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This document was reviewed following the procedures described in WHC-CM-3-4 and is:

APPROVED FOR PUBLIC RELEASE

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WHC Information Release Administration Specialist:

Kara Broz — 08-22-94

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SUPPORTING DOCUMENT

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7. Abstract

Corrosive plutonium solutions stored in 10-L containers at the Plutonium Finishing Plant must be treated to convert the plutonium to a safe, solid form for storage and to remove the americium so that radiation exposure can be reduced. Extraction chromatographic resins will be tested for separating plutonium from these solutions in the laboratory. Separation parameters will be developed during the testing for large scale processing of the 10-L solutions and solutions of similar composition. Use of chromatographic resins will allow plutonium separation with minimum of chemical addition to the feed and without the need for plutonium valence adjustment. The separated plutonium will be calcined to plutonium oxide by direct solution calcination.

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TEST PLAN FOR DEMONSTRATING PLUTONIUM EXTRACTION FROM 10-L SOLUTIONS USING EICHROM EXTRACTION CHROMATOGRAPHIC RESINS

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TEST PLAN FOR DEMONSTRATING PLUTONIUM EXTRACTION FROM 10-L SOLUTIONS USING EICHROM EXTRACTION CHROMATOGRAPHIC RESINS

G. S. Barney

1.0 INTRODUCTION

The Plutonium Finishing Plant (PFP) received plutonium-bearing solutions from the Argonne National Laboratory in 1972 for scrap processing . These solutions are highly acidic and are considered reactive and corrosive. The 27 solutions (207 liters total volume) contain a variety of acids including nitric, hydrochloric, hydrofluoric, sulfuric, and perchloric acids. They have been stored in 10-L containers which have deteriorated over this storage period. In order to store the plutonium in these solutions safely it is necessary to convert the plutonium to a solid form. The purpose of this testing is to develop the separation parameters required for large-scale processing of these (and similar) solutions.

Extraction chromatographic resins are used to selectively remove plutonium and other actinide elements from almost any acidic waste solution. This separation technique removes the reactive components from plutonium waste so that the separated plutonium can be solidified into a stable chemical form for long-term storage. The resin extracts plutonium from waste solutions as they are pumped through a resin bed. The concentrated plutonium is then removed from the resin by eluting with a solution containing a complexant such as citrate. The resulting plutonium solution can be solidified by calcination or precipitation of the plutonium from solution.

Extraction chromatographic resins combine the selectivity of solvent extraction processes with the simplicity of a column chromatographic system. While conventional solvent extraction is a liquid-liquid system, extraction chromatographic resins perform the same separations in a solid-liquid system. This allows use of much simpler equipment, easier material handling, and reduced capital cost in construction of process equipment.

The resins used in extraction chromatography are prepared by adsorption of a conventional or slightly modified extractant on a macroporous polymeric support. This preparation process immobilizes the extractant while retaining the chemical properties (distribution ratios, selectivities, etc.) of the original liquid solvent system. Thus, extractants known to selectively extract metal ions in a solvent extraction process (i.e. CMPO-TBP mixtures used in the TRUEX process for extraction of TRU elements) can be immobilized on a solid support and will retain their selectivity and extraction capacity.

2.0 OBJECTIVE

The primary objective of these tests is to evaluate extraction chromatographic resins for separating plutonium from solutions stored in 10-L containers at the Plutonium Finishing Plant. Plutonium must be removed to levels below the TRU limit (100 nCi/g) for the resins to be considered successful. The waste effluent from the resin columns must also meet the requirements for disposal in high-level waste tanks. Removal of plutonium, americium, and uranium will be studied. The performance of two EIChrom resins (EIChrom Industries, Inc.) and one laboratory-prepared resin will be evaluated. The various resins must be applied in a logical sequence to achieve optimum performance of the actinide removal system. This sequence will be determined from laboratory extraction parameters to be determined in these tests.

The tests will yield several types of extraction parameters. Distribution coefficients (Kd's) for actinides will be determined under a range of conditions (acid concentration, waste composition, actinide concentration, and resin type) expected in the actinide removal system. These batch equilibrium tests will be used for setting experimental parameters for the column tests. Column testing will provide elution data that will be used to predict extraction rates for dissolved actinides, capacities, and regeneration efficiencies.

3. SCOPE

All tests will be performed on a laboratory scale. Both batch equilibration tests and flow-through column tests will be performed. The batch tests will use several grams of resin and less than 20 mLs of 10-L acidic waste solution with a range of compositions. A number of these batch tests will be completed to cover the required range of conditions of acid concentration, waste composition, actinide concentration, and resin type. Laboratory columns filled with resins will have a bed volume of about 50 mLs. Several columns of each resin will be prepared. Volumes of wastes to be pumped through these columns will be up to about four liters.

Interfaces with laboratory coworkers conducting other testing with the 10-L solutions (plutonium separations, precipitation, and calcination), laboratories performing TRUEX testing, and other waste partitioning activities will be important. Exchange of information with personnel performing these tests will be continued during these studies. Another important interface is with the operations group which sets overall goals for plutonium separation and waste disposal.

4. DESCRIPTION OF TEST

4.1 Test Item

Extraction chromatographic resins to be tested were chosen for their known effectiveness in removing specific actinides from 10-L solutions. Specific removal of actinides is important so that the capacity of the resins is not used up by non-radioactive components of the wastes. It is also important that the actinides have high distribution coefficients for extraction onto the resins since it is important to concentrate them. A few commercially-prepared resins are available for this type of application. The resins to be tested and their probable applications are given in Table 1.

TABLE 1

RESINS TO BE TESTED FOR REMOVAL OF RADIONUCLIDES FROM ACIDIC WASTES

Resin	Type	Manufacturer	Applicable Actinides
TRU•Spec	CMPO + TBP extractant on a macroporous polymeric support	EIChrom Industries, Inc.	Pu(IV), Pu(VI), U(VI), Am(III)
U/TEVA•Spec	Dipentyl Pentylphosphonate extractant on a macroporous polymeric support	EIChrom Industries, Inc.	Pu(IV), U(VI),
CMPO-Impregnated Resin	CMPO extractant on a macroporous polymeric support	Laboratory-prepared	Pu(IV), Pu(VI), U(VI), Am(III)

Two resins manufactured by EIChrom Industries, Inc. will be tested as well as a laboratory-prepared resin. TRU•Spec resin is commercially available from EIChrom Industries, Inc. (Darien, Illinois) and is specifically designed to extract transuranic elements in (III), (IV), and (VI) oxidation states from acidic solutions. It contains an impregnated extractant, octyl(phenyl)-N,N-diisobutylcarbamoylmethylphosphine oxide (CMPO) dissolved in tributyl phosphate (TBP), on a polymeric resin substrate. Most other metal ions do not interfere with the extraction. Zirconium and some of the rare earth elements (i.e. La, Ce, Pr, Nd, Sm, and Eu) are extracted by this resin, but are not expected to be present in significant concentrations in the 10-L solutions. Dissolved metallic elements that make up the major portion of the solutions (i.e. Na, Al, Fe, Cr, Mg, and Ca) should not be extracted, according to EIChrom literature. The resin can be regenerated by eluting actinide elements

with dilute acid or with complexing agents. The useful loading capacity of this resin for plutonium is about 1 to 2 grams of plutonium/L of resin bed. The particle size range of resin beads to be tested is 100 to 125μ.

The U/TEVA·Spec resin is an extraction chromatographic resin for the selective sorption of uranium and tetravalent actinides. It consists of a liquid extractant, dipentyl pentylphosphonate, adsorbed onto macroporous polymeric resin beads. The resin is an acrylic ester polymeric adsorbent. Unlike the TRU·Spec resin, this material will not extract americium. A separation of americium from other actinides can therefore be devised using these two resins. Very few metal ions interfere with the extraction of uranium and tetravalent actinides. Zirconium and ruthenium are extracted to a relatively small extent, but should not significantly affect actinide extraction. The resin can be regenerated by eluting the actinides with dilute nitric acid. It has a useful loading capacity of about 4 to 8 grams of actinide/L of resin bed. The particle size range of resin beads to be tested is 100 to 125μ.

In addition to the EIChrom resins, a resin prepared in our laboratory will be tested. It was prepared by loading liquid CMPO onto macroporous resin beads (Amberlite XAD-16) from a methanol solution (Barney and Cowan, 1992). The resulting material is about 50% by weight CMPO and appears to extract americium more strongly than the TRU·Spec EIChrom resin. It should have similar selectivity to the TRU·Spec resin since the same extractant was used (except without the TBP solvent).

The 10-L solutions have not been well characterized for solution composition. Only plutonium and uranium concentrations have been reported and general descriptions of the acids that are present. These solutions shall be characterized thoroughly before the separations work with EIChrom resins can begin.

The proposed testing will treat widely different compositions of the 10-L solutions that are available. This will allow a determination of the general applicability of the extraction chromatographic method to different feed compositions. Depending on blend feasibility five different 10-L solutions will be tested that contain the following combinations of components: (1) 6 M HCl and HNO_3, (2) HCl, HNO_3, and HF, (3) HCl, HNO_3, HF, and U, (4) 1 M HCl, and (5) HCl. HNO_3, $HClO_4$, and H_2SO_4. About four liters of each solution (previously filtered through a membrane with a pore size of 0.3 μm) would be passed through columns of resin at a constant flow rate. The plutonium and americium concentration in the column effluent will be monitored by sampling it periodically and counting the plutonium with a liquid scintillation analyzer. The volume of the column resin beds will be about 50 mLs and the flow rate will be about 15 mLs/min.

After the columns are loaded with plutonium, they will be eluted with an ammonium citrate solution at pH 4. Again, the effluent will be periodically monitored for plutonium concentration to determine the required volume of

eluant. The eluted solution will be directly calcined to produce the stabilized plutonium oxide product.

The information to be obtained in the tests will be (1) loading capacity of the resins, (2) effects of waste composition on the plutonium loading curves, (3) loading and regeneration efficiency of the resins, (4) required volume of stripping solution, and (5) concentrations of reactive impurities in the stabilized PuO_2 product and product density. These data will allow evaluation of the method and provide information for scale-up of the process.

4.2 Test environment

The testing will be performed in PPSL laboratory rooms equipped with open-faced hoods for handling the blended 10-L solutions. Solution preparation, extraction chromatographic experiments, and counting of the solutions containing alpha emitters (plutonium, americium, and uranium) will be performed in Rooms 191, 187 (Hoods 1 to 4), and 183 of the PPSL Laboratory. Samples generated from the testing will be sealed and counted using a liquid scintillation analyzer in Room 183. Non-radioactive solutions, such as the eluant will be prepared in the cold lab (Room 191 of 234-5 Building).

4.3 Equipment and Facilities

At least two open-faced hoods in the PPSL must be available for preparing 10-L solutions and performing the extraction chromatographic experiments. Space in the cold labs and in the counting room will also be used. Appropriate alpha survey equipment must available for contamination control. Applicable Radiation Work Permits for the laboratory rooms used in this work must be approved and followed. Current RWP's do not need revision because of this study.

Equipment required for these tests includes apparatus for batch equilibration, column tests and radioactive analysis. The batch measurements will be performed using 20 ml glass bottles with screw caps to contain the resin-solution mixtures. These bottles will be shaken with a variable speed flat bed shaker to speed equilibration. The solutions will be sampled periodically and the actinide concentrations measured to determine the required equilibration time. Chromatography columns (Spectrum Medical Industries, Inc.) will be used to contain the resin in the column tests. The resin bed dimensions can be adjusted with these columns. Precision metering pumps (Eldex Laboratories, Inc., model E-120-S) will be used to pump solutions through the columns at a known, constant rate. Effluents from the columns will be sampled automatically using a fraction collector (Haake Buchler Instruments, Inc., model LC 200) that will collect constant volume fractions. Actinide concentrations will be measured using liquid scintillation counting (Packard 1500 Tri-Carb liquid scintillation analyzer). The required laboratory equipment for these tests already exists in the PPSL.

The extraction chromatographic resins to be tested are listed in Table 1.

The five 10-L solutions to be tested will be obtained from PFP Operations after the original 10-L solutions have been blended together in batches. Four liters of each solution will be required for the testing.

4.4 Data

The important experimental conditions for batch equilibrium measurements of radionuclide distribution include actinide concentrations (plutonium, uranium and americium), solution chemical composition, resin type, and acid concentration. In addition, since actinide elution will be important, the types of solutions used as elutants and their concentration are important. For the column experiments, flow rate and bed volume are important experimental parameters, in addition to those given above. The ranges of experimental parameters for each type of measurement are given in Table 2.

TABLE 2

EXPERIMENTAL PARAMETERS AND RANGES

Measurement Type	Parameter	Range
Batch Equilibration (Distribution Coefficients)	Actinide Type	Plutonium, Uranium, Americium
	Actinide Concentration	1 to 20 g/L
	Waste Solution Chemical Composition	HCl, HNO_3, H_2SO_4, HF, $HClO_4$, Metal Salts
	Resin Type	See Table 1
	Acid Concentration	1.0 to 6.0 M
Column Tests (Adsorption Rates, Capacity)	Above Parameters	Above Ranges
	Flow Rate	0 to 15 ml/min
	Bed Volume	1 to 50 mls

The experimental concentration ranges are based on estimates of waste solution composition which is the feed for the adsorption columns. Actinide concentrations will cover the range of expected concentrations in the solutions.

4.5 Criteria/Constraints

The criteria for successful application of this testing are (1) that plutonium separation from 10-L solutions using extraction chromatographic

resins is essentially complete, leaving the reactive components of these solutions in the waste along with plutonium concentrations below the TRU limit of 100 nCi/g, (2) the plutonium is concentrated by at least a factor of two before calcination, and (3) the waste will be acceptable for disposal in high-level waste tanks. The separated plutonium will be in a form that can easily be calcined to plutonium oxide. Data generated in these tests will allow prediction of the performance of the resins in large-scale process application.

Some constraints on these tests are as follows:

(1) The chemical compositions of these solutions are not known with a great amount of confidence. Obviously, before any chemical separations are attempted, the solutions shall be analyzed for cations and anions. Adjustment of the feed solution composition will be required in some cases for the separation to be successful. An example is when hydrofluoric acid is present. Aluminum must be added to prevent the formation of plutonium fluoride complexes which are not extractable.

(2) Working volumes of 10-L solutions will be limited due to the 15 g plutonium criticality limits on the open-faced hoods. This may require storage of some of the solutions in hoods other than those used in the extraction chromatography tests.

5.0 EXPECTED RESULTS

The tests will likely show that several resins can be applied to removal of plutonium from 10-L solutions at the PFP. Based on available data from EIChrom, data from other laboratories (i.e. Los Alamos National Laboratory) performing work on extraction chromatographic resins, and on data obtained in this laboratory, commercially-available adsorbents can be found that will fulfill the requirements of plutonium removal and separation from reactive solution components. Sizing the treatment columns for the quantities of solutions to be processed will determine if a given resin is practical to use. The size of the columns will be estimated from expected solution flow rates, rates of extraction and extraction capacities of the resins measured in these tests. One of the most important unknown factors in these tests is the effect of various dissolved salts in the 10-L solutions. These salts may compete with plutonium for extraction on the resins and may reduce extraction capacity.

6.0 TEST PROCEDURE

Procedures for measuring actinide distribution coefficients (Kd values), extraction rates, and extraction capacity are summarized in the

following paragraphs. There are two general types of procedures to be used: batch equilibration measurements and column flow-through measurements.

In the batch equilibration measurements of radionuclide Kd values, a weighed amount of resin is equilibrated with a measured volume of solution containing known concentrations of actinide and non-radioactive components. The acid concentration will be adjusted with HNO_3 and measured after equilibration. The time required for equilibration is determined by sampling the solution periodically and measuring the actinide concentration. When a constant actinide concentration is reached, equilibrium has been attained. The concentration of actinide in the solution after equilibration is used to calculate the amount of actinide adsorbed on the resin. A Kd value is then calculated using this data.

For the column experiments, commercially-available liquid chromatography columns will be filled with the resin(s). Solutions containing known concentrations of actinide and inert components will be pumped through the columns at a constant, known flow rate. Samples of the effluents from the columns will be counted for actinide concentration. Breakthrough curves will be plotted to show actinide concentration in the effluent versus effluent volume. For extraction rate experiments, the flow rate will be varied and the effect of flow rate on the actinide concentration in the effluent will be evaluated. Resin capacities will be determined by measuring the volume passed through the column when the actinide is first observed in the effluent.

The size of the columns to be used will be determined by results of the batch equilibration measurements. Large distribution coefficients will require that smaller columns be used in order to reach the capacity of the adsorbent. Other considerations in determining column size are height to diameter ratio (must be large enough to prevent channeling), and distribution of the influent solution to the bed diameter.

The effects of acid concentration, actinide concentrations, and concentrations of interfering ions will be determined in both the batch and column tests by changing their values over the specified range of these parameters and measuring the effects on adsorption.

7.0 SAFETY

All standard laboratory safety measures will be observed. All radiation work procedures will be adhered to, as described in HSRCM-1, "Hanford Site Radiological Control Manual." The following specific permits will apply: Z-34, "Low Radiological Risk Activities in Radiological Controlled Areas (RCA's)," and Z-12, "Routine Work Inside Gloveboxes and Hoods in SCA's." All work with chemicals will follow the Westinghouse Hanford Company Chemical Hygiene Plan (WHC-SD-CP-HSP-001).

Wastes containing extraction chromatographic resins, inorganic adsorbents, synthetic wastewaters, and actinides (Pu. Am, and U), will be generated from these tests. The resins are not likely to be classified as hazardous wastes because of their relatively high flash point (165 °F), unreactiveness, and low toxicity. The organic resins will contact a weak oxidizer (dilute HNO_3) but will not be an explosive hazard. They will, however, generally be contaminated with radioactive tracers. The aqueous waste solutions will be neutralized with NaOH solution before disposal to neutral pH and will be radioactive. These solutions can be disposed of in the D-4 drain. PFP Environmental Compliance will approve the disposal of any of these wastes. Material Safety Data Sheets are available in the laboratory for all of the resins and reagents that will be used.

8.0 QUALITY ASSURANCE

These tests are assigned Approval Designator SQ, as per Section 12.7 of the WHC-3-5 Manual. The quality assurance implementation is under the direction of the cognizant chemist. All procedures performed by the PPSL will be handled in accordance with the Westinghouse Hanford Company manual "Laboratories Administration" WHC-CM-5-4. All work will be documented in laboratory notebooks according to Section 3.6 of the WHC-CM-5-4 manual.

All analyses will be performed according to written procedures and will be verified with standard samples before use. Other chemical analyses will be performed by Analytical Services which maintains a Laboratory Measurement Control System (LMCS). The LMCS provides for analysis of standards and replicate analyses to assure quality control.

9.0 ORGANIZATION AND FUNCTIONAL RESPONSIBILITIES

Organizational responsibilities are summarized in Table 3.

Table 3. Organizational Responsibilities

Organization	Responsibilities
Plutonium Process Support Laboratories	Test Planning
	Test Execution
	Data Analysis
	Documentation
PFP Process Engineering	Technical Consultation
	Approval
	Data Evaluation
Analytical Services	Sample Analysis
Standards Laboratory	Standards Preparation
PFP Safety/Quality Assurance	Review Test Plan And Reports

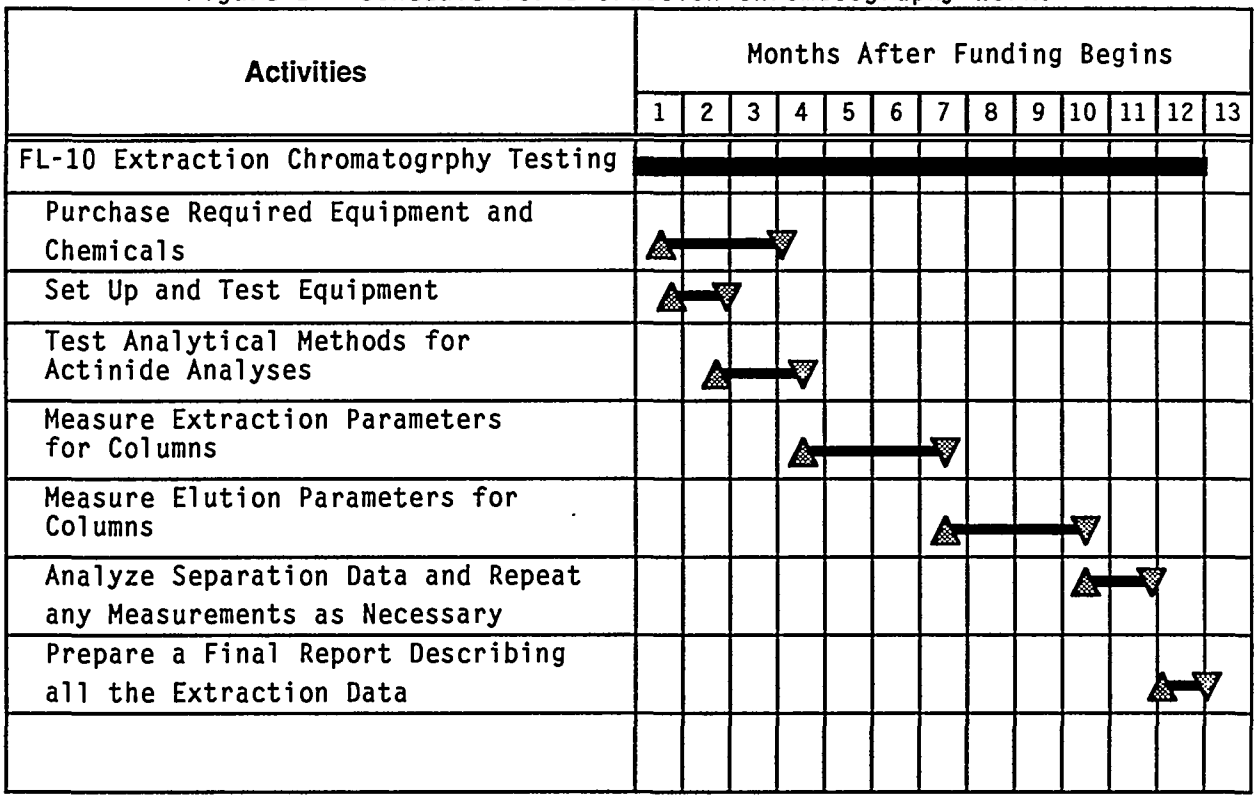
10.0 ESTIMATED SCHEDULE AND COSTS

A summary of the estimated costs is shown below:

Exempt	$40,000	
Nonexempt	$30,000	
Materials	$10,000	
Analyses	$10,000	(Analytical Services)
Total	$90,000	

Laboratory studies are scheduled to begin immediately after approval and release of this test plan and receipt of the 10-L test solutions. The work will continue through the end of FY1995, with a final test report due September 30, 1995. Details of the schedule are presented in Figure 1.

Figure 1 - Schedule for Extraction Chromatography Work.



11.0 REPORTS

Periodic status reports will be issued via the organizational weekly reports. Letter reports will be issued as tasks are completed. A summary report will be issued one year after work begins.

12.0 REFERENCES

1. WHC-CM-5-4, Rev. 0, Laboratory Administration, Westinghouse Hanford Company, Richland, Washington.

2. Barney, G. S. and R. G. Cowan, Separation of Actinide Ions From Radioactive Waste Solutions Using Extraction Chromatography, WHC-SA-1520-FP, Westinghouse Hanford Company, Richland, Washington.

3. WHC-CM-3-5, Document Control and Records Management Manual, Westinghouse Hanford Company, Richland, Washington.

4. Criticality Prevention Specifications, CPS-L-114, Westinghouse Hanford Company, Richland, Washington.

5. Radiological Work Permits, Z-012, Rev. #1, "Routine Work Inside Gloveboxes and Open-face Hoods, Z-034, "Low Radiological Risk Activities in Radiological Controlled Areas," Westinghouse Hanford Company, Richland, Washington.

13.0 DATA SHEETS

Detailed, step by step test instructions will be prepared before any laboratory work is initiated. THese instructions will be entered in a controlled laboratory notebook in sufficient detail to allow operation by trained laboratory personnel according to WHC-CM-5-4, Section 5.4. Data from these tests will be obtained in the form of computer printouts, instrument readings, and measurements. This data will be recorded in a laboratory notebook and in a computer spreadsheet for data analysis.