Administrative Coordinator Research Property Management Accounting Office Procurement Office

Reports Coordinator (OCA) Legal Services (OCA) Library, Technical Reports EES Information Office (2)
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FORM OCA 69,285

SPONSORED PROJECT TERMINATION/CLOSEOUT SHEET

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GEORGIA INSTITUTE OF TECHNOLOGY ATLANTA. GEORGIA 30332

CHEMISTRY June 18, 1981

Dr. A. W. Garrison Analytical Chemistry Branch Environmental Research Laboratory U. S. Environmental Protection Agency Athens, GA 30605

> RE: EPA Cooperative Agreement CR-808565-01 Letter Report 23 March - 22 June 1981

Dear Dr. Garrison:

This letter and attached figure comprise the first quarterly report on our cooperative agreement. This period was spent primarily in ordering and designing equipment and otherwise starting up the project. Thus there are no experimental results to discuss.

One of the research assistant positions has been filled with the appointment of Patricia Flaquer. Patricia is a first-year graduate student who received her BS in Chemistry degree from Florida State University. During this quarter she has been studying assembly language programming for the microcomputer system and has become familiar with the programming developed on the PDP8e for the prototype system. She is ready now to start program development using a microcomputer belonging to a colleague. I will also be heavily engaged in this work during the 2nd quarter.

The contract with the consultant for computer programming has been sent to the consultant, Mr. Harvey Mabry. It should be finalized shortly.

The major progress on the project has been accomplished by Mr. O'Brien. He has completed the circuit design for the microcomputer and has completed the layout of the main computer board. It will be ready for photo reduction and board etching by the end of June. We anticipate having the board ready for mounting of electronic components by the 1st of September, approximately a month ahead of the work schedule in the proposal.

An HPLC electrochemical flow cell has been procurred from PAR and we are in the process of formalizing a verbal agreement to purchase a used PAR 303 Stationary Drop Electrode. This electrode is in addition to the one to be incorporated into the proposed detector system. It will be used with the prototype system and the PDP8e to allow us to proceed with our electrochemical studies of compounds of interest in various solvent mixtures and supporting electrolytes.

Dr. A. W. Garrison June 18, 1981 Page 2

During this quarter, I attended the Southeastern Association of Analytical Chemists which met in Charleston, SC. I also attended the Eleventh Annual Symposium on the Analytical Chemistry of Pollutants which met at Jekyll Island, GA. Finally I spent a half day at the Environmental Research Laboratory discussing various aspects of this project.

Sincerely,

Peter E. Sturrock Professor of Chemistry

PES:gt Enclosure

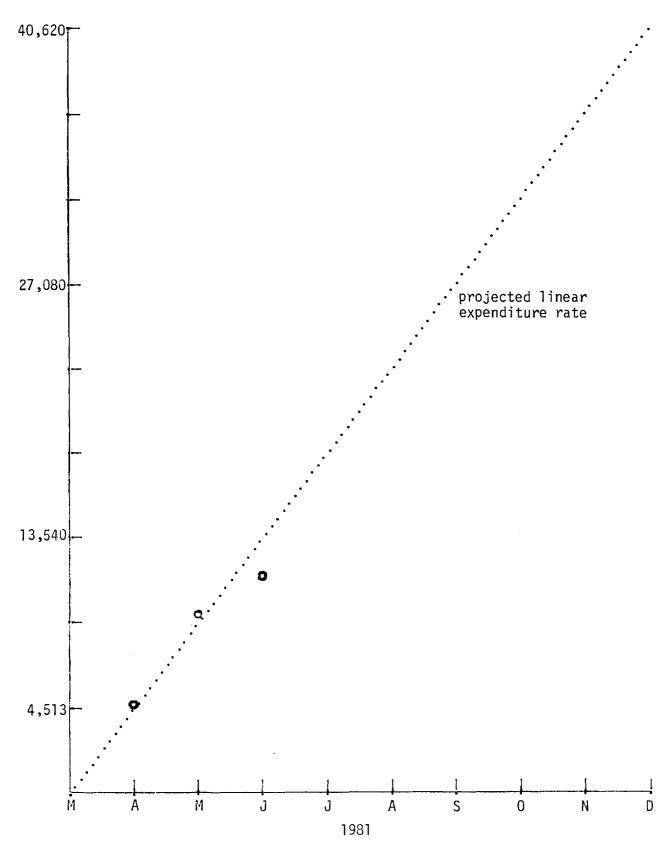


Figure 1. Expenditure Rate for Budget Period; CR 808565-01

PROGRESS REPORT

6 3 5 477

EPA Cooperative Agreement CR-808565-01 23 March 81 through 31 July 81

The first quarterly report (23 March - 22 June 1981) is attached to this report which therefore will stress activities since 22 June 1981.

The second research assistantship has been assigned to Mr. Li-qun Zhang who is presently completing his MS degree in electrochemistry at the University of Peking. Mr. Zhang is expected to begin work on this project about the 23rd of September.

Work on the microcomputer system is progressing nicely. Almost all components are now at hand and the main printed-circuit board is expected in September. At present, the circuit board for the CRT terminal is being laid out.

We have been very fortunate to have available a Z-80 microcomputer system belonging to a colleague. The research assistant is spending almost full time in program development using this microcomputer and the line printer purchased with funds from this project.

The principal investigator has modified the existing square-wave voltammetry program on the PDP8/e so that each sweep is automatically stored on a floppy disk as a separate file. The program is limited to 100 sweeps, but that should be enough to evaluate the flow cells, chemical systems, and column procedures. So far it has been used to test the reproducibility of drops produced by the PARC 303 electrode. Using a 1 x 10⁻⁵ M solution of Cd(NO₃)₂ in 1 M KNO₃, it was found that the voltammetric peak heights for 20 voltammograms on 20 successive electrode drops had a relative standard deviation of less than 1% for each of the 3 drop sizes available with the PARC 303. Additional work is underway on a modification of the valve seat of the PARC 303 to minimize problems of entrapment of air bubbles and high resistance in the electrode assembly. Initial results have been very encouraging with resistance values consistently in the six to eight-ohm region.

The computer consultant has modified the OS/8 driver program for a line printer so that it is compatible with the IDS-460 printer purchased for the microcomputer. The interface card for this purpose has been borrowed from ERL Athens. Now listings of new versions of the voltammetry program can be printed in about 40 minutes instead of over 4 hours as was necessary with the old Teletype.

The consultant is now modifying the operating system for the microcomputer to allow simultaneous disk dumps and data acquisition. This will utilize the two direct memory access channels which will be available in the hardware.

GEORGIA INSTITUTE OF TECHNOLOGY ATLANTA. GEORGIA 30332

CHEMISTRY

June 18, 1981

Dr. A. W. Garrison
Analytical Chemistry Branch
Environmental Research Laboratory
U. S. Environmental Protection
Agency
Athens, GA 30605

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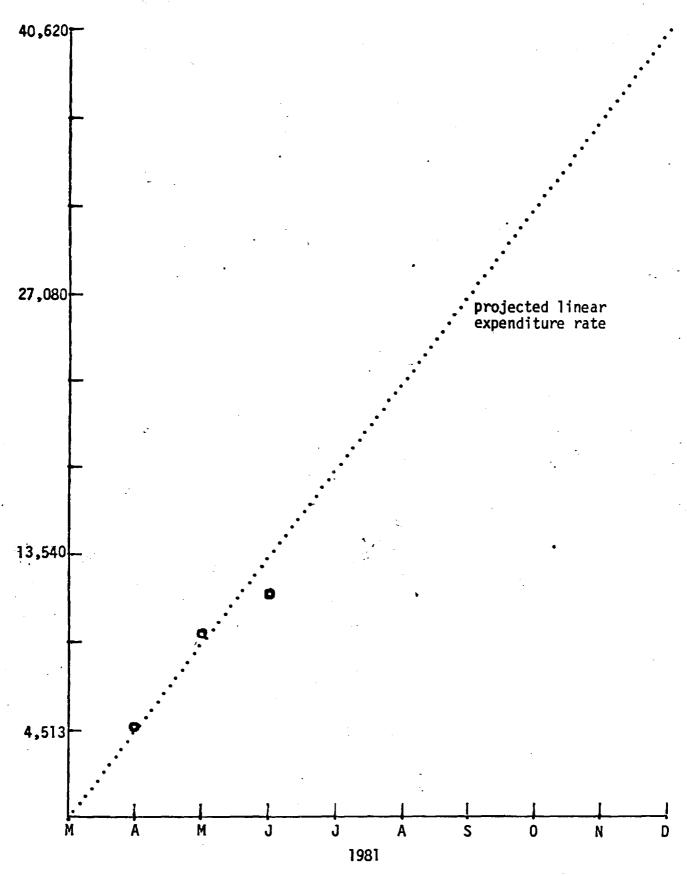


Figure 1. Expenditure Rate for Budget Period; CR 808565-01

EPA GRANT NO. CR 808565-01-0

5.7

The following pertains to your CURRENT budget period (Federal and non-Federal). This page must be completed and submitted with your continuation application.

FROM 3/	23/81	THROUGH	12/22/81	NUMBI	STANCE ER	
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Date		

SUMMARY REPORT OF INVENTIONS AND SUBAGREEMENTS OR SUBCONTRACTS

The following report must be submitted in *triplicate* as part of the interim or final report as provided for by the patent clause in the grant or contract.

Name	of Grantee or Contractor	Address
Georgia Tech R	esearch Institute	225 North Ave., N. W. Atlanta, Georgia 30332
Grant or Contract No.	CR-808565-01	
(Check appropriate	boxes)	
1. Type of Report:		
(F	om_23 March	<u>_, 1981</u>
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Final.		
2. Interim Report D	ata:	
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Inventi	on Disclosures attached h	erewith. Give Grantee or Contractor's docket numbers.
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3. Final Report Data:	Date Submitted	Grantee or Con Docket Nu		
A. Invention(s) previously				
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B. Invention(s) reported				
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C. Others (explain)-				
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D. No inventions were made u	inder the grant or cont	ract.		
4. Patent application(s) filed and or contract:	contemplated to be fil	ed by the Grante	e or Contractor unde	er the terms of the gra
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Date of filing Contractor's Docket No.				
5. Subagreements or Subcontracts	containing patent righ	ts clause:		
X None. Listed below	are subcontractors.			
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б. Attach a copy of the patent righ	nts clause employed in	each subagreem	ent or subcontract s	et forth in 5
o. Attach a copy of the patent rigi	nts crause emproyeu m	cach subagreem	ent of subcontract s	et forth in 5.
7. Grantee or Contractor certificat	ion.			
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U.S. ENVIRONMENTAL PROTECTION AGENCY

Form Approved
OMB No. 158-R0081

PROJECT NO. (Do not use this space)

SSIE

NOTICE OF RESEARCH PROJECT

PREPARED FOR THE SMITHSONIAN SCIENCE INFORMATION EXCHANGE

TITLE OF PROJECT

Electrochemical Detector for High Performance Liquid Chromatography

GIVE NAMES, DEPARTMENTS, AND OFFICIAL TITLES OF PRINCIPAL INVESTIGATORS OR PROJECT DIRECTORS AND ALL OTHER PROFESSIONAL PERSONNEL ENGAGED IN THE PROJECT.

School of Chemistry, Georgia Institute of Technology Principal Investigator - Dr. Peter E. Sturrock, Professor of Chemistry Research Scientist - Mr. Gerald O'Brien

NAME AND ADDRESS OF APPLICANT INSTITUTION

225 North Avenue Atlanta, GA 30332

SUMMARY OF PROPOSED WORK - (1) Objectives, (2) Approach, (3) Current Plans and/or Progress (200 words or less. Omit confidential data), in the Smithsonian Science information Exchange, summaries of work in progress are exchanged with government and private agencies supporting evaluate and are forwarded to investigators who request such information. Your summary is to be used for those purposes.

This is a continuation of a project initiated in March 1981 to develop a complete electrochemical detector system for high performance liquid chromatography. The complete system will include cells, potentiostat, and microcomputer system (hardware and software). The primary mode of operation will use rapid sweep square-wave voltammetry with repeat voltage scans once a second. Data will be stored on magnetic disk and after each chromatographic run, a plot of response versus potential and retention time will be displayed. This swept mode of operation will allow detection of two or more components not completely separated by HPLC column and also will minimize the amount of time required to develop a procedure for a new application since exact potentials do not need to be known in advance.

The systems will be applied to determination of pollutants in water. Test systems will include nitrosamines, nitrophenols and halogenated compounds.

	HTIFY PROFESSI	OHAL SC	HOOL IN	/OLVED (Nedical,	SIGNATURE OF	PRINCIPAL INVESTIGATOR	DATE
	Chemistry						9 Sept 1981
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INSTRUCTIONS

Please type or print legibly. Items 1, 2, 3, 6, 7, 9, 10d, 10e, 10g, 10i, 10l, 11a, and 12 are self-explanatory, specific instructions for other items are as follows:

Item

4 Efter the employer identification number assigned by the U.S. Internal Revenue Service or FICE (institution) code, if required by the Federal sponsoring agency.

10c Enter the amount of all program this period that is required by the federal sponsoring agency.

5 This space is reserved for an account number or other identifying numbers that may be assigned by the recipient.

Entry

Item

- 8 Enter the month, day, and year of the beginning and ending of this project period. For formula grants that are not awarded on a project basis, show the grant period.
- The purpose of vertical columns (a) through (f) is to provide financial data for each program, function, and activity in the budget as approved by the Federal sponsoring agency. If additional columns are needed, use as many additional forms as needed and indicate page number in space provided in upper right; however, the totals of all programs, functions or activities should be shown in column (g) of the first page. For agreements pertaining to several Catalog of Federal Domestic Assistance programs that do not require a further functional or activity classification breakdown, enter under columns (a) through (f) the title of the program. For grants or other assistance agreements containing multiple programs where one or more programs require a further breakdown by function or activity, use a separate form for each program showing the applicable functions or activities in the separate columns. For grants or other assistance agreements containing several functions or activities which are funded from several programs, prepare a separate form for each activity or function when requested by the Federal sponsoring agency.
- 10a Enter the net outlay. This amount should be the same as the amount reported in Line 10e of the last report. If there has been an adjustment to the amount shown previously, please attach explanation. Show zero if this is the initial report.
- 10b Enter the total gross program outlays (less rebates, refunds, and other discounts) for this report period, including disbursements of cash realized as program income. For reports that are prepared on a cash basis, outlays are the sum of actual cash disbursements for goods and services, the amount of indirect expense charged, the value of in-kind contributions applied, and the amount of cash advances and payments made to contractors and subgrantees. For reports prepared on an accrued expenditure basis, outlays are the sum of actual cash disbursements, the amount of indirect expense incurred, the value of inkind contributions applied, and the net increase (or decrease) in the amounts owed by the recipient for goods and other property received and for services performed by employees, contractors, subgrantees, and other payees.

10c Enter the amount of all program Income realized in this period that is required by the terms and conditions of the Federal award to be deducted from total project costs. For reports prepared on a cash basis, enter the amount of cash income received during the reporting period. For reports prepared on an accrual basis, enter the amount of income earned since the beginning of the reporting period. When the terms or conditions allow program income to be added to the total award, explain in remarks, the source, amount and disposition of the income.

Entry

- 10f Enter amount pertaining to the non-Federal share of program outlays included in the amount on line e.
- 10h Enter total amount of unliquidated obligations for this project or program, including unliquidated obligations to subgrantees and contractors. Unliquidated obligations are:

Cash basis-obligations incurred but not paid;

Accrued expenditure basis—obligations incurred but for which an outlay has not been recorded.

Do not include any amounts that have been included on lines a through g. On the final report, line h should have a zero balance.

- 10j Enter the Federal share of unliquidated obligations shown on line h. The amount shown on this line should be the difference between the amounts on lines h and i.
- 10k Enter the sum of the amounts shown on lines g and j. If the report is final the report should not contain any unliquidated obligations.
- 10m Enter the unobligated balance of Federal funds. This amount should be the difference between lines k and l.
- 11b Enter rate in effect during the reporting period.
- 11c Enter amount of the base to which the rate was applied.
- 11d Enter total amount of indirect cost charged during the report period.
- 11e Enter amount of the Federal share charged during the report period.

If more than one rate was applied during the project period, include a separate schedule showing bases against which the indirect cost rates were applied, the respective indirect rates the month, day, and year the indirect rates were in effect, amounts of indirect expense charged to the project, and the Federal share of indirect expense charged to the project to date.

FINANCIAL STATUS REPORT (Follow instructions on the back) 3. RECIPIENT ORGANIZATION (Nouns and complete address, fustuating EIP code) Georgia Tech Research Institute Atlanta Commission 20222		Environmental Protection Agency CR808565-01-0			0	YIND PAGE OF		
		58-0603146 G-33-677		SER OR IDENTIFYING NUMBER 6. FINAL REPORT YES XX NO 9. PERIOD COVERED BY THIS REP		7. BASIS CASH ACCRUAL		
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m. Un	obligated balance of Federal funds	11,200.00	1,324.32	1,722.34	-0-	5,455.89	11,789.50	31,492.05
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SYANDARD FORM 269 (7-76) Prescribed by Office of Management and Budge Cir. No. A-110

GEORGIA INSTITUTE OF TECHNOLOGY

ATLANTA GEORGIA 30332

CHEMISTRY

December 22, 1981

Dr. Walter Shackelford Analytical Chemistry Branch Environmental Research Laboratory U. S. Environmental Protection Agency Athens, GA 30605

> RE: EPA Cooperative Agreement CR-808565-01 Annual Report 23 March - 22 December 1981

Dear Dr. Shackelford:

This letter and attached figure covers the entire budget period of nine months (23 March - 22 December 1981). At this point the project is essentially on schedule: some aspects are a little ahead of the original work schedule while others are a little behind. The general picture is like the scene of a building construction project: some features are clearly recognizable and well defined while much appears to be a jumble of parts and supplies. In the paragraphs below are discussions of personnel, computer hardware, computer software, and other equipment.

The principal investigator has spent over one-fourth of his time during the spring and fall quarters and over two months full time in the summer quarter on this project. Besides overall direction and administration, he has spent a great deal of time modifying and evaluating the PAR 303 electrode stand to obtain reproducible results and low resistance essential for operation of the proposed electrochemical detector in the pulse mode. Modified stands are in regular use at Georgia Tech and at the Georgia State Crime Laboratory in Atlanta.

The principal investigator has been invited to serve as chairman of one of the sessions of the Heyrovsky Discussions at Castle Liblice in Czechoslovakia in May 1982.

The electronics research scientist has designed the microcomputer system, the graphics terminal, and has just completed the design of the potentiostat. The electronics technician has completed construction of the microcomputer system.

One of the graduate research assistant slots has been filled for all three quarters by Patricia Flaquer, a second year graduate student in Analytical Chemistry. She has worked primarily on programming, especially the assembly language programming for the graphics terminal. The second research assistantship was scheduled to be filled starting

the third quarter. The position was offered to and held for Li-qun Zhang who has just completed his MS degree in electrochemistry at the University of Peking, PRC. Unfortunately, Mr. Zhang has not yet received his passport and visa and thus has been unable to come. It is hoped that he will start on the project in January 1982.

Dr. George Robinson, a visiting professor in the School of Chemistry for 1981/82, has started to work on the project. He has considerable experience with HPLC and has taken responsibility for ordering the components and putting together the modular HPLC system. He will be of increasing value to the project during the next two quarters.

The Z-80 based microcomputer system has been designed and constructed. However, it has not yet been tested and debugged. This will be done by the research scientist, Gerald O'Brien, and he has been working almost full time on the design of the potentiosat. The Z-80 based graphics terminal has been designed and all components are on hand. It (actually two of them) will be constructed by the electronics technician in January 1982.

The design of the potentiostat is now complete and the components are on order. The design of this unit has undergone considerable revision from the original concepts of the proposal and now entails more intimate interaction with the computer. Initially the potentiostat will be operated directly by the microcomputer system. Later, a slaved Z-80 microprocessor will be inserted between the potentiostat and the main microcomputer system. The slaved Z-80 will handle routine real-time control chores under instruction from the main microcomputer. The result will be faster throughput of data since the main microprocessor will be able to calculate, display, and store the incoming data without the numerous interupts generated by the real-time experiment.

Two IDS model 460G printer/plotters have been purchased. One will be used with the microcomputer system and is presently being used with the older model Z-80 system borrowed from a colleague. The other IDS 460G is in use with the DEC Lab PDP 8/e that controls the prototype electrochemical system. The OS/8 operating system has been modified to operate the printer.

The computer programming has been progressing well although it has not been possible to test it throughly since the computer hardware is not operational. The operating system for the Z-80 has been upgraded from that used with the earlier Z-80 systems constructed in the School of Chemistry. One of the important changes here is the ability to operate with dual DMA channels so that data can be brought in on one channel while data files are being dumped to a floppy disk through the other DMA channel.

The software for the graphics terminal is almost complete, but also untested. The software includes several modes of operation: alpha-numeric, graphics, and alpha-graphics. The unit will include most of the capability of a Tektronics 4010 terminal. In the graphics

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mode, as each data point is received from the main microcomputer, the Z-80 within the graphics terminal calculates the intermediate points to connect the new point to the previous point by a straight line and then displays the new line as well as continuously refreshing the entire display.

Portions of the programming for the potentiostat control and data acquisition have been written. This task has had to wait until the hardware design of the potentiostat was decided. Now, however, it can proceed.

While failure to fill the second research assistantship has delayed experimental evaluation of electrochemical parameters and the flow-cell operation of the PAR 303, it has allowed the transfer of funds which enabled the acquisition of HPLC equipment ahead of the original schedule. Thus a Haskell pneumatic-amplifier pump and a Valco UCLI injection system have been purchased and an IBM glass wall-jet flow cell has been ordered. These, together with the UV and RI detectors and strip-chart recorder already available, comprise a complete HPLC system. Empty columns, slurry packer, and packing material are also on hand. In January 1982 the system will be assembled and tested. Then evaluation of flow cells and a second pump can begin.

In summary, the nine-month gestation period is over and we are eagerly looking forward to the birth of the electrochemical detector system.

Sincerely,

Peter E. Sturrock Professor of Chemistry

PES:gt

Attachment

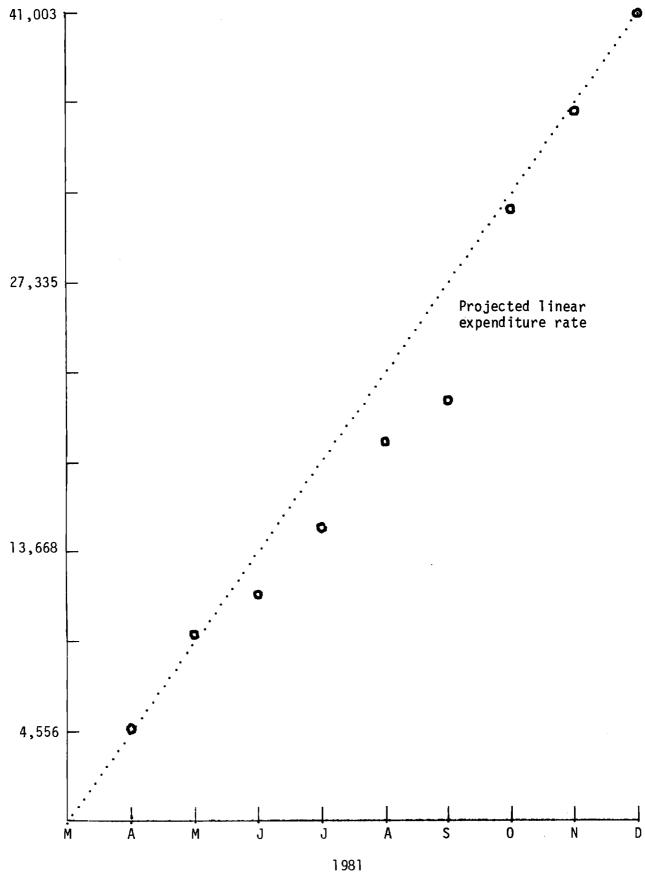


Figure 1. Expenditure Rate for Budget Period; CR-80856S-01