored after irradiation.

The recommended actinometer solution is 0.006 to 0.008 M ferrioxalate at a pH of 1.50 ± .05. Under these conditions, the rate of disappearance of Fe(II) is on the order of 2.5% in 24 hours at 23° C.

Source: M. Wrighton and S. Witz of
Aerojet General Corp.
under contract to
Johnson Space Center
(MSC-14188)

No further documentation is available.

CHEMICAL SPOT TEST FOR ETHYLENE GLYCOL

The present methods for detection of ethylene glycol are either insufficiently sensitive to small amounts of ethylene glycol or give positive responses to other substances. A new, simple thin-layer chromatography technique can be performed in 15 minutes and will selectively reveal 25 ppm of ethylene glycol in water.

A 100-mm capillary tube (I.D. approximately 1 mm) is filled to about 35 mm with a sample and diluted to 100 mm with water. The sample is applied in five 7-mm portions to a silica gel panel, which is heated to 360 ± 5 K (190° ± 10° F) on a hotplate. The solution is allowed to partially dry between each application. The sensitivity of the technique is increased if the silica gel is warmed during the test. The panel is then removed from the hotplate and allowed to dry thoroughly for 5 minutes. After drying, the panel is sprayed in a hood with Solution I (see below).

After partial drying (2-5 minutes), the panel is sprayed with a Solution II from a distance of 25 to 45 cm, until a uniform blue color develops. The blue color is the product of a periodate-benzidine reaction. The appearance of a white spot indicates the presence of ethylene glycol. White spots occur in areas where ethylene glycol has been present, because the glycol oxidizes the periodate and removes it before it can react with the benzidine.

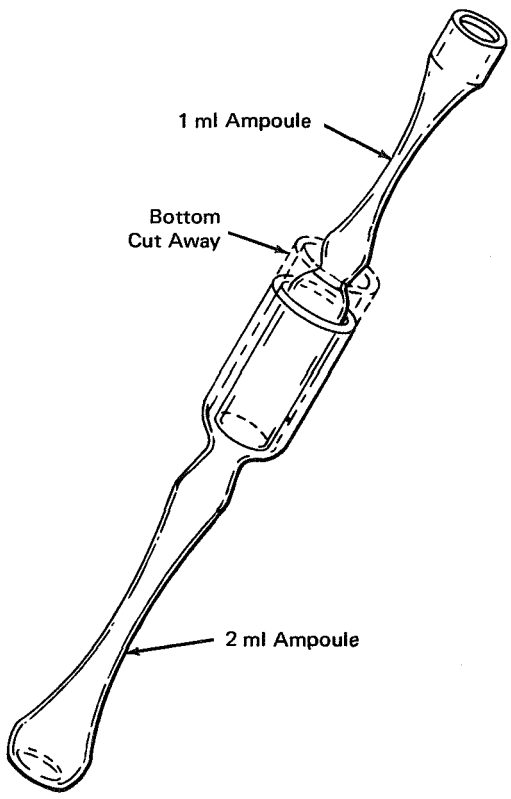
Solution I	Solution II*
0.1% by weight of	2.8 g benzidine,
sodium metaperiodate	80 ml-ethanol,
in distilled water	70 ml-distilled water,
	30 ml-acetone, and
	1.5 ml-normal HCl

*Caution: Safety authorities have suggested that, for health reasons, there should be no bodily contact with Solution II; or that, in the preparation of Solution II, there should be no bodily contact with benzidine.

Source: M. Gussack, E. A. Cheslack, and
C. A. Smith of
Grumman Aerospace Corp.
under contract to
Johnson Space Center
(MSC-12325)

No further documentation is available.

AMPOULE HOLDER



Ampoule Holder

Several procedures in quantitative chemical analysis require the use of 1-milliliter ampoules (calibrated glass bottles). Since these ampoules are very small, they are difficult to handle. This is particularly true when the reagent involved is either toxic or corrosive. Additionally, when the ampoule is handled, a thumb or a finger may conceal the proper fluid level.

This problem can be overcome by using a 2-milliliter ampoule, with the bottom removed, as a holder for the 1-milliliter ampoule. The inside diameter of a standard 2-milliliter ampoule provides a slip fit for the outside diameter for the 1-milliliter ampoule. By cutting out the bottom of the larger one and using its neck for a handle, as shown in the illustration, the smaller ampoule is handled more easily. An index mark may be scribed on the larger ampoule to indicate the proper location for the liquid meniscus in the smaller one.

Source: R. J. Smith of
Rockwell International Corp.
under contract to
Johnson Space Center
(MSC-15907)

No further documentation is available.

Section 2. Chemical Processes and Applied Chemistry

MODIFIED ION SOURCE FOR MATERIALS WITH HIGH VAPORIZATION TEMPERATURES

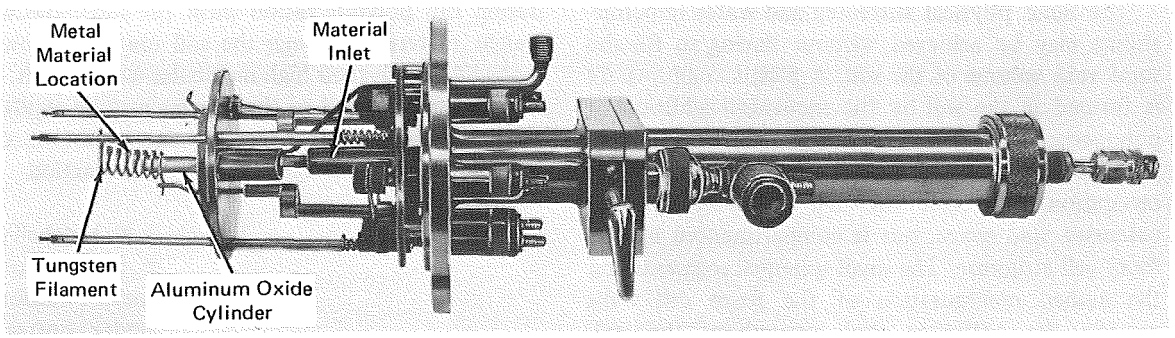


Figure 1. Disassembled View of Danfysik Model 910 Ion Source

A commercially available ion source has been modified to produce ions of metals with relatively high melting points. As originally designed, this machine produces ions from gases, liquids, or solids which vaporize at temperatures lower than 1200° C.

The source (shown in the figure) consists of a heated filament in an ion chamber that is surrounded by a coaxial magnetic field. There is a potential difference of a few hundred volts across the chamber, from front to back. A vaporized sample is introduced through a material inlet and is ionized in the chamber. An electrostatic lens accelerates the positive ions, which then may be used for doping semiconductors.

The maximum temperature reached by the vaporization oven in a standard model is 1200° C. This drastically limits the usefulness of the system, as it excludes gold (2808° C), cobalt (3100° C), nickel (2900° C), and most other metals.

To increase the temperature of the vaporization oven and expand the ranges of ionizable materials, an aluminum oxide cylinder [0.450 cm (0.177 in.) in diameter – 6.93 cm (2.73 in.) in length] is used. The cylinder is placed in the chamber, concentric with the tungsten filament. A depression in the front of the cylinder holds the sample and allows it to be placed very close to the tungsten filament. Because this increased proximity exposes the sample to a higher temperature, a wider range of materials can be ionized with this system. The table presents the ion yield of a number of metals, most not ionizable with the unmodified ion source.

Material	Former Yield (in microamperes)	Yield by new method (in microamperes)
Iron	0	40
Lanthanum	0	10
Gold	0	5
Aluminum	0.25	100
Beryllium	0	5
Nickel	0	30
Magnesium	0	40
Titanium	0	10
Vanadium	0	40
Cobalt	0	40

Ion Yield for Modified Source as Compared with Standard Device

Source: John Burgess, Tucker Clark, and
Herbert D. Hendricks
Langley Research Center
(LAR-11385)

No further documentation is available.

SOIL STABILIZATION WITH IN-PLACE FORMED PLASTIC FOAM

Soil stabilization is used to strengthen soil that serves as the base of a structure or that must otherwise hold its shape against erosion. The usual method is to add very large amounts of a chemical grout, which fills the voids in the soil and hardens to form a less permeable and stronger structure. Large amounts of grout are needed because most of the void volume of the soil must be filled.

The same physical stiffening and water impermeability may be achieved without having to fill the total void volume of the soil. A plastic foam matrix is formed in the soil by the sequential addition of polymerizable, relatively low-molecular-weight compounds that emit a foam-forming gas during polymerization. The resulting soil structure contains extensive void space, but is compartmented by the foam cell structure. The small spherical segments and the arched configuration of the foam cell walls provide the stiffening that strengthens the soil. Though one-third to one-half the amount of foam is needed as of chemical grout, the soil structure is at least as strong with foam stabilization as with grout. Furthermore, since the foam is less expensive than the grout, this method is much more economical.

The foam is formed from low-molecular-weight (around 200) compounds that have a low enough viscosity to penetrate the soil. As these are added in steps, with the appropriate curing agents, higher-weight three-dimensionally cross-linked plastics form. The reaction is slow enough to allow the mixtures to penetrate the soil throughout, before hardening renders the soil impermeable. The gases produced during the polymerization push the low-molecular-weight materials through the soil until the growing molecules reach their high-molecular-weight limit.

The overall speed of the process may be regulated by using compounds which display a fast or slow initiation time in the early stages of the reaction.

Source: J. K. Mitchell and
B. P. Anderson of
University of California
under contract to
Marshall Space Flight Center
(MFS-20882)

Circle 9 on Reader Service Card.

CORROSION INHIBITOR FOR WATER STERILIZATION SYSTEM

A number of water sterilization systems contain the sterilizing chlorine releaser, sodium hypochlorite, and a sodium dihydrogen phosphate buffer. Such systems, when made of aluminum, undergo extensive pitting corrosion. The addition of sodium nitrate prevents this corrosion, provides excellent pH control, and leads to a low chlorine decay rate.

In a system containing 5 ppm of sodium hypochlorite and 120 ppm of sodium dihydrogen phosphate, the addition of 63 ppm of sodium nitrite eliminates approximately 98% of the corrosion. The addition of 137 ppm of sodium nitrite has been found to completely inhibit corrosion. However, 100 ppm would probably be sufficient for many applications. The larger amount would be used for long-term applications.

Source: L. N. Veeder of
Rockwell International Corp.
under contract to
Johnson Space Center
(MSC-15711)

Circle 10 on Reader Service Card.

FREEZE DRYING TECHNIQUE FOR THE PREPARATION OF FILLED POLYMER

The preparation of highly filled polymer is difficult. The viscosity becomes so high that too high a temperature is required for mixing, and the polymer can degrade. When oxidizers are used as the filler, the problem becomes even more acute. The use of solvents to plasticize the mass is indicated, but then there remains the problem of the sedimentation of the filler particles after mixing is complete, as well as that of the removal of the solvent prior to the final molding or the processing.

In working with thermoplastic elastomers, it has been found that such solvents may be removed by a freeze drying process that allows the thermoplastic to be converted to a homogeneous binder without the interference of sedimentation.

The elastomer is made as follows: The polymer binder is dissolved in a sublimable solvent such as benzene. An oxidizer-filler, such as solid ammonium perchlorate, is added. Next, the resulting mixture is frozen by slowly pouring it, in a continuous stream, into a cryogenic bath. Finally, the frozen solvent is removed by sublimation. The resulting composition is suitable for conventional processing, such as compression molding or extrusion.

Typical formulations processed in this manner have up to 80 percent ammonium perchlorate loading and 20 percent elastomer. The method is also satisfactory when up to 5 percent mineral oil is present. The mineral oil has a negligible vapor pressure and is not removed by sublimation.

Source: Billy G. Moser and
Robert F. Landel of
Cal Tech/JPL
under contract to
NASA Pasadena Office
(NPO-10424)

Circle 11 on Reader Service Card.

SOLDERING Ni SPAN C TO STAINLESS STEEL

Tin can be used to solder, at low temperature, Ni Span C to 321 Stainless Steel, to produce a bond compatible with dry nitrogen oxides. The mating surfaces are tinned and sweated together.

The most critical part of the operation is wetting the steel with tin. This operation can be simplified if the steel is nickel plated and the plated surface tinned. The best results are obtained if the tin is applied as pieces of foil.

The procedure is as follows:

1. The Ni Span C is pickled at 130° F (54° C) for 4 to 5 minutes in 20 percent sulphuric acid and 40 g/l $NaNO_3$. It is rinsed with alcohol.
2. A zinc-chloride soldering paste is used to tin the surface with pure tin.
3. The tinned Ni Span C is washed in hot water and rinsed in alcohol to remove all traces of flux.
4. Its mating surface is roughened with emery cloth.
5. The steel is pickled in 50 percent HCl for two minutes and rinsed with alcohol.
6. It is immersed in fresh Duzall paste for two minutes.
7. It is immediately tinned.
8. It is washed and rinsed in alcohol.
9. The tinned surfaces for mating are then sweated together with gentle pressure.

Source: M. Prager of
Rockwell International Corp.
under contract to
Johnson Space Center
(MSC-15963)

No further documentation is available.

CORROSION IN SILVER-TITANIUM SOLAR CELLS: A STUDY

An investigation of corrosion in silver-titanium solar cell contacts showed that several factors contributed to degradation of the cells: humidity, porosity, elevated temperatures, and contaminates such as halides and hydrides.

In a humid atmosphere, moisture will condense in the silver layer of the solar cell whenever a critical humidity is exceeded. The critical humidity depends on the porosity of the layer, which in turn depends on manufacturing conditions such as substrate temperature, deposition rates, and the vacuum used. Moisture in the silver layer causes degradation of the cell and also accelerates degradation due to chemical impurities.

Halide ions (primarily Cl^- and F^-) are present as impurities in the silver and in condensing atmospheric moisture. Even in small quantities, these ions cause accelerated degradation. In addition, the acidity of CO_2 containing atmospheres is sufficient to markedly accelerate degradation.

Several techniques were used to investigate the causes of corrosion. The most informative were mass spectroscopy and internal reflection spectroscopy. Electrical measurements such as current-versus-voltage data were not as useful, as they did not detect silver separation, an important factor contributing to shorter cell lives.

Recommendations to increase the life of cells included:

(a) Use a 25-micrometer layer of solder about the silver contact to protect it against moisture.
(b) Store the cells in a dry nitrogen atmosphere.
(c) Control the trace quantities of halide ions introduced during manufacture.
(d) Replace the silver layer with a gold layer, which is much less porous.
(e) Investigate the possibility of replacing the titanium layer, which is quite reactive, with a nonreactive substitute such as a gold-platinum alloy.

The following documentation may be obtained from:
National Technical Information Service
Springfield, Virginia 22151
Single document price $3.00
(or microfiche $1.45)
Reference: NASA-CR-111768 (N70-42477), Investigation Into the Mechanism of Degradation of Solar Cells with Silver - Titanium Contacts

Source: Charles J. Bishop and Henery Oman of
The Boeing Co.
under contract to
NASA Headquarters
(HQN-10682)

CHEMICAL PROCESSING MANUAL

All the documents used in the NASA Product Engineering and Process Technology Laboratory have been put into a single Chemical Processing Manual. This illustrated 900-page publication discusses cleaning, pickling, surface finishing, chemical milling, plating, dry-film lubricants, and polishing. The manual is divided into sections covering specific materials such as: aluminum alloys, stainless alloys, superalloys of nickel and cobalt, titanium alloys, steels, and nonmetallic materials. Most of the processes are described in detail, many with step-by-step instructions. The manual will be useful to chemical and metallurgical firms and other manufacturers who produce or process materials.

Source: F. J. Beyerle
Marshall Space Flight Center
(MFS-22548)

Circle 12 on Reader Service Card.

CHEMICAL REMOVAL OF BROKEN HIGH-SPEED DRILLS FROM TYPE 347 CORROSIVE-RESISTANT STEEL

Solutions of boiling nitric (HNO_3) and sulfuric (H_2SO_4) acids in a 3:1 or greater ratio will rapidly dissolve high-speed drill bits embedded in Type 347 corrosive-resistant stainless steel and leave the steel unharmed.

It is well known that a metal placed in an acid will begin to dissolve, but it is perhaps less well known that oxidizing acids such as nitric acid also become powerful oxidizers at high concentrations. This oxidizing action can cause the formation of a "passive" oxide coating on the surface of the metal which protects it from attack by hydrogen ions. The HNO_3 concentration at which this occurs depends on the particular metal under consideration. For example, the drill bits, which contain a high tungsten content, require a higher HNO_3 concentration for passivation than does the high-chromium stainless steel. Nonoxidizing sulfuric acid is added to the HNO_3 because it increases the hydrogen-ion concentration and the speed of dissolution, without causing the drill bit to become passified.

To dissolve a broken drill from a stainless steel part, first remove all grease and oil. Then immerse the part in a solution of 250-g/l HNO_3 and 80-g/l H_2SO_4 at a temperature of 200° – 210° F (93° – 99° C). The time required depends upon the drill size and the number of times the solution has been used, but should be in the neighborhood of 3 to 5 minutes for a 0.05-gram drill bit.

Source: R. T. Kessler of
Rockwell International Corp.
under contract to
Johnson Space Center
(MSC-15976)

Circle 13 on Reader Service Card.

STERILIZATION OF LUCIFERASE

Luciferase, an enzyme obtained from firefly tails, is used to detect life. Luciferase emits light when it reacts with adenosine triphosphate (ATP), a substance present in all living organisms. This reaction has several uses; for instance, it is used to detect the presence of bacteria in body fluids. However, false detection may occur when the enzyme itself is contaminated with other living cells.

To insure that no living cells are present as contaminants, the luciferase must be sterilized. The two most common methods of sterilization are by heating or through the action of chemicals. As normally practiced, these techniques make the enzyme unfit for the ATP reaction.

In a new method, luciferase is sterilized by heating, while it is in contact with a molecular sieve (e.g., a commercially available cross-linked polysaccharide dextran as powdered microscopic beads) and under reduced pressure. The enzyme is sterilized by heating it at 135° C for 36 hours at a pressure of 5×10^{-4} mm Hg. The luciferase sterilized in this manner can still be used for the ATP reaction.

Source: E. W. Chappelle and E. Rich
Goddard Space Flight Center
(GSC-10225)

Circle 14 on Reader Service Card.

Patent Information

The following innovations, described in this Compilation, have been patented or are being considered for patent action as indicated below:

Counterflow Electrophoresis System: Improved Resolution (Page 6) MFS-22334

Inquiries concerning rights for the commercial use of this invention should be addressed to:

Patent Counsel
Marshall Space Flight Center
Code A&PS-PAT
Marshall Space Flight Center, Alabama 35812

Electrophoresis Cell Control System (Page 8) MFS-22379

Inquiries concerning rights for the commercial use of this invention should be addressed to:

Patent Counsel
Marshall Space Flight Center
Code A&PS-PAT
Marshall Space Flight Center, Alabama 35812

Stabilized Flow Cell for Electrophoresis (Page 10) MFS-22335

Inquiries concerning rights for the commercial use of this invention should be addressed to:

Patent Counsel
Marshall Space Flight Center
Code A&PS-PAT
Marshall Space Flight Center, Alabama 35812

Differential Spectroscopy of Atmospheric Pollutants (Page 12) MFS-22632

Inquiries concerning rights for the commercial use of this invention should be addressed to:

Patent Counsel
Marshall Space Flight Center
Code A&PS-PAT
Marshall Space Flight Center, Alabama 35812

Modified Ion Sources for Materials with High Vaporization Temperatures (Page 15) LAR-11385

Inquiries concerning rights for the commercial use of this invention should be addressed to:

Patent Counsel
Langley Research Center
Code 456
Hampton, Virginia 23665

PATENT INFORMATION

Freeze Drying Technique for the Preparation of Filled Polymer (Page 17) NPO-10424

Title to this invention, covered by U.S. Patent No. 3,651,008, has been waived under the provisions of the National Aeronautics and Space Act [42 U.S.C. 2457 (f)], to California Institute of Technology, Pasadena, California 91109

Sterilization of Luciferase (Page 19) GSC-10225

This invention has been patented by NASA (U.S. Patent No. 3,745,089). Inquiries concerning nonexclusive or exclusive license for its commercial development should be addressed to:

Patent Counsel
Goddard Space Flight Center
Code 204
Greenbelt, Maryland 20771

Notes: