

Sanitized Copy Approved for Release 2011/09/20 : CIA-RDP78-03424A002400070007-3

1. Summary Report

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11 May 56 -

31 May 57

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MONTHLY LETTER REPORT NO. *11-91*

on the

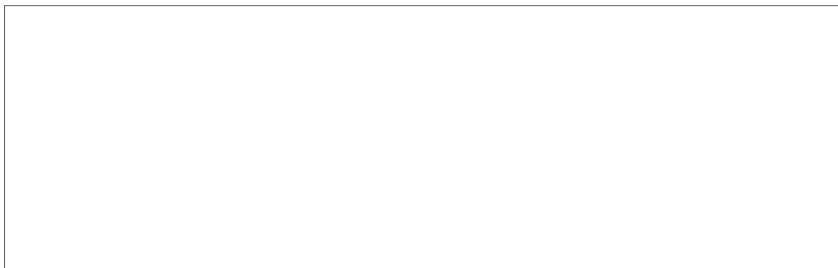
POWER SOURCES STUDY

Period: May 1 to May 31, 1957

Reference:



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I Personnel

[redacted]

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Total man-hours for period: 542

II Trips and Conferences

On May 23, [redacted]

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[redacted] to discuss problems pertaining to measurement of thermal conductivity of zinc antimonide over the temperature range of interest in the development of a thermoelectric generator. Many details of the design of equipment for such measurements were discussed, and improvements in the proposed device were evolved.

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On May 22 and 23, [redacted] attended the Eleventh Annual Battery Research and Development Conference (sponsored by the U. S. Army Signal Engineering Laboratories) at Asbury Park, N. J., which featured, in addition to papers in the field of batteries, presentations on solar energy and nuclear and thermoelectric devices.

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III Progress Achieved During the Period Covered

A. Laboratory Work

1. Several systems of improving the high-temperature stability of bonds between zinc antimonide and contact pieces have been investigated. The conditions to be satisfied are rather stringent: the bond must be solid at the highest temperature experienced by the hot junction; it must possess a low electrical resistance at all temperatures; its mechanical integrity should not diminish after prolonged heating in air; and its ingredients should not have deleterious effects upon the performance of the complete thermocouple, if they diffuse into

the zinc antimonide. It was found that electroplated layers of nickel or iron stripped from the zinc antimonide surface after heating. Electroplating of an extremely thin layer (1-3 microns thick) of silver on the zinc antimonide, followed by admixture of an alloy of lead and tin resulted in good bonds, but adherence of the silver to the zinc antimonide could not be reliably controlled. Two promising methods have been found which produce excellent bonds at room temperature, and whose products are being tested at elevated temperatures. One relies on the alloy formed by zinc antimonide and pure tin for the bond; the other employs a small amount of silver, previously electroplated on the contact piece, to raise the melting point of the tin-zinc antimonide alloy.

Improvements in the handling properties of zinc antimonide pieces have resulted from preparation of the alloy in a two-step vacuum fusion. The weighed ingredients are placed in a Vycor tube, and melted while being pumped in a vacuum system. After being sealed off, the Vycor tube is placed in another furnace, and the molten alloy agitated to assure complete mixing. Material prepared in this manner has exhibited greater uniformity and less occluded gas than that prepared without the initial melting and evacuation.

2. The activation period of bromine-activated silver-silver bromide cells has been reduced from several hours to less than one minute. This improvement has come about through careful adjustment of the components as they are being assembled, and by monitoring the electrical resistance while doing this. A number of cells prepared during the reporting period have developed the full expected voltage in less than one minute after the bromine was admitted to the cell.

Reduction of the internal resistance of activated cells to less than the designed value of 900 ohms per square centimeter has been achieved. Part of this reduction in resistance has been the result of use of layers of silver bromide

which are less than half the customary thickness.

It has been found that the life of cells made up to this time has been limited to the life of some solder joints in the associated test equipment. Elimination of these joints is expected to increase the cell life greatly.

B. Study Program


This portion of the program has been neglected, in order to devote more time to the laboratory work described above.

IV Plans for Next Month

Testing of complete thermocouples, prepared as outlined above, will be carried out under conditions approximating actual operation of the thermoelectric generator. Modifications in the composition of zinc antimonide will be made, to compensate for the diffusion of bonding material into the alloy. Continued refinements in the techniques of preparation of bromide-activated cells will be explored.

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June 26, 1957

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MONTHLY LETTER REPORT NO. 8

on

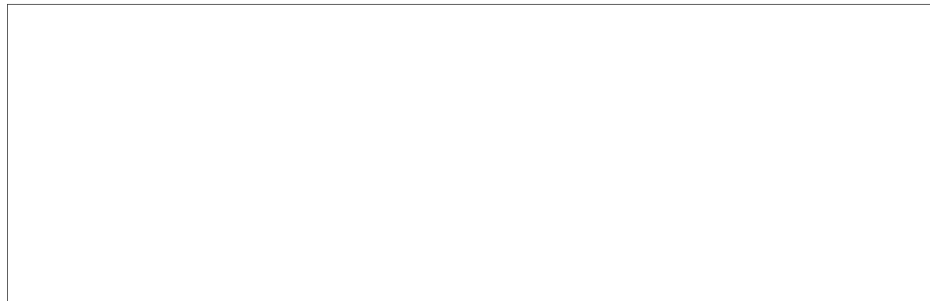
POWER SOURCES STUDY

Period: April 1 to April 30, 1957

Reference:



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I Personnel

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Total man-hours for period: 487

II Trips and Conferences

There were no trips and conferences involved with this program during the reporting period.

III Progress Achieved During the Period Covered

A. Laboratory work

1. The stability at elevated temperatures of two thermocouples - one made from constantan and zinc antimonide originating in the TKG-3 thermoelectric generator and the other from constantan and zinc antimonide prepared in the laboratory - has been determined. With the source temperature approximating 350°C and the sink temperature about 20°C, the actual temperature difference between the hot and cold junctions was about 280 to 300°. Both of the thermocouples described above were subjected to these conditions for more than 300 hours. At the end of this time, the thermal e.m.f. had changed by about 2% in both cases. It is interesting to note that the couple prepared from TKG-3 material increased in thermal e.m.f., while that prepared from our own material decreased. This may be explained on the basis of diffusion of silver and tin from the bonding material into the zinc antimonide pieces. Small additions of

these elements to the TKG-3 zinc antimonide result in an increase in e.m.f. Since its content of these elements is already at the optimum value, material produced in the laboratory decreases in e.m.f. upon addition of more tin and silver.

The technique for bonding was modified to include nickel plating of the ends of the zinc antimonide prior to bonding. This was done in order to diminish the deleterious diffusion of tin and silver from the bonding material into the zinc antimonide under the influence of elevated temperatures. It was found that oxidation of the nickel caused it to chip off during the bonding process despite the fact that its coefficient of thermal expansion approximates that of zinc antimonide. It was also observed that some nickel was electrodeposited on the sides of the zinc antimonide, providing an electrical short-circuit and reducing the observed e.m.f. Despite these difficulties, an efficiency in excess of one per cent was observed for a thermocouple made from constantan and zinc antimonide produced in the laboratory. The efficiency calculation was predicated upon thermal conductivity data supplied by

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2. Bromine-activated silver-silver bromide cells embodying a new, protective, conducting paint have been prepared. Several cells were made, exhibiting the full expected potential of 0.98 volts, although this value was not reached for several hours after activation. Increasing the load current appears to decrease the time required to reach full voltage.

Use of metallic coatings on the silver to prevent corrosion by the bromine has been investigated with some promising results.

B. Study Program

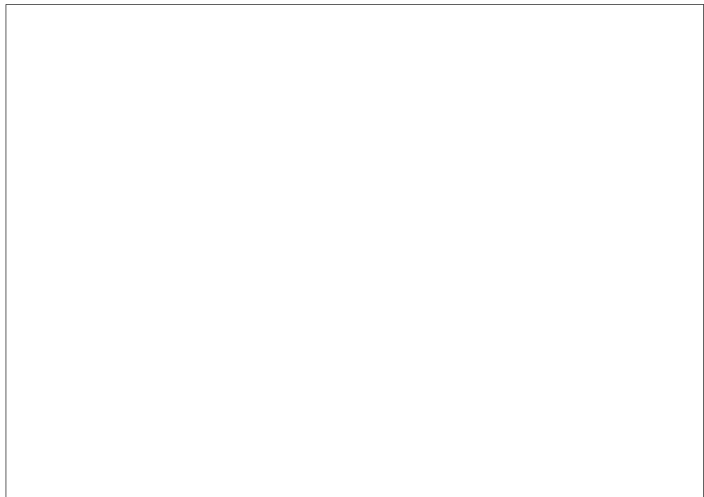
A new group of translations from the Russian periodic literature has been received from Infosearch, a British translation service. Among the papers included is a description of the design and performance of the TKG-3 thermoelectric generator. From comparison with our own data, it would appear that the specimen we examined was somewhat better than average.

IV Plans for Next Month

Substitution of a thin coating of iron for the electroplated nickel at the ends of the zinc antimonide pieces will be attempted. Difficulties are expected as a result of possible thermal shock experienced in making bonds. Other refinements in bonding techniques will have to be made to accommodate this change. Nickel-plated components will be employed in experimental gas-activated cells. In addition, several load - and life - tests will be performed and the production of brominated silver foils under controlled conditions will be carried out. The elimination of the delayed activation time will be attempted.

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May 27, 1957



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MONTHLY LETTER REPORT NO. 7

on

POWER SOURCES STUDY

Period: March 1 to March 31, 1957

*Recd 4 June 1957
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Reference:

[Redacted Reference Box]

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[Redacted Content Box]

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I. Personnel

[Redacted]

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Total man-hours for period: 533

II. Trips and Conferences

On March 26 a summary of the past year's accomplishments and plans for next year were presented at a meeting at [Redacted]

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[Redacted]

Briefly, it was stated that the thermoelectric- STAT

generator portion of the study would continue toward the development of a working device, but that additional effort would be applied toward the development of improved thermocouple materials. The objective of this materials search would be the replacement of the constantan negative branch of the thermocouples with a semiconductor as well as the general improvement of the performance of the generator.

The battery portion would also continue in the development of a gas-activated bromine battery. There would be added an investigation of the feasibility of an electrochemical system employing ground water as the electrolyte.

Rather than maintaining the study program as a separate entity, it was suggested that this portion of the work be considered an integral part of the thermoelectric-generator and battery investigations whenever applicable.

III. Progress achieved during the Period Covered

A. Laboratory work

1. Using an alloy of silver, tin and lead, it has been found possible to produce durable bonds between zinc antimonide and steel contact pieces. The technique consists of electroplating silver on the steel, coating the ends of the zinc antimonide with a

-2-

commercial "tinning compound" and passing large electric currents through the assembly while it is subjected to moderate pressure. By adjusting the thickness of the silver and the "tin" layers, the composition of the bonding material may be altered. Using material obtained from the TKG-3, a zinc antimonide-constantan thermocouple has been produced and is being subjected to a life test. The high temperature in this life test is controlled at 350°C and the low temperature is that of running tap water (less than 20°). Provision has been made for measurement of temperatures at several points. Preliminary indications are that the stability of the TKG-3 material in this configuration is greater than that observed in the TKG-3 thermoelectric generator itself.

The coefficient of thermal expansion of a specimen of doped zinc antimonide prepared in the laboratory has been measured. Although the apparent coefficient of expansion approximates that of nickel or some steels ($15 \times 10^{-6}/^{\circ}\text{C}$), there is a definite change in the coefficient after the material has reached a temperature in excess of 350°C. It is suspected that this phenomenon results from imperfect annealing during the casting process. This hypothesis is being investigated further.

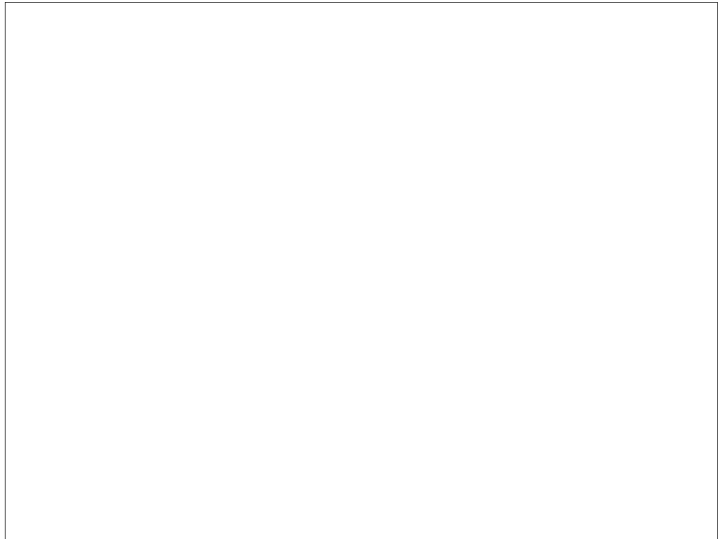
2. A review of previous work on bromine-activated solid electrolyte batteries has shown that a redesign of the experimental single cells and facilities for their assembly is necessary. This has been accomplished, and several cells of the new type fabricated. Particular emphasis is being placed upon reduction of internal resistance of the cells upon study of the effects of water vapor on their performance.

B. Study Program

No special emphasis was placed on this portion of the study during the reporting period.

IV. Plans for Next Month

It is planned to continue the measurement of properties of complete thermocouples using zinc antimonide prepared in the laboratory. Refinements in the bonding technique to increase the number of bonds which can withstand the high thermal gradient will be sought. The work of the gas-activated battery program will continue as during the present reporting period.



April 25, 1957

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MONTHLY LETTER REPORT NO. 6

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on the

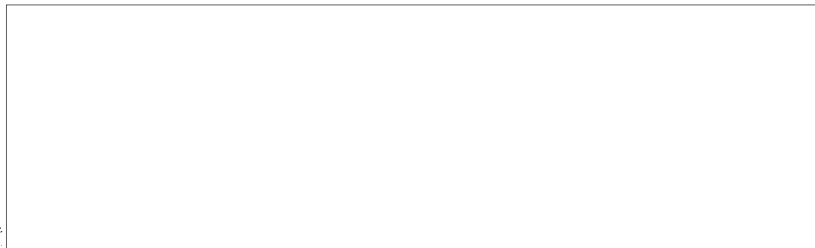
POWER SOURCES STUDY

Period: January 1 to January 31, 1957

Reference:



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I. Personnel

[redacted]

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Total man-hours for period: 293

II. Trips and Conferences

On January 17, [redacted] met with other interested parties in Washington to discuss the status and plans of the Study and to establish the requirements to be met by power sources considered in the Study. It was disclosed that a TKG-10, an improved version of the TKG-3 currently under investigation, might become available to us for study.

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On January 24, [redacted] met with personnel of the Diamond Ordnance Fuze Laboratory to discuss power sources of mutual interest. DOFL is investigating improved zinc-silver oxide systems, which are relatively conventional batteries. A small amount of work at that installation is devoted to a study of the feasibility of using moist earth as the electrolyte and separator of a reserve battery. It would be possible, should the system prove workable, to store the electrode system indefinitely, then to insert the assembly in the ground when it is desired to use the battery. Removal from the ground would restore the battery to an inactive status. DOFL also made an offer of assistance in the form of acting as intermediary in obtaining ASTIA reports, or in approaching the Franklin Institute for information concerning their programs.

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On January 24, [redacted]

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[redacted] Details of techniques which [redacted] has used for bonding of zinc antimonide to steel contact pieces were discussed in detail, as were modifications in composition of the impurity mixture to be added to the material.

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III. Progress achieved during the Period Covered

A. Laboratory work

1. The TKG-3 was maintained at its operating temperature for a total of 1296 hours, and the following changes noted since the start of the experiment.

Property	Original Value	Final Value
Open-circuit e.m.f., volts:		
"Anode" circuit	5.20	4.55
"1-3" circuit	3.04	2.70
Efficiency, per cent	0.917	0.749

Upon termination of the experiment, the TKG-3 was disassembled, and found to contain a total of 168 thermocouples, with 108 couples in the "anode" circuit, and 60 in the "1-3" circuit. Samples have been furnished for spectroscopic analysis, and others will be subjected to conventional chemical analysis.

2. A quantity of zinc antimonide containing appropriate additions of tin, bismuth and silver was prepared. This was found to be capable of being sawed into small pieces by use of a diamond wheel. Some of these pieces were electro-plated with silver as an initial step in the development of bonding techniques for assembly of thermocouples. It is planned to bond the zinc antimonide to steel contact pieces by plating the surfaces with silver (or plating one with silver and the other with tin), then to form an alloy at the bonding point by passage of a large electric current. A jig for holding the pieces during this operation has been tested, and initial results have emphasized the necessity for formation of an adherent coating of silver on the steel.

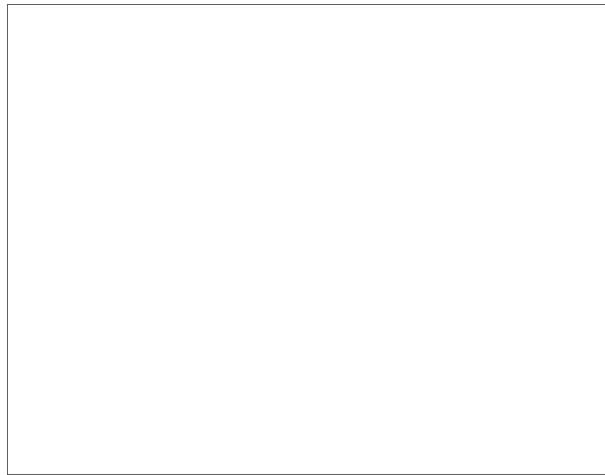
B. Study Program

Most of the information derived from the study program is described briefly under "Trips and Conferences".

IV. Plans for Next Month

Interpretation of the information derived from the chemical analysis of the TKG-3 will take place, correlating the chemical information with the observed electrical behavior. Details of construction will be carefully analyzed and appraised.

The development of reliable and reproducible bonding techniques for zinc antimonide and steel will continue.



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February 21, 1957

MONTHLY LETTER REPORT NO. 5

on the

POWER SOURCES STUDY

Period: December 1 to December 31, 1956

Reference:

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I. Personnel

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Total man-hours for period: 305

II. Trips and Conferences

Due to the pressure of laboratory work, there were no trips or conferences during the reporting period.

III. Progress Achieved during the Period Covered

A. Laboratory Work

1. The TGK-3 thermoelectric generator has been maintained at a temperature approximating its operating temperature for nearly 700 hours. Periodic measurements of its output and efficiency have been made, with the following results:

Time since Start of Experiment, hours	Open-Circuit E.m.f., volts "Anode" Ckt.	E.m.f., "1-3" Ckt.	Efficiency, per cent
0	5.20	3.04	0.917
360	4.88	2.86	0.853
695	4.60	2.75	0.798

Because the efficiency and output voltages are falling, albeit at a decreasing rate, it is planned to continue the measurements for at least one more interval of 300 to 400 hours, then to determine whether the apparent deterioration ceases or continues.

2. Sufficient zone-refined material for production of a batch of zinc antimonide has been accumulated, and production of such a batch was attempted. During this procedure, it became evident that re-design of the furnace used in melting was essential, and that has been accomplished.

3. The remainder of the laboratory work has been devoted to designing equipment, procuring parts for equipment, and refining the design of the finished thermocouple. In addition to the furnace mentioned above, there has been completed a design for a re-casting apparatus for zinc antimonide.

This will permit the production of zinc antimonide in bars of the correct cross-sectional area for inclusion in the finished thermoelectric generator.

Problems of procurement of parts for the "efficiency meter" and of Advance ribbon for the thermoelectric generator occupied a significant portion of the period. Obtaining heaters of sufficient stability, uniformity of temperature, and ability to perform throughout the desired temperature range of the thermoelectric power meter was a difficulty which has finally been surmounted. With the arrival of the material which has been ordered, construction of the measuring devices can take place.

B. Study Program

Because of the great emphasis on laboratory work, the study program has been dormant during this reporting period.

IV. Plans for Next Month

Upon termination of the high-temperature study of the TKG-3 thermoelectric generator, it will be disassembled. Its construction and design will be studied in detail. Subsequently, chemical and spectroscopic analysis of its components will be performed.

Production of zinc antimonide from zone-refined materials will continue, upon completion of the re-designed melting furnace. Special techniques and equipment for handling the material (sawing, preparation for soldering, soldering) will be developed. Assembly of testing and measuring equipment will take place at a rate governed by the arrival of ordered components.

It is planned to consult with during the coming month, in order to obtain more information concerning soldering and contact-resistance problems involved in handling zinc antimonide.

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January 16, 1957

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MONTHLY LETTER REPORT NO. 4

on the

POWER SOURCES STUDY

Period: October 1 to October 31, 1956

Reference:

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October Report Draft

Page 1.

I. Personnel

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Total man-hours for period: 532

II. Trips and Conferences

attended the Electrochemical Society meeting in Cleveland, Ohio, on October 1 - 3, 1956. In addition to reports of progress in the conventional electrochemical battery field, there were some papers in the semiconductor area which were of interest. In particular, a paper by Dr. E. Mooser, of the Physical Institute ETH, Zurich, Switzerland, contained several points of value for predicting the properties of compound semiconducting materials. In discussions with Dr. Mooser and with Dr. T.C. Harman, of Battelle Memorial Institute, Columbus, Ohio, much information concerning the state of the thermoelectric generator art in the United States, in Western Europe, and in Russia was obtained. Dr. Mooser discussed his impressions of a paper presented by the Russian Academician, Dr. A.F. Ioffe, and supported our previous impression that the Russians are successfully expending a large amount of effort in the thermogenerator field.

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Representatives of the Customer visited the laboratory on October 11 and October 25. In the course of these discussions, it was decided to reduce the power output of the proposed thermoelectric generator, as a consequence of the heat-transfer observations described below. We also received from these representatives several reports concerning European progress in the fields of thermoelectric generators and fuel cells.

October Report Draft

Page 2.

III. Progress Achieved during the Period Covered

A. Laboratory Work

1. Measurements of the available heat from various heat sources have continued. A charcoal fire was found to yield approximately 550 watts at a temperature of 350°C. At the same temperature, a wood fire produced over 650 watts, a small gasoline burner 420 watts, a larger gasoline burner over 550 watts, and a 2,000-watt electrical hot plate produced only about 1,000 thermal watts in the copper calorimeter. In order that the design of the final thermoelectric generator remain realistic, permission was requested from the Customer to lower design specifications to a flow of only 600 watts, rather than the former figure of 1,000 watts. The permission was granted.

2. A quantity of zinc antimonide was produced, following the directions received from and some difficulty was experienced in obtaining the substance in a form which was not brittle. Impurities in the raw materials are suspected of being at fault, and some successful attempts to purify them by means of zone-refining have been made.

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3. Design and construction of equipment for measuring the properties of thermocouple materials continues. A device for measurement of resistivity has been constructed, and an "efficiency meter" is being designed.

4. Means have been devised and are being tested for producing an adherent layer of carbon on a silver sheet. The carbon powder should be sufficiently adherent and porous to permit its acting as a gas cathode in a bromine-activated silver-silver bromide cell. Multi-cell arrays of stacked cells have been produced and are being tested and perfected.

B. Study Program

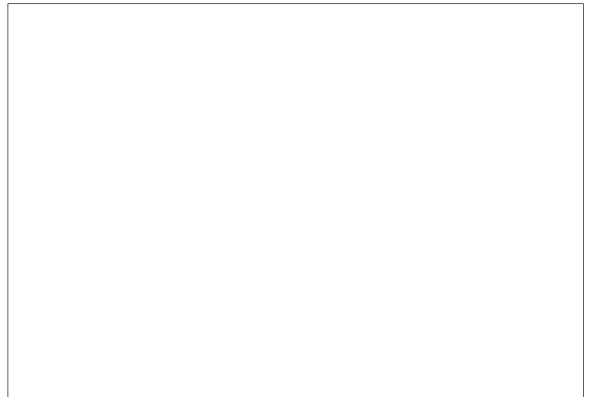
Activity in this area has been confined to study of translations of Russian literature, and to study of some of the documents left here by the Customer's representatives.

October Report Draft

Page 3.

IV. Plans for Next Month

The laboratory work outlined above will continue, with emphasis on design and construction of equipment and experimental gas-activated cells. We have been apprised, by the Customer, of the impending arrival of a Russian radio powered by a thermoelectric generator. Extensive measurements of the properties of this system will be made on its arrival.



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MONTHLY LETTER REPORT NO. 3

on the

POWER SOURCES STUDY

Period: September 1, 1956 to September 30, 1956

Reference:

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September Report Draft

Page 1.

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I. Personnel

[REDACTED]

Total Man-hours for period: 334

[REDACTED] joined the group for an indefinite period on September 24. They are concerned with the gas-activated battery portion of the study. [REDACTED] joined the group on September 1, and is working as a technician on thermoelectric aspects of the program.

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II. Trips and Conferences

There were no trips or meetings during the reporting period.

III. Progress Achieved during the Period CoveredA. Laboratory Work

1. Heat-transfer studies for the proposed thermoelectric generator were continued. Measurements of the available heat from various energy sources were extended to standard stove-type gas burners, and about 750 watts were found to be available from this type of source. By use of a model, the transfer of heat from the "cold" side of the generator to boiling water was found to be readily attained and nearly equal to the heat available from the source employed.

2. With the arrival of information from [REDACTED] concerning production and testing of zinc antimonide-constantan thermocouples, a major effort on design and fabrication of equipment for production and testing of thermocouple elements has begun. A furnace for melting and annealing zinc antimonide has been constructed, and design of a fixture for measurement of resistivity of the material is under way.

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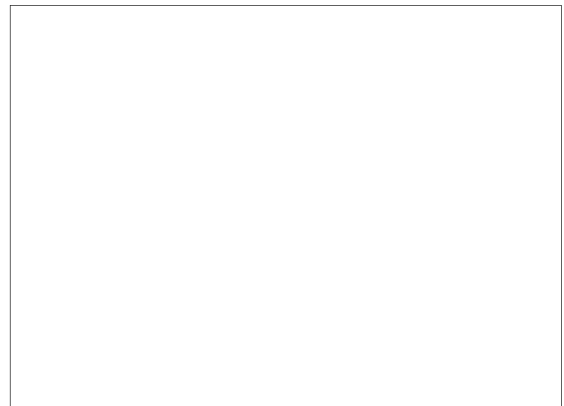
3. Initial design and planning studies for a bromine-activated silver-silver bromide cell have begun. It is hoped that fabrication of some preliminary designs can take place in the near future.

B. Study Program

A series of translations from the Russian periodical literature has arrived and has been studied. From these translations, it has become evident that Russian efforts in the field of thermoelectricity are quite extensive. The material contains much general theoretical and experimental information, but lacks details of specific materials and designs. A major benefit derived from study of these translations has been the acquisition of criteria for intercomparison of materials for thermoelectric generators.

IV. Plans for Next Month

Heat-transfer studies, using wood, charcoal, and other types of fires, will be continued. Production of zinc antimonide will commence, in order that skill in handling may be acquired. It is hoped that some experimental gas-activated cells will be constructed and tested. Several members of the staff plan to attend the Meeting of the Electrochemical Society early in the month, where they hope to learn of some of the recent developments in the battery field and to become acquainted with others active in the area.



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MONTHLY LETTER REPORT NO. 2

on the

POWER SOURCES STUDY

Period: June 12, 1956 to July 31, 1956

Reference:

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I. Personnel

[Redacted]

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Total man-hours for period: 339

II. Trips and Conferences

The staff visited with [Redacted]

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[Redacted] to discuss techniques of handling thermocouple materials for area 2.

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[Redacted] was willing to discuss general aspects of her development of high-efficiency zinc antimonide-constantan thermogenerators, but would prefer to be engaged as a consultant before giving detailed information concerning the preparation and handling of these couples. A search of the periodical and patent literature shows that details of this sort can be conveniently obtained in no other way than by direct consultation with one who has had experience with these materials. An agreement of this type has been drawn up for the approval of the Customer.

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III. Progress Achieved during the Period Covered

a. Laboratory Work: Experimentation on the proposed thermoelectric generator can be subdivided into three areas:

Area 1: Heat-transfer studies on models without electrical output. We are particularly interested in the study of heat transfer from different kinds of fires, burners or hot-plates to the hot-junction terminal plate. We want to be sure that such heat transfer is adequate for the proposed geometry and temperatures.

Area 2: Study and adaptation of the [Redacted] zinc antimonide-constantan couple to our needs.

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Area 3: Integration of areas 1 and 2.

Preliminary skillet-type models for Area 1 have been constructed. However, tests proved the internal thermal resistance of these models to be substantially too high. Improved models are being made.

b. Study Program: Several attempts to obtain copies of reports based on Government-sponsored research in the battery field from ASTIA Document Service Center in Dayton were unsuccessful. The agency reports that it is making every effort to obtain these documents, and that it will forward copies when they are available.

IV. Plans for Next Month

Continuation of experiments on the heat transfer aspects of our proposed thermoelectric generator design is planned. Subject to the Customer's approval a retainer agreement with [redacted] will be signed. Immediately thereafter consultation with [redacted] will be initiated. Our visit to the Signal Corps Engineering Laboratory has been set for August 16 and 17. While the primary purpose of this visit is to collect information for our study, we also have the Customer's permission to discuss our gas-activated silver bromide battery system proposal. Experimental work on this system may be initiated. A German, hermetically sealed, rechargeable pocket flashlight cell has been received on loan from [redacted] [redacted] for evaluation. Upon receipt of the Customer's scientific papers and the ASTIA reports, we shall continue our study of the literature.

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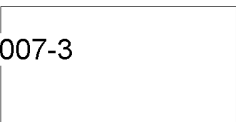
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August 7, 1956



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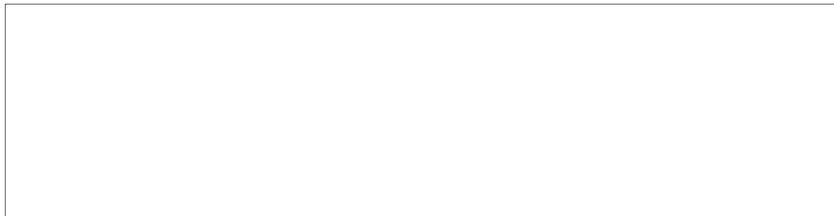
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Monthly Progress Letter No. 1
Covering period May 11, 1956 to June 11, 1956

POWER SOURCES STUDY



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I. Personnel

[redacted]

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Total man-hours for period: 160.

[redacted]

reported for assignment to the Study on May 14. While

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awaiting Department of Defense "secret" clearance.

[redacted]

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[redacted]

and clearance by the Customer, he has been active in the non-classified

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aspects of the program.

II. Work Performed

a. Laboratory Work: Preparations have been made for some initial experiments in the field of thermoelectric generators. Some general consideration has been given to experiments involving gas-activated solid-electrolyte batteries. The staff also visited

[redacted]

on June 5, in order to discuss supplies of component parts for gas-activated solid-electrolyte battery systems.

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b. Study Program: Memoranda on the following subjects were issued for transmittal to the Customer:

1. Possible utility to the Study program of Army liaison group in West Germany.
2. Electrical energy available from a fuel cell operating from a portable hydrogen supply.
3. Russian development of a thermopile generator.
4. German development of a thermocouple generator.

In addition, the staff attended the Signal Corps Engineering Laboratory Battery Conference in Asbury Park, N.J., on May 23 and 24, where they made the acquaintance of personnel from the American Machine and Foundry Corp. and NiCad Corp., both of which are active in the production of wet batteries.

[redacted]

on May 31, visited the Customer in Washington. The

following topics were discussed:

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1. The reactivation of STAT
2. Difficulties in obtaining U.S. information for our study phase, due to security classification of our contract number.
3. Difficulties in obtaining information from abroad.
4. Recent German and Russian developments in the following fields:
 - thermoelectric generators
 - nuclear batteries
 - hydrogen fuel cells

Our Customer indicated his willingness to help, wherever possible.

III. Plans for Next Month

In addition to continuing development of an experimental program, several visits to authorities in the battery field and organizations sponsoring research in the field are contemplated. STAT

has agreed to discuss her accomplishments in the field of thermo- STAT
electric devices. Another visit is planned for the Signal Corps Engineering Laboratory, provided permission to discuss the silver bromide gas-activated system can be obtained from the Customer.

June 18, 1956

STAT