

Progress Report for the Period of January -June 1967

NASA Grant NGR 05-009-032

A Feasibility Study on Miniaturizing an Automatic Amino Acid Analyzer for use on Apollo Mission and Mars Voyager Mission.

GPO PRICE \$ \_\_\_\_\_

CFSTI PRICE(S) \$ \_\_\_\_\_

Hard copy (HC) 3.00Microfiche (MF) .65

# 653 July 65

FACILITY FORM 802	<b>N67-31303</b>	_____
	(ACCESSION NUMBER)	(THRU)
	<u>8</u>	<u>1</u>
	(PAGES)	(CODE)
	<u>CR-85860</u>	<u>14</u>
	(NASA CR OR TMX OR AD NUMBER)	(CATEGORY)

Stanley L. Miller - Principal Investigator  
 Joseph Kraut - Co-Principal Investigator  
 Karl M. Dus - Co-Principal Investigator

Department of Chemistry  
 University of California, San Diego  
 P. O. Box 109  
 La Jolla, California 92038

## Introduction.

In the period since the last progress report, substantial progress has been made in showing the feasibility of automatic amino acid analysis by ion exchange chromatography for use on Apollo and Mars Voyager missions. The semi-micro model has been completed and tested. The design of the micro model has been completed and the construction of its component parts is in progress. These developments re-enforce our confidence that the construction of an automatic amino acid analyzer suitable in size and weight for space flight application will prove to be feasible. The details of these developments are as follows:

### 1. Semi-Micro Model

The overall construction of the semi-micro model according to the design outlined in the preceding progress report has now been completed. The dimensions of this instrument are 30" x 24" x 20".

An extensive series of test experiments was performed in order to ensure compatibility of component parts and to establish the performance characteristics of the instrument.

a) The elution pattern and the resolution of individual amino acids was found to be in close agreement with the data obtained on a 73 cubic foot commercial amino acid analyzer.

b) This semi-micro model offers several advantages over the commercial instruments. These are the automatic sample selection and a completely automatic program for stepwise multi-buffer elution of amino acids followed by regeneration and re-equilibration of the ion exchange column.

c) Construction of the unit for automatic hydrolysis and separation of amino acids from contaminating inorganic material is also nearing completion and this unit will be tested shortly.

d) Although the present sensitivity of our instrument is already higher than the sensitivities provided for commercial analyzers ( $10^{-8}$  moles), we consider it necessary to raise this level still higher. This will be possible with the aid of a stabilized operational amplifier which is presently being tested in our laboratory.

#### 1. New Developments.

The construction of the semi-micro model has greatly aided our efforts to further miniaturize the instrument. Each component part was carefully tested and re-evaluated with regard to its functional design and the possibilities for further reduction in size and weight.

This search for improvements led to the following developments:

a) A more compact arrangement of buffer compartments has been achieved by combining the containers for all buffers and for the ninhydrin reagent into one unit made from molded plastic. The size of each individual compartment of this unit is made proportional to the respective volumes required by the elution pattern.

b) In order to eliminate the possibilities of bubble development during the elution period of the fourth buffer, containing sodium carbonate, a suitable sodium borate buffer was provided as replacement.

c) Although our present pumping system proved completely adequate for the requirements of the semi-micro model we intend to further improve the control of the liquid flow-system by adjusting the piston size of each pumping head in proportion to the desired output. The design of this component also permits the use of plastic instead of metal as the major construction material of the pumping system. This replacement will therefore result in a substantial reduction of the size and the weight of this component while simultaneously improving the control of the liquid system.

d) Experiments were conducted with the aim of producing suitable micro ion-exchange columns. One solution to this problem is to use a capillary column made from plastic tubing and polystyrene resin beads. Another possibility would be the construction of a polystyrene column coated with activated resin. Both approaches are being developed and tested.

e) Much effort was spent on devising a detection system suitable for high sensitivity measurements. Again two different possibilities were studied.

Our high sensitivity flow cell of 30 mm light path in combination with a suitable colorimeter and a stabilized operational amplifier of low current requirements was found to permit qualitative amino acid analyses down to the level of  $10^{-10}$  moles.

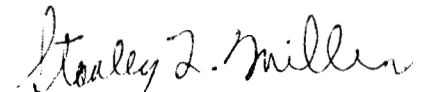
The alternate possibility entails the use of a compact monochromator system connected to a photomultiplier. This arrangement has enabled us to raise the sensitivity for both visible and ultraviolet to the level of  $10^{-12}$  moles. However, it will be necessary to develop a suitable means of suppressing the relatively high background noise presently observed at this high level of sensitivity.

f) Electrical heating of the reaction coil occasionally resulted in spot heating, and this undesirable side effect was prevented by immersing the coil in a small volume of paraffin oil. Other solutions to this problem are being investigated.


g) An entirely new approach to the pressure pump for elution has been developed. This device consists of a bellows which acts as a piston on the buffer. The bellows is driven by air pressure. This system has been successfully tested and is being further refined.

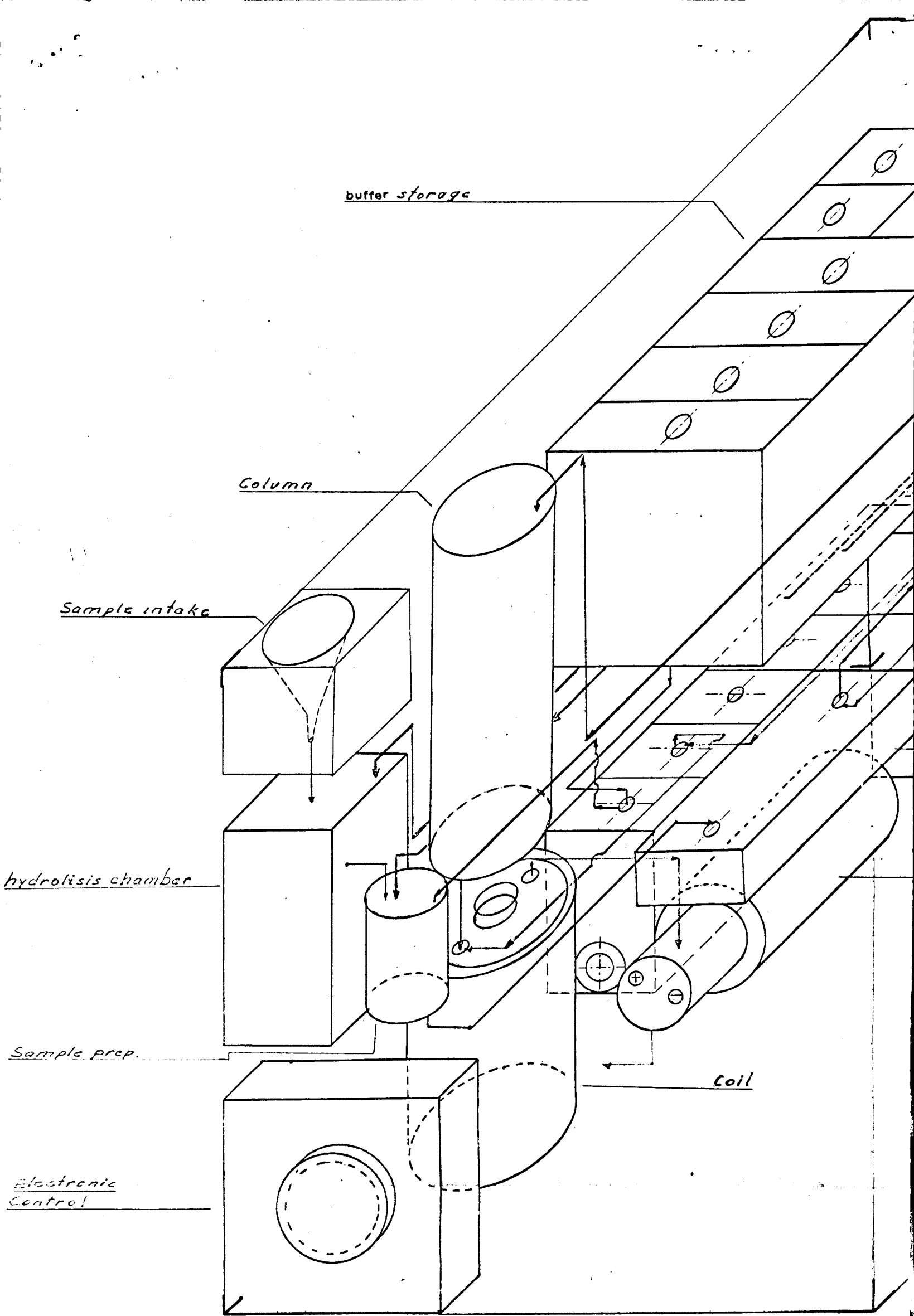
### 3. Micro Model

Making extensive use of our experience in the construction of the semi-micro model we have completed the design of most of the component parts of the micro model. A schematic diagram indicating the overall outlay and the functional arrangement of these components within the confinements of  $\frac{1}{2}$  ft<sup>3</sup> is shown in the figure. The individual weights have been calculated for the components. It appears that the electronics components will be less than 3 pounds, and the mechanical devices will be about 7 pounds. These components are presently under construction.

  
Stanley L. Miller

  
Joseph Kraut

  
Karl M. Dus



buffer storage

Column

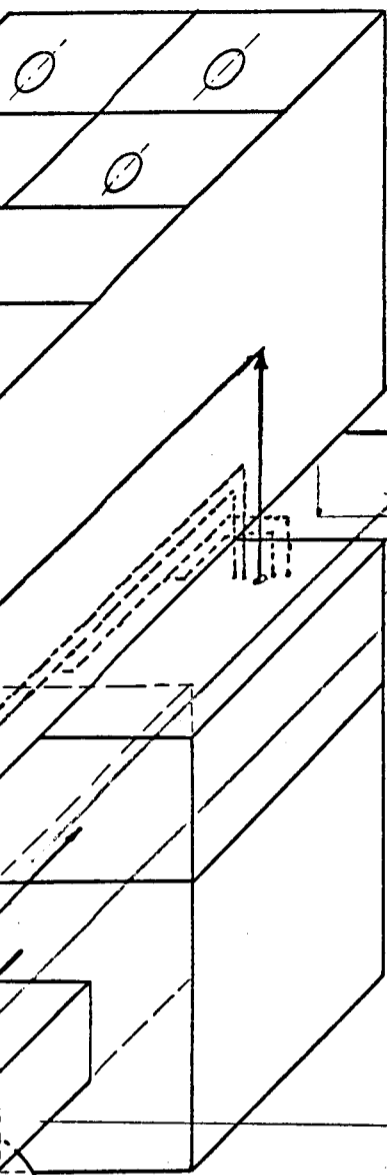
Sample intake

hydrolysis chamber

Sample prep.

Electronic Control

coil



buffer valves

evaporator

detector

- HCl
- hydrolysed
- Sample prep.
- buffer dil.
- Nitr.
- Water
- ..... 1' buffer
- ..... 2' buffer
- ..... 3' buffer
- ..... 4' buffer
- ..... Ammonia

Vol. : 576 in<sup>3</sup>

Scale 1/2 : 1 in