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FINAL REPORT

DEVELOPMENT OF A LOW TEMPERATURE BATTERY FOR SPACE PROBE APPLICATIONS

by

William F. Meyers and George M. Armstrong June 24, 1964 to June 23, 1965

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

CONTRACT NAS 3-6009



LIVINGSTON ELECTRONIC CORPORATION Subsidiary of G. & W. H. Corson, Inc.

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William F. Meyers and George M. Armstrong

ABSTRACT

The objectives of the NASA-Lewis Research Center low temperature battery research program are the development, design and fabrication for space applications of low temperature cells which are capable of not less than 72 hours operation at temperatures as low as -90°C and which have a wet stand capability of at least one year. The Livingston Electronic Corporation was awarded Contract NAS 3-6009 under this program and the following accomplishments are reported.

Seventy-two hour primary reserve ammonia cell operation over the temperature range +20°C to -73°C was accomplished with Mg/KSCN: NH₃/HgSO₄ cells by the partial replacement of potassium with magnesium ions, the addition of sulfur to the cathode, and the evaluation of numerous other variables. Operation was extended to 21 hours at -90°C through further study and modification of the ammonia electrolyte. It is believed that the primary limiting factor to wet stand in liquid ammonia batteries has been identified.

authr

SUMMARY

<u>Phase I</u> of this program was to extend the state-of-the-art of low temperature ammonia batteries to 72 hour performance in the temperature range -40°C to -73°C. Despite numerous technical difficulties, including the need for more reliable battery hardware below -60°C, this principal objective was accomplished. The single, primary reserve, cell design:

Anode	/	Electrolyte	/	Cathode
Magnesium Sheet	/	KSCN in NH3 25 Weight %	/	Hg SO₄:S :C Bobbin

provided more than 72 hours at -73°C.

<u>Phase II</u> concerned the development of a pure primary ammonia low temperature battery with a wet stand capability of one year. As a minimum, the following methods were to be investigated.

- (a) Plating or protective coating of the anodes.
- (b) Formulating the electrolyte as an acid, anodeprotecting, plating solution.
- (c) Study local action mechanisms.

Items (a), (b) and additional related areas were studied extensively and many cells were constructed without success. Approach (c) revealed that the primary limiting factor to wet stand in this type of ammonia battery starts at the cathode reaction mechanism and proceeds only secondarily with anode corrosion. Recognition of this local action pattern is considered a substantial accomplishment. While experiments arranged to utilize this new knowledge gave indications of potential wet stand, the construction and testing of batteries of an additional cathode material were beyond the scope.

<u>Phase III</u> was concerned with the extension of ammonia battery operation below -73°C with a goal of -90°C. Study of the KSCN:NH₃ electrolyte system revealed that 34 weight percent KSCN in NH₃ and resulting spent electrolyte mixtures remained fluid and highly conductive even at -90°C. With this modified electrolyte, the Mg/HgSO₄:S:C cell supplied the full electrical load for 21 hours and the light load for a total of 37 hours. Reference electrode measurements and further cell tests defined the -90°C limiting factor as cathode polarization. Changes in cell design aimed at correction of -90°C polarization gave favorable test results.

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1. INTRODUCTION

A number of attempts have been made over the years to make an efficient low temperature battery. The inability to solve this problem has produced a number of alternate methods for providing electrical energy at low temperature. The alternate methods involve additional equipment and more complicated devices. Prior to this work, the ammonia battery possessed some of the features which were desired in that it produced the same level of service down to -55°C. The three goals of this development program were: performance in the temperature range -40°C to -73°C, wet stand capability, and extension of the ammonia battery operation even below -73°C,

During the first three months of this investigation, numerous direct attempts were made to accomplish the goals by the use of previous ammonia battery techniques. At the end of the first quarter, it was concluded that a number of new techniques as well as improved vehicles and equipment would be required to provide the desired battery performance.

During the second and third quarters, new techniques were proposed, translated into test cells, and evaluated. During the last quarter, cells were actually operated under load at -90°C and substantial progress was made towards the eventual attainment of wet stand in liquid ammonia batteries.

Many variations in cell geometry, composition, and treatment were tested to improve performance and understanding of the factors which affect the capacity of these cells. While it may be desirable to evaluate single changes in design, it is not usually possible to do so in a battery. Comparisons have, therefore, been made by tabulation of related runs; and a broader area of factors has been studied.

For temperatures down to -73°C, investigations were made into cell geometry, separation, ion exchange, combinations of various oxidants and a variety of special treatments and electrolyte studies. Activated stand was approached from both theoretical and experimental considerations. Anode protective coatings, inhibitors, improved separation, ion exchange and cathode composition comprised the more important areas of study. Operation below -73°C with a goal of -90°C was initiated by determining the effects of KSCN concentration on the freezing point and conductivity of the electrolyte. Both cathode composition and cell geometry were found to be important factors.

Supporting data is provided in Appendix A describing cell vehicles, instrumentation, and equipment; a substantial Appendix B defines the majority of the cells tested and their performance. Duplication was limited in order to expedite the study of a broader field of variables and to establish relative trends. It was felt that group correlation of many related runs featuring minor variations would provide the desired reliability by accumulation. Numerous tests exceeded the requirements of 72 hours.

Livingston Electronic Corporation feels that the material presented is a substantial contribution to the battery art. Primary reserve cell performance was obtained from $\pm 20^{\circ}$ C to -73° C. The primary limiting factor to wet stand in ammonia batteries has been uncovered. Performance at reduced rate or life was observed down to -90° C.

2. CELL DESIGN

Statistical methods overcome many limitations of conventional design of experiments and the need for repeated duplication. The direct application of statistical techniques was not practical in this new area of extremely low battery temperatures due to the unknown and overriding effects of many of the variables. However, some of the thinking¹ which underlies statistical design of experiments was utilized to alleviate slow single steps and the need for repetition in searching for important facts. Many single tests at varying levels and factors were conducted to provide a general understanding of the significance of these ammonia parameters in the relatively unexplored battery area approaching -90°C.

For example, sulfur was found to be an extremely desirable additive for $HgSO_4$ cells². Increasing quantities provided increased life resulting in a maximum at $S/HgSO_4 = 2$. The regularity of this group of points tends to verify the reliability of each individual test on a group basis.³

Similarity of performance from cells incorporating minor variations from a tentative design center⁴ also supplements the establishment of system reproducibility presented in Section 6 "Reproducibility," on page 55.

1	"Design and Analysis o	f Industrial	Experiments, "	pp. 4 and 5,
	Edited by Owen L. Dav	ies, Hafner	Publishing Con	npany, 1960

- ² See Table V, Section 3.1, page 13.
- See Figure 1, page 4.
- ⁴ See Section 3, page 10.

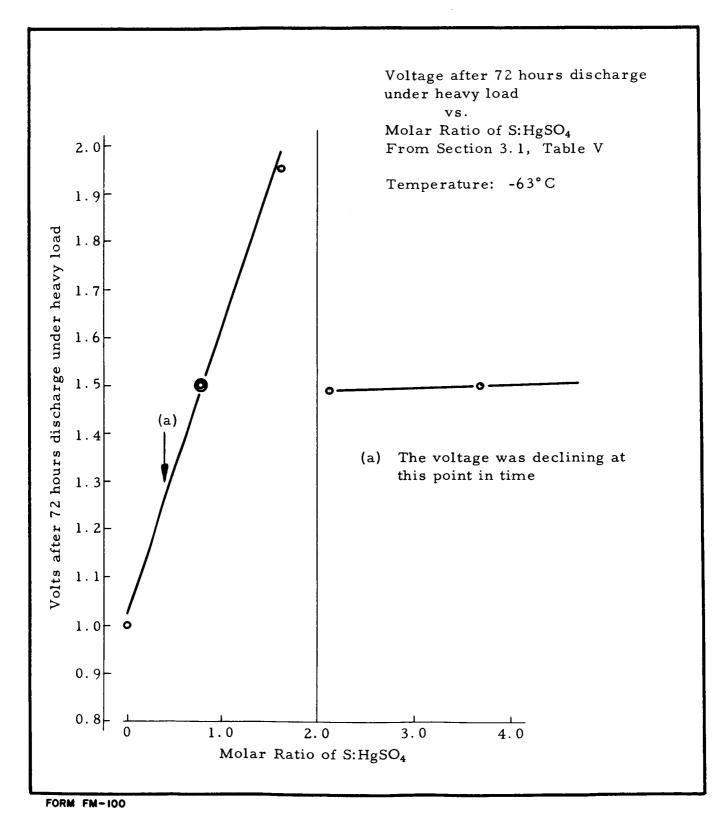


FIGURE 1 Voltage at 72 Hours vs Molar Ratio of S:HgSO₂

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2.1 Anode Variations

Numerous types of magnesium anodes were tested including rods, finned and fluted rods, sheets of varying thickness, expanded sheets, and tubes. Magnesium alloy AZ31B-0 sheet was found to perform better than more complex structures when the surface away from the cathode was inhibited. A thickness of 0.025 inch of this material proved ample and consistent with a resilient but workable structure.

TABLE I

Anode Variations at 20 grams of Sulfur and -63°C

<u>Test No.</u>	Anode	Hg SO 4 [:] NH3	Hrs.to
	Thickness	Coord. No.	<u>1.5v</u>
T-37 & 41	0.016''	2	91
T-64	0.040''	2	71
T-66	0.014''*	0	87
T-97	0.025''**	0	107
T-104	0.025''**	0	96

* Pure magnesium sheet; all other anodes were the alloy, AZ31B Mg.
** A microporous rubber separator was employed in this cell.
*** One inch cathode dia.; all others were 1-1/8" in dia.

Examples of cell tests at $-63^{\circ}C^{1}$ are given in Table I indicating 'the trends evident for the variables of anode thickness, composition, and diameter. While further optimization should be possible, it is evident that sufficient life at low temperature is available with simple anode structure but that the interaction with factors subsequently discussed is of importance.

¹ A test temperature selected for convenience and appropriateness to the temperature range -40 to -73°C.

5

2.2 Cathode Design

One group of cells was designed primarily to examine cathode and anode construction details suitable for a low-temperature ammonia, bobbin cell.¹ The bobbin diameter was varied from 5/8 inch to 1-1/2 inches with constant weight of mercuric sulfate as HgSO₄. The 1-1/8 inches diameter cathode is shown in Table II to be superior to both larger and smaller diameters and has been adopted as tentative design center. Triple cathodes of small diameter were inferior when used in conjunction with either a common anode or with individual anodes.

TABLE II

<u>Test No.</u>	Cathode Diam.	HgSO₄; NH₃ Coord. No.	Hrs.to <u>1.5v</u>	Hrs.to <u>1.3v</u>	Anode No.	Cathode No.
T-40	1.50''	2	48	72	1	1
T-37 & 41	1.13"	2	91	102	1	1
т-60	1.13"	0	106	119	1	1
T-104	1.00''	0	96	105	1	1
Т-65	0.75''	0	63	74	1	3
Т-82	0.63"	0	55	59	3	3

Bobbin Cathode Design - 20 grams Sulfur at -63°C

Minus 90°C cell testing was initiated in the latter part of the program by attempting to operate the Mg/KSCN:NH₃/HgSO₄:C:S cell design. Such cells had exceeded the 72 hour requirement in the -40 to -73°C region. Electrolyte Studies (Sections 4.6 & 4.7) indicated a conductive fluid at 34 weight percent KSCN. This composition was chosen as -90°C electrolyte. After one day of discharge at -90°C, these cells fell below the adopted cut-off of 1.5 volts under the heavy load portion of the cycle. Reference electrode measurements showed that the magnesium anode was performing very well but that the bobbin cathode was polarizing.

Nos. T-11 to T-17

In order to examine the effect of cathode current density upon polarization at -90°C, thin pasted plate positives consisting of $HgSO_4$:S:C (in the weight ratio of 5:1:3) were prepared using a polystyrene binder and silver-plated expanded copper sheet as the cathode collector. A cell containing two magnesium anodes² discharged at the full cyclic load for 9 hours. Since the quantity of $HgSO_4$ present was only 1/5 that utilized in the bobbin cell, it is concluded that the limiting factor in the -90°C bobbin cells was geometric cathode current density (mA/cm²). Other investigational cells eliminated cathode matrix (graphite to silver wire) conductivity as being significant at -90°C. Additional tests conducted on bobbin cells at reduced drains provided the full energy requirement over a prolonged period of time. It was concluded from these investigations that further study of pasted plates or bobbins of higher porosity may well provide additional discharge at -90°C.

The best cathode design for use in the temperature range of +20°C to -73°C was found to be a bobbin having a diameter of 1-1/8 inches and sufficient length to accommodate the required amount of oxidant and carbon. This was used in most of the test cells. However, since the pasted plate performed well at -90°C, it should be investigated further over the full range of temperature.

² No. T-132 (the anode area was the same as for the 1-1/8" bobbin cell)

2.3 Separators

The separator or separators in a battery may perform a number of functions. The simplest of these functions is to mechanically separate the anode from the cathode and to provide by this means electrical insulation. A second and less obvious function which is frequently of importance is the inhibition of diffusion of the anode reaction products to the cathode and the cathode reaction products or intermediates towards the anode. Separators such as non-woven fabrics and plastic screens serve to separate or insulate the anode from the cathode. Materials such as microporous rubber and microporous Teflon actually exert a screening effect on the size of molecules which may be transmitted by either diffusion or electrolytic transference. Ion exchange membranes go a step further and are discussed in Section 2.4. Webril M-1365, a non-woven rayon fabric, and Webril No. SM-91, a non-woven polypropylene fabric, were used as mechanical type separators in this program.

TABLE III

Comparison of M-1365 and MPR Separators at -63°C

<u>Cell No.</u>	Separator	HgSO4 gms.	S gms.	C gms.	Hrs.to <u>1.5v</u>	Hrs.to <u>1.3v</u>
Т-60	M-1365	106	20	60	106	119
T-97	M-1365 and MPR	106	20	60	107	119
Т-86	M-1365	50	50	80	83	96
T-115	M-1365 and MPR	50	50	80	85	94

In Table III, two types of bobbin cells are compared at -63°C, with and without a microporous rubber separator. Under the conditions of primary reserve operation, the discharge life to 1.5 volts cut-off showed negligible difference. However, as it will be pointed out in Section 5 of this report, "Activated Stand," the additional separation was beneficial in another way.

The non-woven polypropylene fabric became available relatively late in the program. This material was used successfully on the pasted plate cathode and on several bobbin type cathodes. It was readily heat-sealed, retained its physical strength in ammonia, and appeared to be unaffected chemically by a KSCN solution. Concerning its electrochemical characteristics, two five mil layers of the polypropylene fabric gave good performance on the pasted plate-type cathode and retained its integrity upon removal and examination after the cell discharge. When two layers of five mil polypropylene fabric were used on the bobbin type cathode, a high internal resistance was noted. A one mil variety is reported to be available.

Considering the above data in general, it is concluded that five to seven layers of four mil thick non-woven rayon fabric will provide efficient separation for the primary reserve mercuric sulfate bobbin cell.

2.4 Ion Exchange Membranes

The purpose of an ion exchange membrane is to limit diffusion or transference on the basis of the sign of ionic charge of the conducting species within the electrolyte. For example, in a cathode of the second kind such as silver peroxide, in a conventional aqueous cell, silver ions are tangibly soluble and must be confined to the catholyte. Ion exchange membranes are generally available in two types: anionic and cationic. The purpose of an anionic membrane is to allow free transference of negative ions and to inhibit the transfer or diffusion of cations or positive ions. The purpose of the cationic membrane is the converse.

Practical utilization of ion exchange membranes in aqueous systems is a relatively new addition to the battery art. At the present time, many new ion exchange membranes are being offered and others less than a year old are no longer available. Concerning the application of ion exchange membranes to non-aqueous systems, even less is known and materials specifically designed for non-aqueous media are not commercially available. However, a number of commercial ion exchange membranes were obtained, efforts were made to utilize these membranes in various designs of ammonia batteries, and the effect of the membranes upon cell performance was noted.

In only a few instances were favorable indications obtained for the membraned cells. The sulfur cathode did not respond favorably to either anionic or cationic membranes. The use of an anionic membrane¹ did improve performance of the Mg/KSCN:NH₃/m-DNB:NH₄SCN:C cell at -54°C. Performance of this cell at +20°C was not affected by the use of the membrane. AMF-104SB anionic ion exchange membranes were used in a similar cell structure and gave an improved coulombic efficiency at room temperature. The performance at -54°C was very poor due to high internal resistance. The application of two kinds of membranes to the HgSO₄:S wet stand tests resulted in improvement comparable to the use of microporous rubber.

It is therefore concluded from these preliminary investigations that ion exchange membranes characterized for their performance in liquid ammonia will be required before further progress can be made with respect to their utilization in liquid ammonia batteries.

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¹ See 3.10.3, p. 28.

3.

In this section, pages 10 through 29, the cell test data are examined for the influence of variations in chemical composition. Several cell designs were employed, and the following treatments were investigated in the temperature range $+20^{\circ}$ C to -73° C.

- 3.1 Effects of Sulfur on HgSO₄ Bobbin Cells
- 3.2 Effects of Degree of Coordination of HgSO₄
- 3.3 Variations in the Proportions of C to $HgSO_4$, and S
- 3.4 Oxidants other than HgSO₄ with S Additive
- 3.5 Comparison of Three Heavy Metal Sulfates
- 3.6 Lead Sulfate Cells
- 3.7 PbSO₄ Battery Plate Experiments
- 3.8 Addition of Sulfates
- 3.9 Cell Tests at +20°C
- 3.10 meta-Dinitrobenzene Cells

Duplication was limited in order to expedite the study of a broader field of variables and to establish relative trends. Progress would have been impeded if more time had been devoted to replication. It was decided, therefore, to rely more on group correlation, as indicated in the following tabulation, rather than to belabor any particular design since improved performance was the object of this work. For example, tests T-67, -97, -101, and -110 all exceeded 100 hours discharge and involved relatively minor variations from the tentative design center represented by Cell No. T-60 which is heavily supported by interlaced correlation.

Cells Similar to Tentative Design Center

Discharge Temperature:		rature:	-63°C
Cathode Composition:			106g HgSO4.0NH3 20g Sulfur 60g Carbon
Electroly	te:	25 Wt. % KSCN	
Cell No. Hours to: <u>T- 1.5v 1.3v</u>			Minor Variations from Tentative Design Center
60	106	119	Tentative Design Center
37	97	98	HgSO ₄ .2NH ₃
41	84	107	$HgSO_4$. 2NH ₃
66	87	94	Pure Mg anode
67	101	109	Half-spent electrolyte: half new.
74	78	87	80% -spent electrolyte: $20%$ NH ₃ (1).
85	79	95	6 grams acetylene black added
87	85	93	6 grams acetylene black added and (50% of $17\%Mg(SCN)_2$ electrolyte + 50% of 25% KSCN)
97	107	119	Microporous rubber separator
100	87	115	10 grams $(NH_4)_2SO_4$ added
108	84	99	10 grams K_2SO_4 added
101	103	108	50 Wt. %KSCN electrolyte
104	96	105	l inch diameter cathode
110	106	121	10 grams of m-DNB added.

3.1 Effects of Sulfur on HgSO₄.2NH₃ Bobbin Cells

A review of some earlier experimental data indicated that a combination of sulfur and $HgSO_4$ might provide longer life than $HgSO_4$ alone. A number of such cells were tested and a summary is given in Table IV.

TABLE IV

The Effect of Sulfur Upon the HgSO₄. 2NH₃ Bobbin Cathode at -63°C HgSO₄ Total Life in Test Sulfur 2NH₃ Carbon NH₄SCN Cathode Hours to No. gms. 1.5v gms. gms. gms. gms. T-26 44.1 19.3 49.6 50 22.0 135 T-27 44.1 19.3 49.6 22.0 135 44 T-31 39.2 34.2 44.1 19.5 137 47.5 T-4 0 119 51 0 170 63 T-18 0 63 0 59 147 210 T-19 0 147 63 0 210 53

The results show that a substantial increase in $HgSO_4$. 2NH₃ in the presence of sulfur did not increase the cell life significantly. The cells which did not contain any NH₄SCN or sulfur performed best.

Cells assembled in Table V include variations in $HgSO_4$. $2NH_3$:S:C without NH_4SCN . The quantity of sulfur was varied from 0 to 40 grams, and the ratio of $HgSO_4$. $2NH_3$:C was held at 7:3 by weight. The total cathode weight was maintained at 200 grams. The quantity of sulfur and the life to cut-offs of 1.5 and 1.3 volts at the heavy load are tabulated at two temperature levels. Twenty grams of sulfur yielded the longest life at -63°C.

At -73°C, the difference between 10 and 20 grams did not appear to be significant to the 1.5 volts level, but performance to a cut-off of 1.3 volts was substantially improved by 20 grams of sulfur.

TABLE V

Test No.	gms. S	Hrs. to <u>1.5V</u>	Hrs. to 1.3V	Temp. °C
Typical 2nd quarter	0	60	68	-63
Т-36	5	67	73	-63
T-33	10	73	78	-63
T-35	10	72	78	-63
T-37	20	97	97	-63
T-41 (excess electrolyte) 20	84	107	-63
T - 54	25	71	92	-63
T -44	40	72	97	-63
Т-6	0	57	59	-73
T-39	10	68	77	-73
T-42	20	60	85	-73

Effects of Sulfur on HgSO₄. 2NH₃ Bobbin Cells

Some of the tests of Table V were plotted as hours to 1.5V against the ratio of $S:HgSO_4$ in Figure 1, page 4. Compounds of sulfur are reported in the literature¹ for cyanogen. The thio-analogues of those listed would be:

$$S (SCN)_2 \xrightarrow{\text{Thermal}} S_2 (SCN)_2 \& S_3 (SCN)_2$$

and a possible reaction mechanism would be:

 $HgSO_4 + 2KSCN + 2S \longrightarrow Hg^{\circ} + K_2SO_4 + S_2 (SCN)_2$

The abrupt -63°C discontinuity at the stoichiometric ratio $S:HgSO_4 = 2$ would logically be related to an increased stability of S_3 (SCN)₂.

At -73°C, the discontinuity (not plotted) appears to have shifted to a $S:HgSO_4$ ratio of 1.

See also Section 5.1 of this report.

Sneed, Maynard & Brasted, Comprehensive Inorganic Chemistry, p. 226, Vol. III, D. Van Nostrand Co., Inc., N.Y., 1954

More data in finer increments at the -73° C area would clarify this point. This emphasizes the competition in importance between replication and variation in treatment level. The cell capacity at -73° C was not significantly reduced by exceeding the ratio of S:HgSO₄ of 1, but the -63° C capacity was reduced above a ratio of 2. The ratio of 1.64 or 20 grams of sulfur was adopted into the tentative design center in view of these data.

TABLE VI

<u>Test No</u> .	gms. S.	Hrs. to <u>1.5V</u>	Hrs. to $1.3V$	Temp. °C
T-4 7	0	56	57	-63
T - 76	10	72	80	-63
Т-60	20	106	119	-63

Effects of Sulfur on HgSO₄. 0NH₃ Bobbin Cells

An examination of the effect of the addition of sulfur upon life to 1.5 volts using non-ammoniated $HgSO_4$ is given in Table VI. Note that had it been desirable to schedule additional cells of this series, tests at greater than 20 grams of sulfur would have been more informative than duplication. However, other treatments such as coordination, the three component system $HgSO_4$:S:C, and other additives demanded attention.

3.2 Effects of Degree of Coordination of HgSO₄

126

106

20

20

The degree of coordination of the mercuric sulfate used in these cells has been seen to exert a significant effect. This factor was examined by the data assembled in Table VII.

Effects of Degree of Coordination of HgSO4 with Ammonia								
Test <u>No. T</u>	HgSO4 grams	S grams	C grams	Coord. No.	Temp. °C	Hrs.to <u>1.5V</u>	Hrs.to 1.3V	
20 & 21	143	0	62	4	-63	53		
Typ. 2nd Qtr.	130	0	56	2	-63	60	68	
47	126	0	60	0	-63	56	57	
			~ ~	-	<i>.</i>			
33 & 35	133	10	57	2	-63	72	78	
76	116	10	60	0	-63	72	80	
51	126	20	54	4	-63	38		
37 & 41	126	20	54	2	-63	91	102	
60	106	20	60	0	-63	106	119	
9 & 14	161	0	69	4	-73	37		
6 & 16	143	0	62	2	-73	5 2		
4.2	1.24	20	E 4	2	72	62	0 5	
42	126	20	54		-73	63	85	
61	106	20	60	0	-73	85	102	

TABLE VII

In the absence of sulfur, the effect of coordination was insignificant at -63°C but at -73°C, the diamine appeared superior to the quaternary compound. At the 10 grams of sulfur added level, variation in coordination number had no effect. At the 20 grams of sulfur level, the uncoordinated HgSO₄ was distinctly superior at the lower temperatures. At +20°C, the diamine yielded a much longer life to 1.5 V than the uncoordinated material. In Table XIV, page 23, results of special treatment of cells operated at +20°C will be given. The results of these observations are considered from the theoretical viewpoint as follows:

54

60

2

0

+20

+20

49

30

76

91

50

62

Latimer¹ gives aqueous reactions for the mercuric ion with both the thiocyanate ion and with ammonia. The equilibrium constants for these reactions are approximately the same at 2×10^{19} as indicated below.

$$Hg^{++} + 4CNS^{-} \xrightarrow{Aq} Hg (CNS)_{4} (K = 2*10^{+19})$$
$$Hg^{++} + 4NH_{3} \xrightarrow{Aq} Hg (NH_{3})_{4}^{++} (K = 2*10^{+19})$$

The implication of these aqueous data is that the mercury ion of the mercuric sulfate is at least partially coordinated by the thiocyanate ion rather than ammonia when activated with electrolyte at -63°C. Furthermore, if coordination with ammonia has been accomplished prior to this, a transformation to the pseudo halide complex does not take place. This metastable condition appears to exist or be of sifnificance in the presence of sulfur.

¹ W. L. Latimer, <u>Oxidation Potentials</u> (1952), pp. 180-1

3.3 Variations in the Proportions of Carbon to $HgSO_4$ and S

In previous subsections, the role of mercuric sulfate and the role of sulfur in the HgSO4:S:C bobbin cathode have been examined. In Table VIII, data are arranged to display the effect of variation in the C:HgSO4 and C:S ratios. Previously presented data indicated that the $3:7 = C:HgSO_4$ bobbin cell produced the longest life to 1.5V at the 20 grams of sulfur level. In the range of 40 grams of sulfur an increase in carbon was slightly beneficial, but the capacity per gram of total oxidants $(HgSO_4 + S)$ increased substantially. Thus, while the watt hours obtained were not greatly increased, the weight of the cathode reactants was substantially reduced¹. A further increase in the C:HgSO₄ ratio to 2:1 yielded a pronounced decrease in discharge time to $1.5V^2$, and when the HgSO₄ was eliminated, the cell life was reduced to $\frac{1}{2}$ of the required value. However, the capacity per unit weight of oxidants (sulfur) increased to above 900 coulombs per gram. In one cell of this series, six grams of the graphite were replaced with desiccated and micronized acetylene black. While performance at -63°C was still in excess of the required 72 hours, considerable loss in capacity accompanied this change. However, a marked improvement in electrolyte absorption was noted.

TABLE VIII

V	Variations in the Proportions of Carbon to $HgSO_4$ and S at -63°C								
Test <u>No. T-</u>	HgSO4 grams	S grams	C grams	Coord. No.	Hrs.to <u>1.5V</u>	Hrs.to <u>1.3V</u>	Coulombs per gram Oxidants		
37 & 41	126	20	54	2	91	102	466		
44	112	40	58	2	72	97	355		
48	72	36	72	2	78	93	534		
60	106	20	60	0	106	119	628		
80	75	39	72	0	83	98	544		
86	50	50	80	0	83	96	620		
99	40	60	80	0	60	72	451		
85 (a)	106	20	60	0	79	95	468		
55	0	30	90	-	41	45	968		
56	0	30	90	-	33	45	775		
59	0	42	84	-	25	45	431		

Note (a) 60 g of C included 6 g of acetylene black.

² Cell No. T-99

Cell Numbers T-44 and -48 or -80 and -86

3.4 Oxidants other than HgSO₄ with S Additive

Other oxidants might well have advantage in this type of battery. Two obvious mercuric salt alternatives (sulfide and thiocyanate) and another heavy metal sulfate were tested. The materials results are listed in Table IX. With mercuric thiocyanate, the similarity in performance at -63°C and -73°C is noted.

TABLE IX

Test	Oxid	ant	S Additive	Temp.	Hrs. to
<u>No. T-</u>	Material	Grams	Grams	°C	<u>1.5V</u>
73	Hg (S CN)₂	106	20	-73	60
70	Hg(SCN)₂	106	20	-63	63
71	Hg S	130	0	-63	21
77	Hg S	106	20	-63	35
96	$PbSO_4$	108	20	-63	34

Oxidants other than HgSO₄ with S Additive

3.5 Comparison of Three Heavy Metal Sulfates

Three ammoniated heavy-metal sulfates were also compared in cells having four gram cathodes. The discharge was carried out at -50 °C with a load resistance of 6.67 ohms and continued to a 10 percent voltage drop from the closed circuit peak. The relative performance at a high rate of drain is shown in Table X.

			TAB	LE X	Peak Anode	
Test	Metal	PCCV	End	Hours to	C.D.	F/mol of
<u>No. T-</u>	Sulfate		Voltage	End Voltage	$\underline{mA/cm^{2}*}$	Oxidant
142	HgSO4	1.98	1.78	0.73	21	$0.91 \\ 0.44 \\ 0.01$
143	PbSO4	1.50	1.35	0.46	16	
144	Sn(SO4)2	1.42	1.28	0.01	15	

No further work was done with $Sn(SO_4)_2$, but the study of $PbSO_4$ was continued.

* Based on projected anode area.

3.6 Lead Sulfate Cells

Lead sulfate, a less reactive oxidant than mercuric sulfate, was examined in cells so as to better understand the mechanism by which $HgSO_4$ functions as an oxidant in KSCN:NH₃ solutions. The theoretical basis for this approach is given in Section 5.1. If this theory were correct, it would be expected that $PbSO_4$ cells could be effectively discharged at room temperature without the additive, sulfur. Furthermore, if $PbSO_4$ functions as a truly "insoluble" oxidant rather than via soluble intermediates as predicated for $HgSO_4$, difficulties with cathode current density, in the bobbin structure, particularly at low temperatures would be anticipated.

The data of Table XI bears out the above predictions and show that $PbSO_4$ operates favorably at +20°C without sulfur as an additive. Improvement in low temperature, heavy load characteristics is considered separately in this section and in Section 3.7.

TABLE XI

Lead Sulfate Bobbin Cells

Test <u>No. T-</u>	PbSO ₄ grams	Sulfur grams	Carbon grams	KSCN: NH ₃ <u>Wt. %</u>	Temp. °C	Hrs.to	Hrs.to 1.3V	F per Mol
96	108	20	60	25	-63	34	53	
112*	108	20	60	25	-63	34	104	
117	120	0	60(a)	25	-63	0	33	
118	120	0	60(a)	25	+20	65	70	1.16
125	100(b)	0	50(a)	25	- 50	33	49	0.67
139	87(b)	0	43	34	-85	0	0	
139	87 (Ъ)	0	43	34	- 50	0	0	

Cyclic Load = 2.7/13.5 Ohms

* 10 grams m-DNB added to cathode mixture

(a) grams carbon includes 5 grams acetylene black

(b) PbSO₄. 2NH₃

The discharge of T-117 was continued under cyclic load for 89 hours to 0.8 volts, followed by alternating the light load and open circuit for 143 hours. The over-all cathodic efficiency was 90 percent at these lighter drains. T-118 was discharged under cyclic load for 72 hours to 1.0 volts and then for 69 hours under a cyclic loading of 13.6 ohms for six minutes and open circuit

for 54 minutes. Its efficiency to 0.87 volts was 66 percent. Since PbSO₄ cathodes swell badly when immersed in the electrolyte, the PbSO₄ used in the cathode of T-125 and in subsequent cells was pre-ammoniated. This cell performed reasonably well and considerably better than T-117.

The purpose of Cell No. T-139 was to test the effectiveness of a non-woven polypropylene fabric, Webril No. SM-91. It is suspected that the rayon degenerates with PbSO₄. The cell did not discharge well at -85°C. The initial light-load voltage was only 1.15. The run was continued for 22 hours to 0.35 volts under heavy load and then for an additional 73 hours at -50°C to an end voltage of 0.83. After 116 hours, however, the open-circuited voltage was still 1.85. The discharged cell and electrolyte were extremely clean. However, there were metallic lead deposits between the inner M-1365 and the outer SM-91 separators. The trial of a single layer or of thinner sheets of SM-91 is indicated.

Table XII summarizes the results of some miniature bobbin cells that were tested at -85°C and -50°C at two electrolyte concentration levels. It appears that 34 percent KSCN is better than 25 percent, and two cells ran better at -85°C than at -50°C. This latter observation is inconsistent with other experience but may well be of importance.

The faradic efficiency to the 1.5 volt cut-off which was obtained in T-135 in 10 hours extrapolate to that from T-125 (a full sized bobbin) in 33 hours.

TABLE XII

Miniature PbSO₄. 2NH₃ Cells

Test <u>No. T-</u>	PbSO ₄ grams	Carbon grams	KSCN Wt.%	Temp. <u>°C</u>	Hrs.to <u>1.5V</u>	Hrs.to	F per Mol
137	2	1	25	- 50	6.8	6.9	0.46
141	2	1	34	- 50	7.8	8.1	0.56
135	2	1	35	-85	10	10	0.68
131	1.5	1.5	25	-50	4.5	4.6	0.41
140	1.5	1.5	34	-50	7.4	7.5	0.69
133	1.5	1.5	35	-85	9.0	9.0	0.80

Cyclic Load = 47/220 Ohms

3.7 PbSO₄ Battery Plate Experiments

Since the $PbSO_4$ bobbin type cathode did not discharge well at low temperature, a more efficient geometry was considered. A commercial lead-acid battery was charged and then completely discharged followed by dismantling. The plates were thoroughly washed to remove all acid. After drying, the discharged plates were used as cathodes against Mg anodes in 25 weight percent KSCN:NH₃. A total of 19 experimental cells were attempted to establish control over Mg/PbSO₄ cell performance using discharged negatives from commercial aqueous lead-acid batteries. Refer to Table VI of Appendix B for details of these preliminary experiments.

The discharged positive plates when used as cathodes, produced an initially high voltage (2.40 volts) which rapidly dropped to normal PbSO₄ voltage and total run time was not as long as can be obtained from the negatives (Run S-1). The PbSO₄ plates obtained from the lead-acid battery showed deformation after use in the NH₃ system, probably due to expansion of PbSO₄ upon ammoniation. Best results were obtained by using two discharged negative plates as cathodes versus one Mg anode, (Run S-4), with a run time of 11 hours above cut-off volttage (1.5 volts) under cyclic load. When short-circuited, there was a 12 ampere pulse which decreased to 9 amperes and held. These short-circuit currents in relation to anode and cathode area equal 44 and 33 mA/cm², respectively.

Additional studies were conducted on $PbSO_4$ cathodes in order to examine their wet stand capabilities. The results are reported in the section on "Activated Stand," starting on page 42.

The advantages of suitable current density in the rectangular $PbSO_4$ flat plate cells and the implications of wet stand for bobbin cells provide a realistic avenue for eventual wet stand in ammonia electrochemical cells.

3.8 Addition of Sulfates

In the interest of a sound coverage of possible significant variables, 10 grams of 3 different sulfates were added to the tentative design center type cell characterized by test T-60. The results obtained are listed in Table XIII; $(NH_4)_2SO_4$ was the least detrimental.

TABLE XIII

Effects of the Addition of Sulfates to the Cathode

to Depress the Solubility of HgSO₄ at -63°C

(Basic cathode = 106g. HgSO₄ + 20g. S + 60g. C)

Test <u>No. T-</u>	Sulfate Additive	Electrolyte Code*	Hrs. to 	Hrs. to <u>1.3V</u>
81	10g. MgSO4	F	49	57
100	$10g. (NH_4)_2SO_4$	А	87	115
108	10g. K ₂ SO ₄	А	84	99

* Refer to Electrolyte Code, Section 4, Page 32.

3.9 Cell Tests at +20°C

It was not an object of this program that cells should run for 72 hours at +20°C. However, tests were conducted at this temperature for the knowledge which might be gained with respect to corrosion problems and the achievement of wet stand.

The best results, a 76-hour life to 1.5 volts, was obtained in No. T-68 which had the following features:

- 1. $H_{g}SO_{4}$. ONH_{3} :S:C = 106:20:60;
- 50 percent spent electrolyte from a similar discharged cell, plus 50 percent of 25 weight percent KSCN in liquid ammonia; Electrolyte code "B" in Table XIV
- 3. activation at -63°C
- 4. the outside of the anode was coated with K-M rubber cement to reduce corrosion.

Other significant variations are included in Table XIV.

TABLE XIV

Cell Tests at +20°C

Test No.	HgSO4. NH3 Coord.#	S grams	Electro- lyte Code*	Activa - tion: Temp.	Hrs.to <u>1.5V</u>	Hrs.to 1.3V	Special Features
T-38 T-43	4 4	10 20	A C	+20 +20	11 0	19	Incomplete actv.
T-45	4	20	A	+20	5.5	9.5	Mg tube anode
T-52 T-53 T-50	2 2 2	20 20 20	A A A	-63 -63 -63	33 35 49	45 62 76	Center anode Double anode
T-62 T-90 T-68	0 0 0	20 20 20	A A B	-63 -63 -63	30 52 76	91 93 92	Coated anode Coated anode
T-91 T-83 T-88	0 0 0	20 10 10	H G H	-63 -63 -63	60 49 67	86 76 78	Coated anode Coated anode Coated anode

* Refer to Electrolyte Code, Section 4, page 32.

The 76-hour life of T-68 was further examined by a similar test using all fresh electrolyte (52 hours) and a 3rd cell which had neither spent electrolyte or coated anode (30 hours). The last three entries of Table XIV contain mixtures of $Mg(SCN)_2$ and KSCN. Electrolyte coded "H" containing a higher concentration of $Mg(SCN)_2$ yielded good results. The use of "slaked" $HgSO_4(HgSO_4.4NH_3)$ at +20°C was unsuccessful and none of the special anodes worked as well as a single sheet of AZ31B Mg wrapped around the outside of the cathode. The data imply that $HgSO_4.2NH_3$ would be compatible with partially spent electrolyte and protective coating of the back surface of the anode.

The most significant factors in obtaining $+20^{\circ}$ C reserve cell performance were the addition of sulfur to the HgSO₄ cathode, the partial use of spent electrolyte from previously discharged cells, and the inhibition of inactive anode surface. Considering the effectiveness of these techniques, a re-examination of the use of spent electrolyte at lower temperatures may be found in Table XVII, page 33.

3.10 meta-Dinitrobenzene Cells

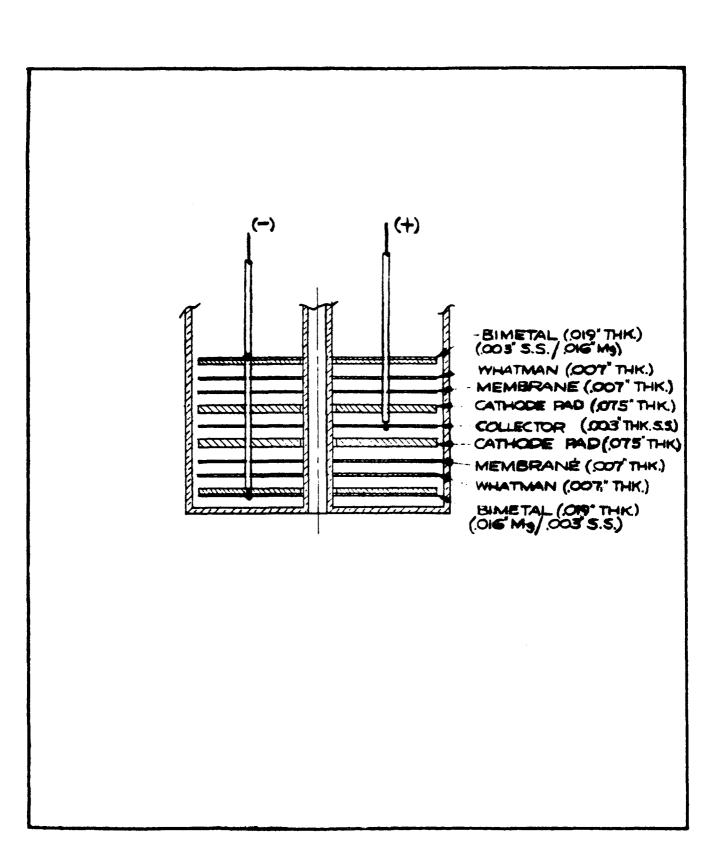
The meta-Dinitrobenzene (m-DNB) cathode was tested in flat plate, compressed ring, and spiral flat plate structures to reduce cathode current density and to provide for convenient installation of ion exchange membranes. These structures are described in Figures 2, 3, and 4 on the following pages. The bobbin cell structure was also checked using m-DNB even though past experience had indicated that this configuration would not perform efficiently at the higher drain rate of the cyclic load. m-DNB was also combined with other oxidants and some wet stand tests were conducted. The bulk of the data on these cells may be found in Appendix B, Table III, and the more notable results are singled out in this section of the report.

3.10.1 Flat Plate Configuration

Two of the best flat plate cells gave the following results:

Cell	Temp.	Ion Exch.	Hrs.to	F/Mol
No.	<u>°C</u>	Membrane	<u>1.5V</u>	Oxidant
2319	-55	SB-6407	70	1.36
2396	-73	None	44	0.97

In a similar configuration with an AMF-104 anionic membrane, four faradays per mol were achieved at ambient temperature, but the efficiency was poor at low temperature.



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ASSEMBLY DRAWING OF A-622 FLAT-PLATE CELL

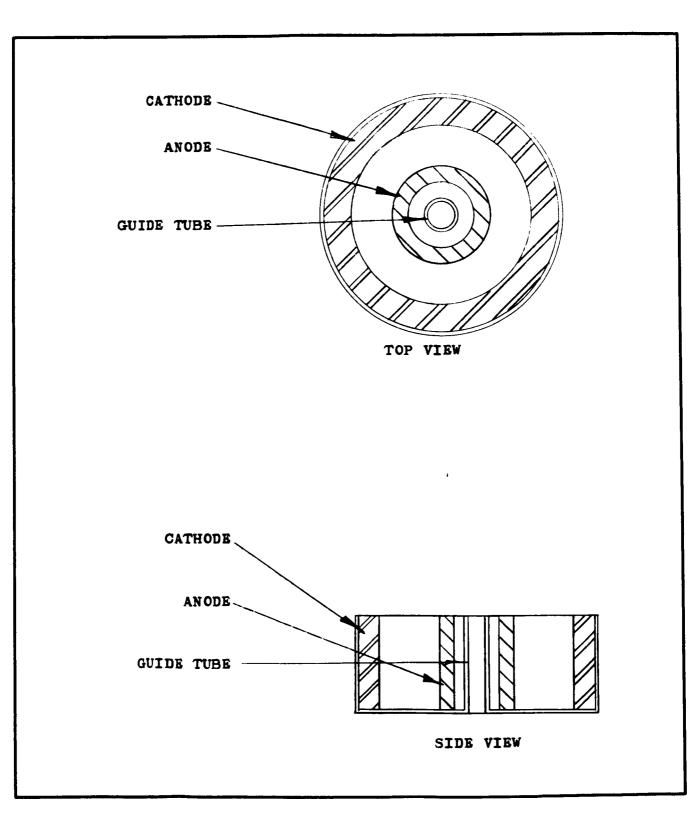


FIGURE 3

COMPRESSED RING BATTERY

26

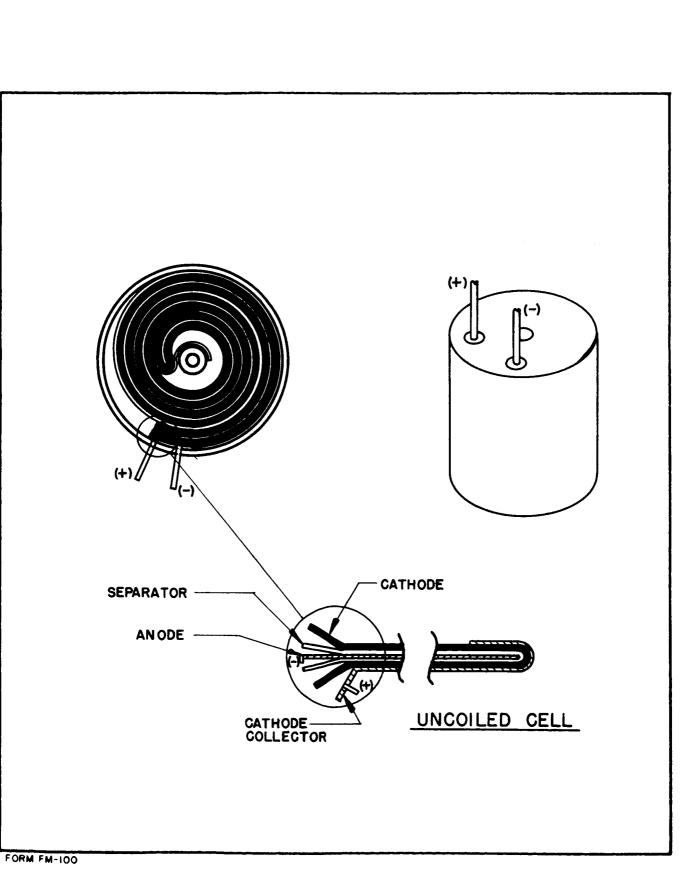


FIGURE 4

3.10.2 Spiral Wrapped Cells

The performance of this type of cell is shown in Table III of Appendix B. The 72 hour goal was exceeded at low temperature. However, at +20°C, life to 1.5V was reduced to approximately 1 day; life to a 1.3V cutoff increased to 3 days. Despite the relatively large voltage variation and structural complexity of these cells, duplicate runs showed a substantial degree of reproducibility as will be presented in Section 6. Inasmuch as the +20°C performance required a 1.3V cutoff, the hope of obtaining wet stand in these structures appeared less favorable than with mercuric sulfate and sulfur. See Figure 4, page 27.

3.10.3 Compressed Ring Cathodes

While the compressed ring configuration provides increased cathode current density in many applications, fairly low cathode current density is achieved with the inverse ring configuration, as shown in Figure 3, page 26. In this battery design an increased cathode surface area is obtained at the expense of anode area. Thus, even though a considerable amount of active material can be compressed into the cathode to prolong performance, reasonable cathode surface area is retained. It is significant that the compressed ring configuration and the battery design illustrated lend themselves to effective utilization of ion exchange membranes. Results of the following four tests showed that the use of the anionic membrane produced no effect on performance at room temperature, but doubled the hours to end voltage at -54°C.

Compressed Ring Cathode

m-DNB:C = 2.50:1.67 Drain Rate = 12 mA End Voltage = 1.44

IE	Temperature	Hours to
Membrane	°C	End Voltage
None	+20	56
None	- 54	18
SB 6407	+20	56
SB 6407	- 54	36

These results suggest that at low temperature the anionic membrane prevents electrode polarization caused by precipitation of a partially soluble compound at the small anode.

The suggestion is based on the premise that the partially soluble secondary compound is distinctly less soluble at low temperature. Consequently, these anions would tend to be precipitated as the magnesium salt and block the anode unless contained by an ion exchange membrane.

Due to greater progress in other areas, this line of approach was put aside. Additional basic data on ion exchange membranes could change this balance of factors.

3.10.4 meta-Dinitrobenzene Bobbin Cells

Bobbin cells containing 30 grams of m-DNB and 90 grams of carbon were discharged at -63°C and after 70 hours of wet stand at -63°C. Without wet stand, the life to 1.5V was 22 hours, and 70 hours of activated stand reduced the closed circuit voltage of the cell to below cutoff. It was considered evident that m-DNB showed little promise in this type of construction. Mixtures of m-DNB and sulfur were also tested but the m-DNB alone was superior to the mixtures. m-DNB was used as an additive in a number of other bobbin cells and these data are summarized in Table XV.

TABLE XV

Cell <u>No.</u>	Hg SO 4 grams	PbSO ₄ grams	m -DNB grams	S grams	C grams	Hrs.to <u>1.5V</u>	Hrs.to <u>1.3V</u>
Т-60	106	0	0	20	60	106	119
T-110	106	0	10	20	60	106	121
T-111	106	0	20	0	60	89	102
T-112	0	108	10	20	60	34	104
T-96	0	108	0	20	60	34	53

Effects of m-DNB on Other Oxidants

In some cases meta-DNB appears to be a reasonable substitute for sulfur.

In general, it may be concluded that m-DNB is a satisfactory oxidant at the lower temperatures, especially when used in the spiral-wrapped configuration; but it does not provide sufficient life under heavy load to a cut-off voltage of 1.5 when discharged at a temperature of approximately 20°C. It does, how-ever, run for long periods of time between 1.5 and 1.3 volts.

4. ELECTROLYTE STUDIES

The design center electrolyte selected for this development program was 25 weight percent KSCN in anhydrous liquid ammonia. After the major effort was centralized around Mg/HgSO₄:S:C type of cell, the chemical composition of the electrolyte after complete discharge of the cell became of considerable importance.

TABLE XVI

Chemical Analyses of Spent Electrolyte

Spent Electrolyte from Cell T-62: (1)

10 cc (approximately 9 grams) spent electrolyte added to 200 grams of water

	grams/9 grams sample
Mg ⁺⁺	0.17
$\rm NH_3$, as $\rm NH_4SCN$ from reaction of $\rm Mg(SCN)_2$,	
$NH_3 + H_2O$	0.24
NH ₃	4.29
SCN	0.92*
TOTAL	5.62

(2) Spent Electrolyte from Cells similar to T-62

SCN⁻ equivalent to 0.702 g Mg = 3.35 g

			grams/43.5 grams sample
	Mg ⁺⁺		0.702
	NH3, as NH4SCN from reaction of NH3 + H2O NH3	Mg(SCN)₂,	0.982 33.39
	SCN ⁻ + (SCN) ₂ or related compount than S ⁼ and/or CN ⁻	nds other	8.40**
	TOTAL		43.474
K CN	none (by flame photometer) none (by X-ray)	Sulfide Polysulfide	none (by X-ray) none
*	SCN ⁻ equivalent to 0.17 g Mg = 0.81	g	

**

Of major significance is the absence of potassium ion in the spent electrolyte. This means that all potassium ion originally supplied in the 25 weight percent KSCN electrolyte was probably precipitated within the cathode structure as insoluble K_2SO_4 . XNH₃. It seemed logical, therefore, to test cells with $Mg(SCN)_2NH_3$ electrolyte and mixtures of $Mg(SCN)_2$ and KSCN in ammonia. Since anhydrous $Mg(SCN)_2$ is not commercially available and since $Mg(SCN)_2$. $4H_2O$ is most difficult to dry^1 , two other techniques were employed. First, spent electrolyte from previous similar cells and blends of fresh and spent electrolyte were used to activate $Mg/HgSO_4$:S:C cells. Secondly, $Mg(SCN)_2$ solutions were prepared in liquid ammonia via the reaction:

 $Mg + 2NH_4SCN \longrightarrow Mg(SCN)_2 + 2NH_3 + H_2$

Since it is questionable whether this reaction goes to completion, several methods were employed to insure more complete conversion of this "synthetic" spent electrolyte. Magnesium sulphate was added to both the electrolyte and to the cathode bobbin in order to precipitate the rather insoluble $(NH_4)_2SO_4$ by means of the sparingly soluble MgSO₄. A second method of completing the reaction was the electrolysis of the synthetic spent electrolyte using a magnesium anode and an iron cathode. The end point used was the generation of a tenacious blue coloring resulting from the presence of electrons. Results of cells tested with these electrolytes are given in Table XVII.

At -63°C only a slight loss in capacity to 1.5 V occurred when 50% spent electrolyte was used. On the other hand, the capacity for this cell structure increased beyond 72 hours at +20°C using 50% spent electrolyte. The use of magnesium sulfate appeared detrimental to the operation of the cell but electrolysis resulted in satisfactory performance.

The effect of variation in the concentration of KSCN in the starting electrolyte was also given preliminary examination. The higher concentration of 50 weight percent appears suitable and the lower concentration of 10 weight percent fell below 72 hours.

¹ Magnesium chloride cannot be desiccated by ordinary means since the basic chloride and HCL gas result. This is overcome commercially by desiccation under an anhydrous HCL atmosphere. Anhydrous HSCN is not stable and, hence, this path cannot be employed to obtain anhydrous Mg(SCN)₂.

Electrolyte Code:

- A 25 weight percent KSCN in liquid ammonia
- B 50 percent spent electrolyte from similar discharged cell, plus 50 percent of (A)
- C liquid ammonia
- D 80 percent spent electrolyte plus 20 percent liquid ammonia
- E 10 weight percent KSCN in liquid ammonia
- F 12.5 weight percent $Mg(SCN)_2$ in liquid ammonia; saturated with $MgSO_4$
- G 50 percent of (A) plus 50 percent of (F)
- H 50 percent of a 17 weight percent solution of Mg(SCN)₂ in liquid ammonia plus 50 percent of (A)
- I 25 weight percent KSCN in liquid ammonia was electrolyzed 3 minutes using two Mg electrodes at 0.75 volts
- J 50 weight percent KSCN in liquid ammonia
- K 5.6 weight percent NaBr in liquid ammonia
- L 34 weight percent KSCN in liquid ammonia

TABLE XVII

$\frac{\text{Electrolyte Comparisons - New, Spent and Synthetic Spent}}{\text{HgSO}_4 + 20 \text{ grams Sulfur}}$

	<u></u>	Electro	lyte						
Test Cell#	New <u>%</u>	Spent%	<u>Synth</u> #1 <u>%</u>	<u>#2</u> <u>%</u>	Code	Cathode Additive	Temp <u>°C</u>	Hrs.to 1.5V	Hrs.to <u>1.3V</u>
Т-60	100				А		-63	106	119
т-67	50	50			В		-63	101	109
T-74	0	100			D		-63	78	87
T-81			100		F	l0 gram MgSO4	-63	49	57
T - 87	50			50	Н	6 grams acet.blk.	-63	85	93
T-61	100				А		-73	85	102
T - 72	50	50			В		-73	67	75
т-62	100				А		+20	30	91
T-90	100				А		+20	52	93
T-68	50	50			В		+20	76	92
T-91	50			50	Η		+20	60	86
T-87	50			50	Н	6 grams acet.blk.	-63	85	93
T-85	100				А	6 grams acet.blk.	-63	79	95
T-93	elect: in L.	rolyzed : NH3:	in KSC	N	I		-63	75	89
T-101	50 W L.NH	t. % KSC: I3	N in		J		-63	103	108
T-102	10 W L. NH	t. % KSC I ₃	N in		E		-63	52	60

4.1 Chemical Studies of Cell Gas

Chemical analysis was also made of the gases resulting from two discharged cells. The initial electrolyte in T-62 was a 25 weight percent solution of KSCN in liquid ammonia. That in T-68 was a 50/50 mixture of 25 weight percent KSCN in ammonia and spent electrolyte from a similar discharged cell. The back of the anode in the second cell was coated. The results of these analyses were as follows:

	T-62 % by Vol.	T-68 <u>% by Vol.</u>
NH3	68.2	65.7
H_2	12.4	10.5
N_2	<u>19.4</u>	23.8
	100.0	100.0

Nitrogen and hydrogen are the significant components in these data and the predominance of nitrogen, coupled with its valence of three, accounts for the loss of a substantial portion of the cathode capacity. The significance of the gas analyses is also considered in Section 5.1, page 42, of this report.

The net result of these experiments indicates that considerable benefit can be derived from spent electrolyte at +20°C, but 50 percent spent electrolyte slightly decreased the performance at -63°C and more so at -73°C. Optimum performance over the range +20°C to -73°C is interpolated in the region of 25 percent spent electrolyte.

4.2 Concentration of KSCN Electrolyte

The following excerpts from Table V of Appendix B, pages 45 and 46 illustrate the relative performance of $KSCN/NH_3$ electrolytes in concentrations of 10 percent, 25 percent, and 50 percent. Ten percent KSCN was unsatisfactory, but both 25 percent and 50 percent provided long cell life, although the latter generated voltage at a much lower level during most of the high load portion of the cycle.

Taken from Table V of Appendix B

Test	%	Hi	gh Load V	Voltage aft	Hours to	Hours to	
<u>No. T-</u>	KSCN	l day	2 days	<u>3 days</u>	4 days	<u>1.5V*</u>	1.3V*
102	10	1.82	1.59			52	60
60	25	2.15	2.10	1.95	1.55	106	119
101	50	1.70	1.81	1.75	1.58	103	108

See Table XVII, page 33 for cell variables.

* under heavy load

In a series of tests in Livingston commercial hardware, three cell, 6 volt, $HgSO_4$ batteries were discharged at 20°C with KSCN:NH₃(1.) electrolytes of several concentrations. The current drain on 4 gram cathodes was at a nominal 300 mA rate. The life of each battery to a 10 percent voltage drop from the peak is as follows:

8	Weight	Percent	KSCN	5	minutes
16	Weight	$\mathbf{Percent}$	KSCN	27	minutes
24	Weight	Percent	KSCN	45	minutes

Further discussion of electrolyte concentration may be found under "Theoretical Considerations" in the section on "Activated Stand," page 42.

Electrolyzed KSCN:NH₃ and Hg(SCN)₂ with Spent Electrolyte 4.3

In order to inhibit anode corrosion, an attempt was made to assure alkalinity by passing a current through a 25 weight percent KSCN solution in liquid ammonia, using two Mg electrodes. A potential of 0.75 volts was applied for three minutes. Gassing and a deep blue coloration which faded into a gelatinous white floc were observed. This mixture was used as "electrolyzed" electrolyte in Cell No. T-93 which performed reasonably well as shown in Table XVII, page 33.

Table XVIII shows that the more concentrated $Mg(SCN)_2$ /liquid NH₃ electrolyte, (H), was superior to the more dilute (G), which also contained MgSO₄ at the 10 gram of sulfur level which may be preferable for -73°C.

TABLE XVIII

Electrolyte Comparisons - New vs Synthetic Spent HgSO₄ + 10 grams Sulfur at +20°C

		Elec	trolyte				
Test <u>No.</u>	New _%_	Synt #1 <u>%</u>	hetic #2 %	Code	Hrs. to <u>1.5V</u>	Hrs. to <u>1.3V</u>	
T-83 T-88	50 50	50	50	G H	49 67	76 78	

 $Hg(SCN)_2/S$ cells performed almost equally well at -63°C with either new or 50 percent spent electrolyte.

TABLE XIX

Electrolyte Comparisons - New vs Spent

 $Hg(SCN)_2 + 20$ grams Sulfur at -63°C

		Electrolyte	_			
Test	New	Spent	Code	Hrs. to	Hrs. to	
<u>No.</u>	%	<u>%</u>		<u>1.5V</u>	1.3V	
T-70	100	50	A	63	74	
T-78	50		B	60	70	

4.4 Pre-treatment of Cathode Mix with 25% KSCN/liquid NH₃

The cathode mix contained in cells T-69 and T-75 was pretreated with 25 percent KSCN/liquid NH₃ electrolyte; after which the NH₃ was evaporated and the mixture pulverized. The pulverized mixture was then used to build bobbin cathodes. Since the combined weight of the solids did not increase as a result of the treatment, it is assumed that none of the NH₃ coordinated with the HgSO₄. This leads to the further assumption that all of the HgSO₄ must have reacted in some form with the KSCN. Subsequent cell tests with this material were not encouraging.

4.5 NaBr:NH₃ Electrolyte

The negative results obtained above in Section 4.4, combined with the theoretical considerations which were concurrently being evolved in Section 5.1, led to experiments concerning the use of salts other than KSCN. An electrolyte comprising 5.6 weight percent NaBr was tried in Mg/HgSO₄ cells¹ and while 67 hours was obtained at -63°C, the ± 20 °C cell did not perform well. Since stability at the higher temperature was being sought, studies with this electrolyte were discontinued in favor of other more promising areas.

4.6 Freezing Points of Solutions

Measurements were made of the freezing temperatures at different concentrations of KSCN in liquid ammonia, and the results were plotted, Figure 5, page 39. At -95°C, the liquid range was between 35.5 and 36 weight percent of KSCN. At -90°C, the liquid range extends from 33 to 37 percent of KSCN. The eutectic solution was estimated to be approximately 36 percent KSCN. The range covered was from 20 percent to 47.5 percent KSCN in liquid ammonia.

The addition of a drop of distilled water to simulate the action of humidity caused no observable change in the freezing temperatures of the solutions. The composition frange covered was the same as above.

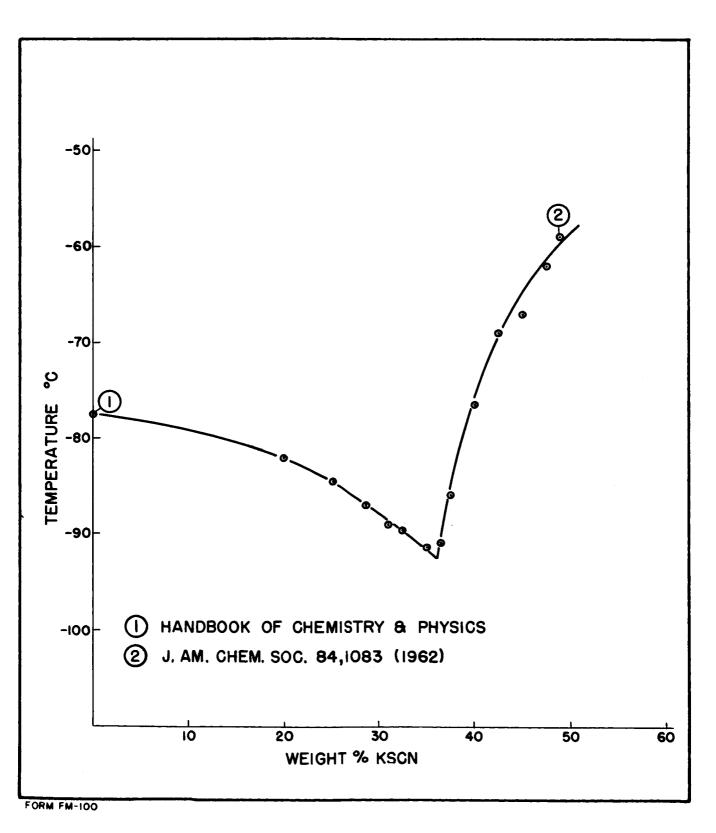
Freezing points were determined for several concentrations of spent electrolyte from $Mg/HgSO_4$:S cells, and the results are shown graphically in Figure 6, page 40. Concentrations above 25 percent were obtained by the evaporation of the liquid ammonia. The curve up to the eutectic point (approximately 36 percent solids) is almost identical with that of unused KSCN electrolyte, a most important factor in the operation of cells at -90°C. At the higher concentrations, a minor additional phase was observed. It was a very fine suspension which disappeared at higher temperatures.

⁴ Appendix B, Table VI, Cells T-116 and -120

4.7 Conductivity of Electrolytes

Figure 7, page 41, shows the effects of temperature and concentration of salt on the conductivity of solutions of KSCN in liquid ammonia, as well as the freezing point diagram of the same electrolyte. It shows a concentration of about 34 weight percent of KSCN to be optimum at -90°C from the standpoint of conductivity and freezing point. There seems to be a definite relationship between the conductivity values and the freezing point at the eutectic concentration of 36 percent.

The conductivity of a saturated solution of $MgSO_4$ in liquid ammonia at -80°C was almost the same as that of pure liquid NH_3 .



FREEZING POINT DIAGRAM FOR L. NH₃-KSCN SOLUTIONS

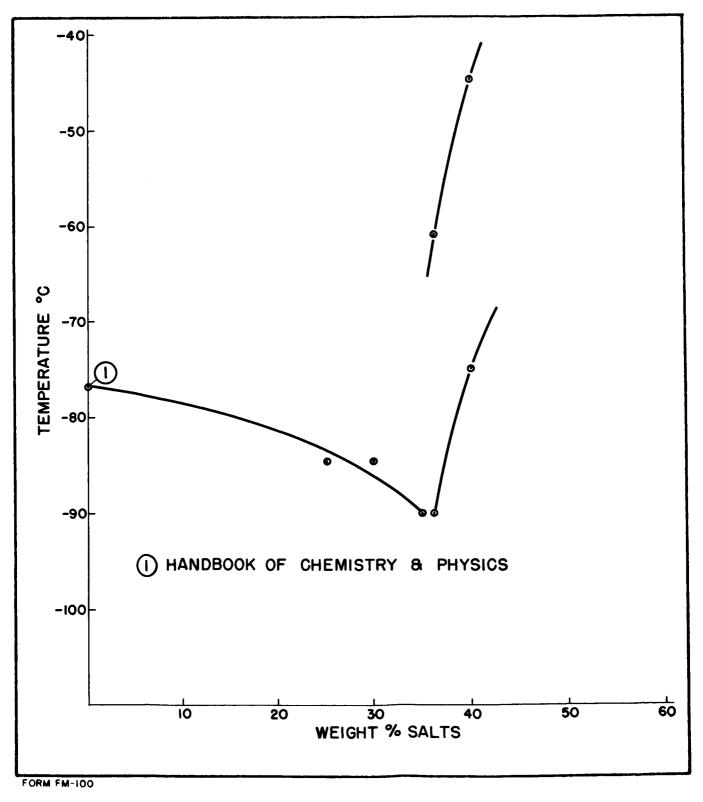
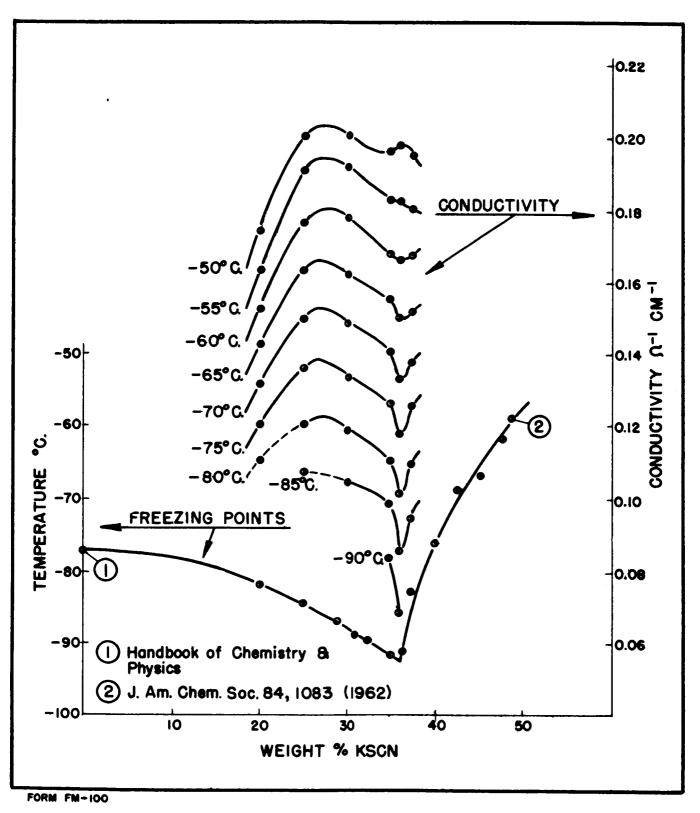


FIGURE 6

Freezing Point Diagram of Spent Electrolyte

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CONDUCTIVITY AND FREEZING POINT DIAGRAMS OF LIQUID AMMONIA/KSCN SOLUTIONS

5. ACTIVATED STAND

Goal II of this contract is aimed at limited wet stand to +70°C.

5.1 Theoretical Considerations

The theoretical aspect of the wet stand characteristics of the $Mg/HgSO_4$ and $Mg/PbSO_4$ cells, using KSCN : liquid NH₃ electrolyte, has been examined in some depth, and tentative conclusions have been drawn.

Five gram portions of HgSO₄ were placed in each of three test tubes. The first contained 10 weight percent KSCN, the second 25 weight percent KSCN and the third 50 weight percent KSCN, all in liquid ammonia. After approximately 16 hours, the material in the first tube had converted from the essentially coarse crystalline material into a fine white bulky floc, a characteristic of maximum coordination with liquid ammonia. The third tube containing 50 weight percent KSCN contained a layer of fine white crystals overlaid by a substantial thickness of black amorphous powder. Subsequent analysis by X-ray indicated the contents of the third tube to include pure mercury (fine black solid at -50°C), K_2SO_4 , KSCN, NH₃ and other unidentified materials. The contents of the black deposit. An equilibrium involving HgSO₄ and KSCN yielded K_2SO_4 and Hg° plus oxidized species stemming from ammonia and the thiocyanate ion.

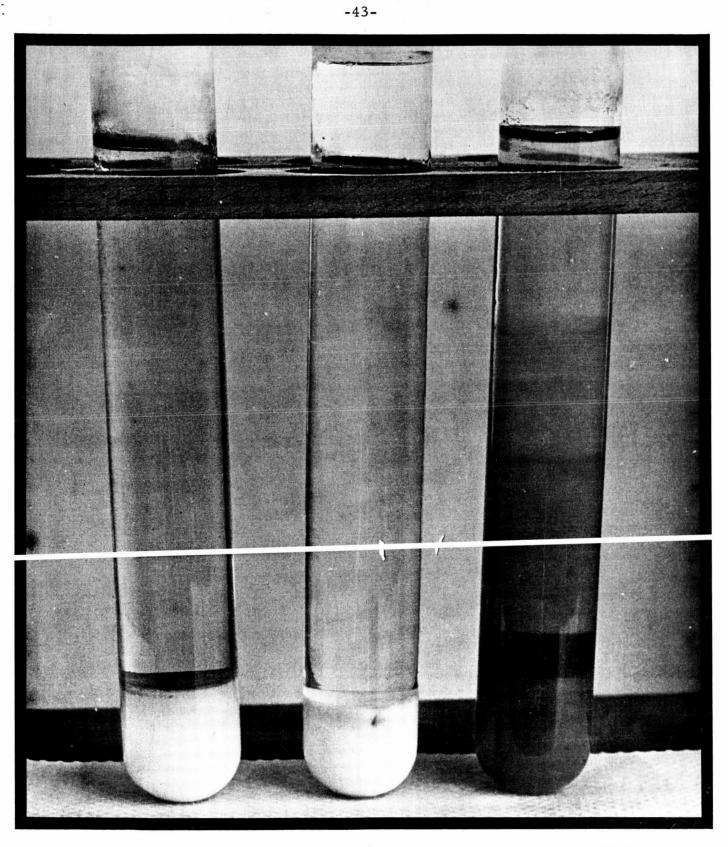
The test tubes used in this experiment were photographed and this picture has been produced as Figure 8, page 43. Thiocyanogen is known in aqueous chemistry from the oxidation of thiocyanic acid and electrolysis in the alcoholic solutions.¹

Thus, the primary limiting factor to wet stand in this type of ammonia cell appears to begin at the cathode reaction mechanism by the oxidation of the thiocyanate ion at cathode potentials in the order of 2 volts, reference magnesium. This is followed by evolution of nitrogen and accelerated corrosion of the anode due to the soluble oxidation product of the thiocyanate ion.

Yost and Russell² comment upon this point, and state, "Some of the reactions are noteworthy because a more noble metal can sometimes be made (to) replace sodium, the reason for the reversal being due to the formation (of) very insoluble salts of the noble metal." This point is further emphasized by the effect

¹ Sneed, Maynard and Brasted, <u>Comprehensive Inorganic Chemistry</u>, Page 226, Vol. III, D. Van Nostrand Co., Inc., N. Y., 1954.

² Yost and Russell, <u>Systematic Inorganic Chemistry</u>, Page 153, Prentice-Hall, Inc., 1946.



25%KSCN

50%KSCN

EFFECTS OF KSCN/LIQUID AMMONIA SOLUTIONS ON ${\rm HgSO_4}$

FIGURE 8

of magnesium upon Teflon. Teflon is known to be subject to attack by molten alkali metals and experience at this laboratory extends this to include the alkali metal-ammonia solutions.

In view of the equilibrium pressure cited above for the operating cell it would appear that the hydrogen generated at the anode diffuses to the cathode where an inter-action between hydrogen and the oxidizing agent can further reduce the capacity of the cell. This cyclic and inter-related chain of events must, of course, be interrupted if we are to achieve wet stand capability in this system. The experiments subsequently described constitute preliminary attempts to break this cycle of local actions.

Sneed, Maynard and Brasted³ give an oxidation potential series for the halides (including pseudo-halides) where the potential for the formation of $(SCN)_2$ is given as -0.77 volts. The bromide to bromine reaction is listed at a potential of -1.07 volts.

For this reason, sodium bromide was used in place of KSCN. The cells gave fair low temperature performance but failed at room temperature. Further examination of the NaBr/NH₃ solution's freezing point showed many anomalies. For this reason a substitute for the mercuric sulfate was instead chosen for examination. Mg/PbSO₄ cells have been previously examined in the Corson Laboratory and are known to have greater stability, but less current handling capability, than the mercuric sulfate cells. In addition, the potential of PbSO₄ is about one-half volt less than HgSO₄. Three test tubes were prepared containing 10 percent, 25 percent and 50 percent KSCN in ammonia. Five gram portions of lead sulfate were added to each of the tubes. Aside from coordination reactions and the formation of an intermediate liquid phase containing the lead sulfate, no evolution of metal was observed after two weeks at -63° C. Chemical stability of the oxidizing value of lead sulfate in a liquid ammonia solution of KSCN is indicated. Consequently several lead sulfate cells were tested.

From the above evidence of stability of $PbSO_4$ towards KSCN:NH₃, the instability of HgSO₄, and the reactivity of sulfur towards pseudo-halides,⁴ it would appear that the role of sulfur in the Mg/HgSO₄ cells using KSCN electrolyte lies in the storage of the oxidizing value of thiocyanogen.

⁴ Sneed, Maynard and Brasted, ibid, Page 227

Sneed, Maynard and Brasted, ibid, Page 225.

Later experiments have included efforts to obtain low temperature current handling capability from various forms of the lead sulfate electrode including discharged negatives from aqueous lead-acid batteries as detailed in this report on pages 19 to 21. Substantial currents were drawn (at -50°C) from these electro-formed cathodes. The potential of the anode was also investigated in order to break the cyclic degradation pattern at the evolution of hydrogen. Passivation of the anode by coating with lead and zinc was attempted both in liquid ammonia cells and by aqueous techniques.

5.2 Experimental Investigations

These studies included (1) the chromate coating of Magnesium anodes, (2) vacuum-vapor deposition of lead on Mg, (3) the "tinning" of Mg surfaces by a proprietary process, (4) immersion coating of Mg with zinc, (5) electroplating Mg with Zn, (6) the formulation of the cell electrolyte as a plating solution, (7) coating the outer anode surfaces with rubber cement, (8) electrolyte modifications such as "spent" and synthetic $Mg(SCN)_2/NH_3$ solutions, (9) cathodic stabilization through the addition of sulfates, (10) modifications of cathode composition, (11) improved separation, (12) active stand tests under light drain, (13) analyses of spent electrolyte and gaseous cell products, (14) considerations of cell chemistry, (15) and the use of anionic and cationic ion-exchange membranes.

The experimental work, its results, and a discussion thereof are given in the following presentation.

5.2.1 Effects of Chromate Coating on Magnesium Anodes

A "wet stand" study of chemically treated and untreated magnesium anodes was performed in 25 percent by weight KSCN - liquid ammonia at -62°C. The purpose of these experiments was twofold:

- (a) To determine the passivation effect of chromate films on the corrosion rate of pure magnesium anodes.
- (b) To establish a method of increasing the wet-stand of magnesium in ammonia activated batteries.

This activated-stand investigation was performed on four single cells of $Mg/25\%KSCN-NH_3/HgSO_4.2NH_3-C:Ag$ for a period of twenty-one days at -62°C. Each cell was tested for its open circuit potential after activation; the open circuit voltages varied from 2.24 to 2.30.

The shelf life of ammonia-activated batteries or single cells is inversely proportional to the rates of the self-discharging reactions. These self-discharge reactions or "open-circuit losses" result in decreased electrochemical capacity. The exact relationship and interplay of the anode (Mg) and cathode (HgSO₄) in the self-discharge mechanism are currently unknown. However, several hypotheses are being considered to elucidate the primary reactions.

The rate of deterioration of magnesium anodes in KSCN-NH₃ solutions may be explained in terms of chemical and galvanic corrosion. In general, the dissolution rates of metals are determined by the composition and the properties of the films that form on the metal surfaces. Anodic processes involving the emergence of Mg++ ions from the metal lattices in contact with ammonia-KSCN solutions can be broadly divided, from the thermodynamic standpoint, into those in which the cation (Mg++) finally becomes solvated or complexed in solution such as [Mg(NH₃) ++] and those in which it becomes part of a solid phase [Mg(NH₂)₂]. Which type of process occurs depends on the thermodynamic properties of the particular metal/solution system, on the progress of the anodic self-discharge reaction and, in part, on factors involving the nature of the cathodic material. Generally, corrosion processes occur much faster if the system is such that the anodic product is freely soluble.

Procedure

Two sets (A and B) of magnesium anodes (2" width, 4" length, .008" thickness) were degreased in acetone and treated with a special chromate solution (CrO_3 - 12 grams/liter; $CaSO_4$. $2H_2O$ 8 grams/liter) by an immersion process. Both sets were rinsed in water and allowed to air dry. Afterwards, set "B" anodes were heated at 165°F for 24 hours. The control set (C) consisted of magnesium anodes containing their natural oxide-carbonate film. Anodes from sets A, B, and C were wrapped around 20-gram cathodes of mercuric sulfate - carbon (7:3 ratio). The individual open-circuit potentials were determined in 25 percent KSCN-NH₃ solution. The cells were placed in polypropylene bottles, covered with 25 percent KSCN-NH₃ solution, and allowed to stand for twenty-one days. Next, the cells were removed and placed in freshly prepared 25 percent KSCN-NH₃ solutions. Negligible open-circuit voltage was obtained. The extent of the self-discharge reactions was readily apparent upon examining the spent cathode, mixture. Metallic mercury in small bead-like form was distributed throughout the carbon matrix giving a speckled appearance.

Dissolution of the magnesium anodes in sets A B, and C was extensive. The chromate treatment gave fair protection to the underlying magnesium. The weight losses of the chromate-treated anodes were considerably less than the control set of untreated magnesium anodes.

There was no significant difference between the unheated chromate-treated anodes (Set A) and those heated (Set B) at 165°F.

Conclusions

The chromate passivation by the immersion process decreased the corrosion rate of magnesium in 25 percent KSCN-liquid ammonia solution at -62 °C. Loss of complete corrosion protective properties of the film was due to penetration of the electrolyte through film pores and defects, onto the magnesium surface. The electrochemical nature of magnesium and the magnitude of its corrosion potential in KSCN-NH₃ are probably contributing factors in the initial deterioration of the chromate film. The catalytic or inhibitor effect of chromate on the self-discharge of the cathode material is unknown. It has been postulated that in neutral (KSCN) ammonia solution, magnesium corrodes slowly, forming the insoluble magnesium amide $[Mg(NH_2)_2]$ and evolving hydrogen (H₂).

In general, the corrosion rate is primarily a function of the metal purity. Hence, relatively pure magnesium was used in these studies. Chemical analysis showed only 0.02 percent Cu, 0.001 percent Ni, and 0.20 percent other impurities.

5.2.2 Vapor Deposition of Lead on Magnesium

Magnesium anode stock was <u>vapor-deposited</u> with a thin coating of lead metal. Immersion of these test coupons in electrolyte resulted in a potential of onehalf of a volt, reference the $HgSO_4$ cathode, for periods ranging up to five seconds followed by a rapid rise to above two volts. Heavier coatings are apparently required.

5.2.3 Investigation of a Protective Coating of Zinc over the Magnesium Anode Surface

Because of the smaller activity of Zn metal compared to that of Mg, it would be expected that a thin Zn coating on a Mg-anode would considerably improve the wet stand of ammonia cells. The cell would have to be "activated" by stripping the Zn coating from the anode by some process such as applying the load or short-circuiting the cell with a fuze before its use.

The realization of this idea presented two problems. First, to coat Mg effectively with a uniform thin layer of Zn is a delicate procedure considering the difficulty of plating Mg; and secondly, to strip quickly and effectively this coating leaving the surface of the Mg anode free from Zn. As is well known, Mg cannot be directly electroplated because of an MgO layer which renders its surface inactive in alkaline baths. In acid baths, on the contrary, Mg is violently attacked and no electroplating is possible. Mg must, therefore, first go through a preliminary treatment in a pyrophosphate bath in which MgO is replaced by a thin layer of Zn (immersion coating) and as such becomes susceptible to further plating in cyanide baths by usual electroplating procedures. (Re: U. S. Patent 2, 526, 544)

It was thought at first that immersion coating might be sufficient protection to the Mg anode. This was not so. Several cells made of pure Zn and Zn-coated Mg electrodes (in 25 percent KSCN-NH₃) proved that immersion coating of Zn is far too thin to give a Zn coated Mg anode the potential of the Zn for more than a minute. This was also evident from the fact that immersion coated Mg could not be further electroplated with Zn in acid baths. The acids attacked the Mg easily through the immersion coating of Zn and at best a rather porous plating of Zn was obtained.

Electroplating in cyanide baths after the immersion dip finally gave a satisfactory answer. Zn-plated Mg made in this manner gave (in test cells with pure Zn) a negligible voltage for apparently an indefinite length of time. The Zn coating was very effective without being bright.

Different thickness layer platings were produced and tested as anodes in cells with $HgSO_4$ and $PbSO_4$ cathodes with 25 percent KSCN-NH₃ electrolyte (temperature -50°C). A typical result was the following:

On open circuit, the cell behaved as though there were no Mg present. After loading (25 ohms) and then placing on open circuit, the voltage returned to the initial value of approximately 1.3 volts. An attempt was then made to strip off Zn by applying a heavier load (6.6 ohms) but without results. The cell would return to the Zn voltage after opening the circuit. A still heavier load (1 ohm) made the subsequent open circuit voltage rise first slowly and then sharply to approximately 2 volts. This showed that Mg had been uncovered. This voltage, however, did not continue but instead kept decreasing which proves that there was no effective stripping of the Zn coating. It is believed that the stripping occurs over small areas of anode which induces large current densities which, in turn, cause polarization of these parts and hence reduce the voltage. In some cases, the voltage oscillated between approximately 2 volts (Mg voltage) and approximately 1.5 volts. This was interpreted as blocking of stripped areas by deposits from the cathode. These phenomena disappeared as a better separator between cathode and anode was introduced.

Conclusion: The Zn-plating of Mg anodes was not found useful at this time due to non-uniform stripping of Zn coating.

5.2.4 Formulation of Battery Electrolyte as a Plating Solution

In the research cell structure of Figure 9, page 50, a $Mg/PbSO_4$ cell was fitted with a zinc electrode as well as a magnesium anode. The structure consisted of a lead sulfate bobbin cathode surrounded by an expanded magnesium anode followed by a solid zinc sheet anode. Both anodes were coaxially situated around the circular bobbin cathode. The cathode was discharged via the zinc anode for the purpose of introducing zinc ions into the electrolyte. All voltage combinations of the three electrodes were continuously monitored by separate recording devices. It was hoped that the resulting electrolyte would comprise an ammonia zinc-plating solution, and that the magnesium anode potential would decay without load to the zinc level. No indication of the automatic formation of a protective zinc film was observed. Following discharge via the zinc anode, a separate power source was connected between the zinc electrode and the magnesium electrode. The passage of 1/2 ampere, Mg (-) Zn (+), for a short period had a negligible effect upon the potential. The current was increased in steps until 2-1/2 amperes was reached. At this current some indication of passivation of the magnesium anode was observed during subsequent open circuit.

The zinc electroplating current was then increased to 6 amperes and maintained at this level for one hour. Upon removal of the plating current the magnesium potential immediately decayed to 1/4 volt negative with respect to the zinc electrode. This passivation persisted for approximately 45 minutes under meter load of 3,000 ohms. After the 45 minute period, the magnesium electrode potential recovered in an erratic fashion and subsequently stabilized at the normal level. The process was varied three more times, and the decay of the magnesium potential to about 1/4 volt negative with respect to zinc was observed each time. The period of passivation tended to decrease with repetition. The repeated observation of anode passivation is considered favorable.

5.2.5 Activated Stand Cell Tests with HgSO₄

The activated stand cell tests with $HgSO_4$ are summarized in Table XX. In order to gain an insight into the relative effects of various factors, the initial wet stand tests were conducted at -63°C.

The first entry of Table XX is a typical example of the previous lack of wet stand capability for the Mg/KSCN:NH₃/HgSO₄ cell. After 4 days of wet stand, even at -63°C, no capacity remained in the cell to a 1.5V cutoff. The anode was badly corroded and covered with scale, and even a new anode did not rejuvenate this cell. A similar cell was prepared and the cathode only was subjected to wet stand. At the start of the discharge period, the anode was inserted, whereupon an immediate evolution of gas occurred and the anode turned black. The cell ran only 10 hours. Undesirable effects of the cathode upon the anode and the anode upon the cathode were indicated.

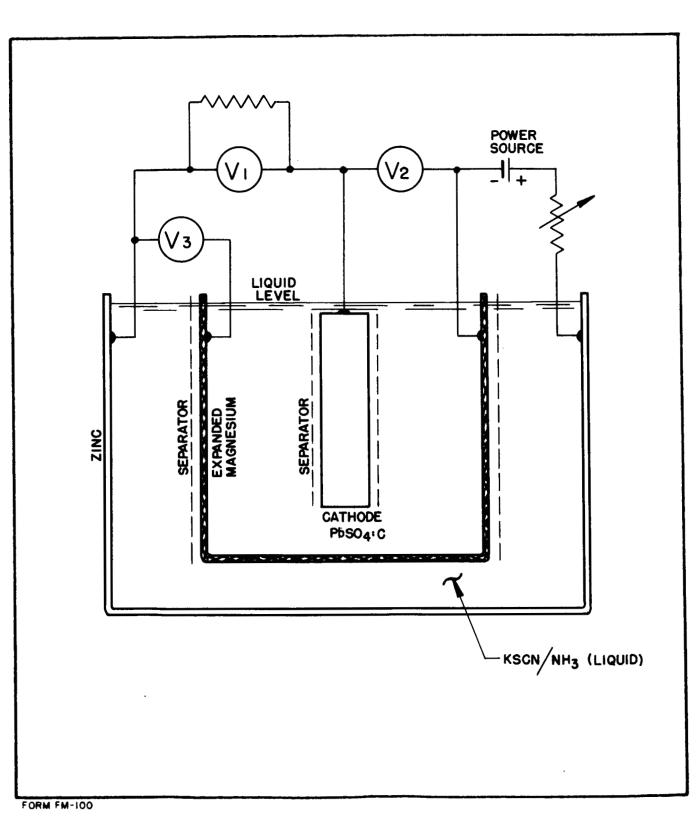


FIGURE 9

Formulation of Battery Electrolyte as a Plating Solution

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TABLE XX

Activated Stand

$Mg/HgSO_4$ and $Mg/HgSO_4{:}S$ Cells in $KSCN/NH_3$ (1) Electrolyte

Discharge Temperature = -63°C

Activated Stand Discharge								arge_	
	HgSO4	Coord.	S	С		Temp.	Load	Hour	s to
<u>r</u> -	gms.	No.	gms.	gms.	<u>Hrs.</u>	<u>°C</u>	Ohms	<u>1.5V</u>	<u>1.3V</u>
					•		O .C.	0	0
Co	mplete d	cell expos	sed to a	ctivated	stand.				
	140	2	0	60	96	-63	0 C	10	10.5
Ca					•	05	0.0.	10	10.9
	126	2	20	54	64	-63	0.C.	12	17
Ca	thode on	ly expose	ed to ac	tivated a	stand.				
_		2				-63	0.C.	10	16
Ca	thode on	ly expose	ed to ac	tivated	stand.				
	1.04	â	20	60	4.4	63	0.0	1 =	20
Ca									
Oa		ily expose	su to ac	livatcu				Dack w	itti i abbei
							e on.		
the	followin	ng tests th	ne comp	olete cel	ls wer	e expose	d to acti	vated s	tand:
		0	-			-			
	106	0	20	60	45	- 63	3000	30	39
Су	clic load	d applied	for 1 h	our afte:	r first	day's sta	and.		
		_		<i>.</i>	<i>.</i> –	(a			
	106	0	20	60	65	-63	3000	41	58
	1.04	0	20	60	65	63	2700	16	44
E1.						-05	2700	10	44
	courdigle	2 Wab 11a1.	- sherre	,	× • •				
	106	0	20	60	65	-63	645	41	50
	Ca Ca Ca Ca the	<u>I</u> - <u>gms.</u> <u>119</u> Complete of <u>140</u> Cathode on <u>126</u> Cathode on <u>120</u> Cathode on <u>106</u> Cyclic load <u>106</u> <u>106</u> <u>106</u> <u>106</u> <u>106</u> <u>106</u> <u>106</u>	$\underline{\Gamma} - \underline{gms.} \underline{No.}$ $119 2$ Complete cell expose $140 2$ Cathode only expose $126 2$ Cathode only expose $120 2$ Cathode only expose $106 0$ Cathode only expose the following tests th $106 0$ Cyclic load applied $106 0$ Electrolyte was hal	Γ -gms.No.gms.11920Complete cell exposed to a14020Cathode only exposed to ac126220Cathode only exposed to ac120220Cathode only exposed to ac106020Cathode only exposed to ac106020Complete cell exposed to ac106020106020106020106020106020106020106020106020106020106020106020106020106020106020106020106020106020106020	Γ -gms.No.gms.gms.1192051Complete cell exposed to activated1402060Cathode only exposed to activated12622054Cathode only exposed to activated12022060Cathode only exposed to activated10602060Cathode only exposed to activated10602060Cathode only exposed to activated10602060Cathode only exposed to activated10602060Cathode only exposed to activated10602060Cyclic load applied for 1 hour after1060206010602060106106106106020601061061061060206010610610610602060106106106106020601061061061060206010610610610602060106106106106020601061061061060206010610610610602060106106106106020601061061061060206010610	HgSO4Coord.SCIgms.gms.gms.Hrs.119205196Complete cell exposed to activated stand.1402060140206096Cathode only exposed to activated stand.1262205464Cathode only exposed to activated stand.1202206066Cathode only exposed to activated stand.1060206066Cathode only exposed to activated stand.1060206045Cyclic load applied for 1 hour after first10602060651060206065106020606510602060651060206065Electrolyte was half-spent, half new.6065	HgSO4Coord.SCTemp.Igms.No.gms.gms.Hrs.°C119205196-63Complete cell exposed to activated stand.140206096-63Cathode only exposed to activated stand.1262205464-63Cathode only exposed to activated stand.1202206066-63Cathode only exposed to activated stand.1060206066-63Cathode only exposed to activated stand.1060206045-63Cathode only exposed to activated stand.1060206045-63Cathode only exposed to activated stand; anodes c from her1060206045-63Cyclic load applied for 1 hour after first day's stationary1060206065-631060206065-631060206065-631060206065-631065-631065-63Electrolyte was half-spent, half new.10610206065-63106	HgSO4Coord.SCTemp.LoadI-gms.No.gms.gms.Hrs. $^{\circ}$ COhms119205196-63O.C.Complete cell exposed to activated stand.140206096-63O.C.Cathode only exposed to activated stand.1262205464-63O.C.Cathode only exposed to activated stand.1202206066-63O.C.Cathode only exposed to activated stand.1060206066-63O.C.Cathode only exposed to activated stand.1060206045-633000Cathode only exposed to activated stand; anodes coated on from here on.the following tests the complete cells were exposed to acti1060206045-633000Cyclic load applied for 1 hour after first day's stand.1060206065-632700Electrolyte was half-spent, half new.	HgSO4Coord.SCTemp.LoadHourT-gms.No.gms.gms.Hrs. $^{\circ}$ COhms1.5V119205196-63O.C.0Complete cell exposed to activated stand.140206096-63O.C.10Cathode only exposed to activated stand.1262205464-63O.C.12Cathode only exposed to activated stand.1202206066-63O.C.10Cathode only exposed to activated stand.1060206066-63O.C.10Cathode only exposed to activated stand.1060206066-63O.C.15Cathode only exposed to activated stand.1060206045-63300030Cathode only exposed to activated stand; anodes coated on back w from here on.from here on.1060206045-63300030Cyclic load applied for 1 hour after first day's stand.1060206065-633000411060206065-63270016Electrolyte was half-spent, half new.

TABLE XX

(continued)

						Act	ivated St	and	Disch	narge
Test		$HgSO_4$	Coord.	S	С		Temp.	Load	Hou	rs to
<u>No.</u> 1] -	gms.	No.	gms.	gms.	<u>Hrs</u> .	<u>°C</u>	Ohms	<u>1.5V</u>	<u>1.3V</u>
98		106	0	20	60	65	-63	3000	58	76
90	ЬA		micropoi					3000	56	10
		artronar			ssor sop		()			
103		106	0	20	60	65	-63	3000	61	88
	On	e side o	f MPR pa	rtially	masked	with ru	ubber cei	ment.		
1.0/		1.0/	~	2.0	(0		()	2700		74
106	-		0						64	74
	во	th sides	of MPR	partiall	y maske	d with	rubber o	cement.		
107		1.06	0	20	60	65	-63	2700	69	78
101	Do		er of MP		•••	00	•••		- /	
		1								
95		50	0	50	80	65	-63	3000	45	54
		(<i>.</i> –	<i>(</i> -		4.0	0.4
113			0			65	-63	3000	69	84
	M	1365 + G	elman SA	-6404 -	+ MPR					
114		1.06	0	2.0	60	65	-63	3000	0	3
	M		onics SB-			0.5	00	0000	Ū	-
114		106	0	20	60	65	-63	3000	36	82
	M	1365 + Io	onics SB-	210 + M	IPR (Dis	charge	e temp. i	ncreased	d to +20)°C).
100		1.0/	0	20	(0		120	2000	0	2
109			0 1rs to 0.8			65	+20	3000	0	3
	лa	.m 75 not	115 LO U.O	U VOICS	•					

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Many techniques were explored to improve the wet stand capabilities of the $Mg/KSCN/HgSO_4$ cells. These techniques included: additives, light drain, increased separation, ion exchange membranes, and combinations of these treatments. The best -63°C results obtained were from the $Mg/KSCN:NH_3/Hg-SO_4:S:C$ cell which gave 69 hours of life after 65 hours wet stand under light loading with a double layer of microporous rubber or a strong acid ion exchange membrane.

5.2.6 Activated Stand Cell Tests with PbSO₄

A life of 65 hours was obtained from the cell Mg/KSCN:NH₃/PbSO₄:C at +20°C without the use of coordination, additives, or dense separation. Considering the difficulties observed in increasing the wet stand capability with HgSO₄, the wet stand characteristics of PbSO₄ were examined as outlined in Table XXI. A number of additional cell tests gave poor results traced to erratic behavior of the rayon non-woven separator in the presence of PbSO₄. One cell, number T-134, gave 34 hours at -50°C after 65 hours wet stand at +20°C. This is as long as a fresh cell of this type delivered. While positive indications of wet stand were not actually accomplished by these exploratory tests, the data of Section 3.6 retains its implication of potential wet stand capability for the Mg/PbSO₄ cell.

TABLE XXI

Activated Stand at Room Temp. (20° to 30°C)

$Mg/PbSO_4$ Cells in KSCN/NH₃ (1) Electrolyte

					Acet.	Activate	ed Stand	Dis	scharge	<u> </u>
Test	F	PbSO ₄	Coord.	С	Black	Hours	Load	Temp.	Hour	s to
<u>No. 1</u>		gms.	No.	gms.	gms.		Ohms	<u>°C</u>	<u>1.5V</u>	<u>1.3V</u>
121		120	0	55	5	40	3000			
121	Ago		or wire co							
	0									
122		120	0	55	5	65	3000	+20	0	0
	Ran 77 hours from $1.17V$ at 13.6 ohms to $0.52V$ at 2.7 ohms.									
123	~ .	100	0			65	O .C.	-63	13	22
	Cath	node or	ily on acti	ivated s	stand.					
127		100	2	45	5	65	O .C.	- 50	0	0
Zn anode during stand: Mg anode during discharge.										
		- (<i>.</i> _	• •		24	20
134	9	86	2		0		O.C.	- 50	34	38
	Sepa	arators	s: M-136	5 Webr:	il + Mici	oporous	rubber.			
136		94	2	46	0	48	3000	- 50	0	28
	Initi	al 18 h	nours star	nd at - 5	$0^{\circ}C + 48$	8 hours a	t R. T. °			

6. **REPRODUCIBILITY**

With respect to reproducibility, four pairs of cells are listed in Table XXII which differ only with respect to slight variation of electrolyte weight and anode area. The average variation in life between pairs to 1.5 volts was 8 percent. Considering the small value of $\Delta V / \Delta t$ (the slope of the voltage time curve), this correlation is felt to be rather good.

TABLE XXII

Replicates

Outside Anode; Low Temperature Liquid Ammonia

T-183059430T-19305336211%T-3330733371%	tion ife
T-33 30 73 337 1%	0
T-3 5 30 72 377)
T-37 30 97 368 14% T-41 59 84 368)
B-4 33 21 671 5% B-6 33 22 671)

Average 8%

See, also, pages 2, 3, 4, 10, 14, 28.

7.

This last phase of the program was concerned with the exploration of ammonia battery operation below -73°C with the ultimate goal of operation at -90°C. Initially, a tentative design center cell:

$Mg/KSCN:NH_3/HgSO_4:S:C = 106$ grams: 20 g: 60 g

was selected for -90° C testing. The electrolyte studies of Sections 4.6 and 4.7 indicated that a 34 weight percent concentration of KSCN would be most advantageous from the standpoint of conductivity and freedom from solidification of both the starting and spent electrolytes. On the basis of these studies, a cell was constructed and tested with the 34 weight percent electrolyte and operation for 21 hours above the 1.5V cutoff was directly obtained. Variations and a number of exploratory tests were conducted on subsequent cells. The details are presented, cell by cell, following a Summary Table XXIII. One cell of particular interest was No. T-128 which was initially operated at -90°C for 35 hours to end voltages of 1.7 and 0.7 under light and heavy loads, respectively. The temperature was subsequently raised to -50°C, and the cell was operated for a cumulative total of 79 hours to 2.1/1.9 volts at which time the temperature was again reduced, but to -85°C, for further examination. Preliminary tests of lead sulfate at -85°C were also conducted.

TABLE XXIII

Test <u>No. T</u> -	U	Sulfate Coord. <u>No.</u>	S gms.	C gms.	Acet. Black gms.	KSCN: NH3 Wt. %	Temp. <u>°C</u>	Hrs.to	Hrs.to 1.3V	Load Ohms²
	1.0/									2 7/12 5
119	106	0	20	60	0	34	-90	21	27	2.7/13.5
126	100	0	20	55	5	35	-90	0	0	2.7/13.5
128	106	0	20	60	0	34	-90	0	0	2.7/13.5
132	18.9	0	3.8	11.3	0	34	-90	9	13	2.7/13.5
138	100	0	20	60	0	34	-90	7	19	2.7/13.5
138	100	0	20	60	0	34	-85	0	28	2.7/13.5
130	2.8	2	0	1.2	0	35	-90	2		47/220
		_	-		Ū		, -	_		
	$PbSO_4$									
	gms.									
139	87	2	0	43	0	34	-85	0	0	2.7/13.5
133	1.5	2	0	1.5	0	35	-85	9	9	47/220
		2								
135	2	2	0	1	0	35	-85	10	10	47/220
•										

Cell Performance at Temperatures Below -73°C

Cyclic load: Heavy load for 6 minutes; light load for 54 minutes.

Cell No. T-119

A Mg/HgSO₄:S bobbin cell, T-119, was discharged at -90°C. From the results of the freezing point determinations, an electrolyte was selected for this test containing 34 weight percent of KSCN in liquid ammonia.

The cell life was 21 hours to 1.5 volts. However, the light load voltage at this time was still 2.15; so the run was continued for an additional 44 hours to 0.8 volt under cyclic load. This was followed by 32 hours of operation with alternate 54-minute periods on open circuit and 6-minute periods under load to a cut-off of 1.47 volts under heavy load. Cathode polarization appeared to be the limiting factor.

The coulombic efficiency to this point was 25 percent based on $HgSO_4$ and sulfur and 68 percent based on $HgSO_4$, alone.

Next, the temperature was reduced to -95°C; and the cell was allowed to stand on open circuit for 24 hours. The final open circuit voltage was 2.20.

The liquid in this cell did not freeze at -95°C.

<u>Cell No. T-126</u> - Table VII, page51 of Appendix B. Compare with Cells T-119, 128 and 138.

The initial closed circuit voltage under heavy load was only 1.27, but the run was continued to investigate the effects of changes in loading and temperature. The following tabulation summarizes the cell performance:

Hours	Temp.		Voltage or		
<u>(Cumulative)</u>	°C	<u>O.C.</u>	13.5 Ohm s	2.7 Ohms	
0	-90	2.40	2.17	1.27	
89	-90	-	1.45	0.45	
96	- 50	2.30	2.20	1.95	
114	-90	-	1.25	-	Light load only
121	-90	-	1.20	0.42	-
159	- 50	2.30	-	-	O.C. only
161	-90	2.23	_	-	O.C. only
167	-90	-	1.40	0.65	-
238	-90	2.10	1.50	-	13.6 ohms for 6 mins.:
					O.C. for 54 mins.

The principal known difference between this cell and No. T-119 was the addition of acetylene black.

Hours	Temp.		Voltage on		
(Cumulative)	<u>°C</u>	0.C.	13.5 Ohms	2.7 Ohms	3
0	-90	2.38	-	-	
2	-90	-	2.15	1.28	
13	-90	-	2.15	1.64	Gradual Increase
15	-90	-	2.15	1.40	Gradual Decrease
17	-90	-	2.12	1.30	
35	-90	-	1.67	0.65	Gradual Decline
35 - 40	-90		(0.75-1.65)	0.40	Very Erratic
39	-90	1.96	-	-	
40-42 42-47	trode	t o deter		decay. Re	ith a Mg reference elec- sults: cathode polarization. 35 - 40 hours.
47	- 50	-	2.26	2.15	
91	- 50	2.15	2.05	1.88	
92					Reducing Temp. to -85°C
95	-85	-	1.62	1.35	-
114	-85	-	1.52	1.00	
140	-85	-	1.15	0.53	
140 - 165	-85	1.60	-	-	Open Circuit
166	-85	-	1.20	0.90	
208	-85	-	0.80	0.63	
209	-85	0.92	-	-	Open Circuit

Cell No. T-128 - Table VII, page 52, of Appendix B. Compare with Cells T-119, 126 and 138.

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Cell No. T-132

A pasted plate cathode was constructed as follows and used in Cell No. T-132 which was discharged at -90° C. HgSO₄, sulfur and carbon in a ratio of 5:1:3 were made into a paste with a mixture of one gram of polystyrene concentrate as a binder per 50 cc of toluene. The paste was spread onto a silver plated expanded copper grid (Exmet) and air-dried. The plate was enclosed in a double layer of SM-91 polypropylene non-woven fabric which was then heat-sealed. Two Mg sheets were used as anodes. The cell was enclosed in a polyethylene bag and placed in the refrigerator for testing.

Even though the cathode weight was less than 1/5 that of the usual 180 gram bobbins, the cell ran for 9 hours to 1.5 volts under the standard cyclic loads of 2.7 and 13.5 ohms; thereby sustaining five times the previous current densities (expressed as mA/gram) at -90°C. It is likely that the cell life could be extended greatly under the same loading if both the cathode weight and area were increased five times. The maximum flash current of this cell was 15 amperes.

In this flat plate construction, the cathode surface area is about 7 cm²/gram; whereas, the 180 gram bobbins have an external surface area of only 2 cm²/gram of cathode. The miniature **3** gram bobbins have a surface area of 5.4 cm²/gram. Further investigation of this factor seems to be warranted.

Cell No. T-138

This cathode was constructed with two collectors in order to study internal resistance at -90°C. The usual silver wire with a surface area of 11.0 cm² was inserted in the center of the cathode, and a silver Exmet (expanded metal) sheet having an area of 192 cm² was wrapped around the outside of cathode.

There was only a minor difference in voltage between the two collectors during discharge; 0.02 volts on light load and 0.08 volts on heavy load. A peak short circuit of 4 amperes was measured from anode to each collector. Further testing indicated no differences between collectors when connected separately or when slaved together.

Cell No. T-130

This cell did not run well at -90°C; so the temperature was raised to -50°C, after which it ran for 10 hours to a cut-off of 1.46 volts under heavy load. It is not certain whether the low yield was due to the omission of sulfur or the pre-ammoniation of the HgSO₄.

Cell No. T-139

This cell was designed to test a non-woven polypropylene separator. Its performance is covered under the discussion of $PbSO_4$ cells on page 20.

Cells T-133 and T-135

These miniature cells performed moderately well at -85° C. The faradic efficiency to the 1.5 volts cut-off which was obtained from T-135 in 10 hours is equivalent to that from T-125 (a full-sized bobbin) in 33 hours.

APPENDIX A

Test Hardware Refrigeration Instrumentation

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Test Hardware

Two basic types of reusable chambers (A-601 and A-622) were used in the majority of the cell tests conducted during the first three months of the program.

A-601

The A-601 test chamber is shown in Figure 1. The magnesium/mercuric sulfate bobbin cells tested in this vehicle occasionally met the requirements at -73°C, but a disheartening lack of reproducibility was attributed to the failure of the "O" ring seals. The effects of this leakage were, unfortunately, not always visibly apparent. Due to the previous failure of the mechanical refrigeration equipment to maintain -73°C, a liquid CO_2 box was being used for this temperature. While the cells did not require pressurization at the low temperature, absorption of CO_2 from the refrigeration atmosphere following leakage and loss of pressure was detrimental to the discharge characteristics in an erratic fashion.

The A-601 test chamber features a convenient, quick-access, screw-type closure, but it did not prove practical to utilize a pressure-type gasket or luting compounds; though many variations along these lines were attempted.

<u>A-622</u>

The A-622 chamber shown in Figures 2 and 3 utilizes a bolted top which allowed the substitution of a gasket in place of the "O" ring seals for which it was originally intended. Oversized Teflon seal rings were machined and used for this purpose. Adequate sealing was obtained at room temperature and -55°C. However, this vehicle was still unreliable at -73°C. Detail A of Figure 3 shows the capture cross-section of this seal. It was discovered that by assembling the top in a hydraulic press and tightening the bolts under force, a -73°C seal could be obtained.

In addition to the A-601 and the A-622 test chambers which were used for the majority of the tests, a third vehicle designated the A-606, was also used and is shown in Figure 4. The design is similar to the A-601, but the dimensions are different.

Evolution of the A-624

Continued study of the A-601 test chamber led to the conclusion that the comparatively large coefficient of thermal expansion of seal materials compared to the stainless steel container, made adequate closure much below -55°C most difficult. Typical coefficients of expansion are as follows: Silicone Rubber = 670×10^{-6} in. /in. /°F Butyl Rubber = 320×10^{-6} in. /in. /°F Fluorocarbons = 55×10^{-6} in. /in. /°F Stainless Steel = 10×10^{-6} in. /in. /°F

In view of the occasionally good performance of the magnesium/mercuric sulfate cell in this geometry, a new cell container was evolved as part of this program. Figure 5 describes this new test vehicle, designated as A-624. Preliminary hydraulic and pneumatic tests conducted on the seals utilized in this unique cell container indicated that it could withstand pressures in excess of 1,000 pounds per square inch at room temperature. Furthermore, as the temperature was reduced, the thermal coefficients involved act to great advantage.

The A-624 utilizes cross-linked polyethylene pipe with reusable stainless steel end plugs force-fitted.

The H-622

During the second quarter, the spiral wrap cells were tested which utilized the H-622 hardware shown in Figure 6. The container was designed for Livingston Electronic Corporation production batteries; it is hermetically sealed and not reusable.

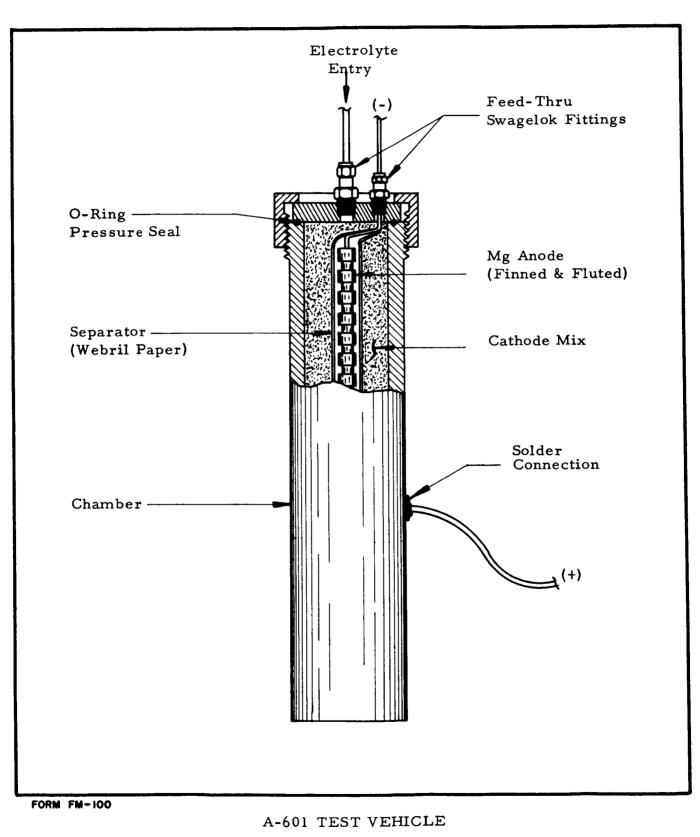
Test Tube (TT) Cell

The need to observe corrosion effects and gas evolution, in addition, to the ease of changing components led to the use of the Pyrex test tube. The cells were enclosed in test tubes of 1.8" I.D. x 16" and 2.4" I.D. x 20" (diameter controlled by polyethylene pipe inserted in the test tube). Miniature cells were tested in .875" I.D. x 12" test tubes.

This test tube was immersed in a circulating alcohol bath at temperatures down to -90°C. This provided an operating cell of the full diameter which could be visually examined periodically throughout discharge.

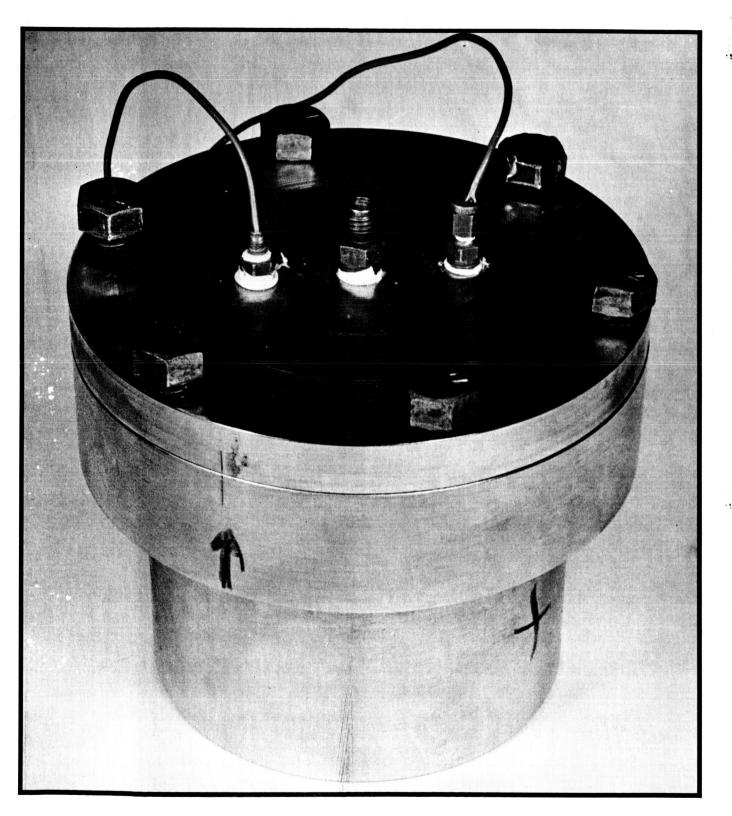
The use of this test vehicle gained considerable success in pin-pointing cell failures and is undoubtedly responsible for the long run times achieved in the latter months of the contract.

Photograph on pageAl0 Figure 7.



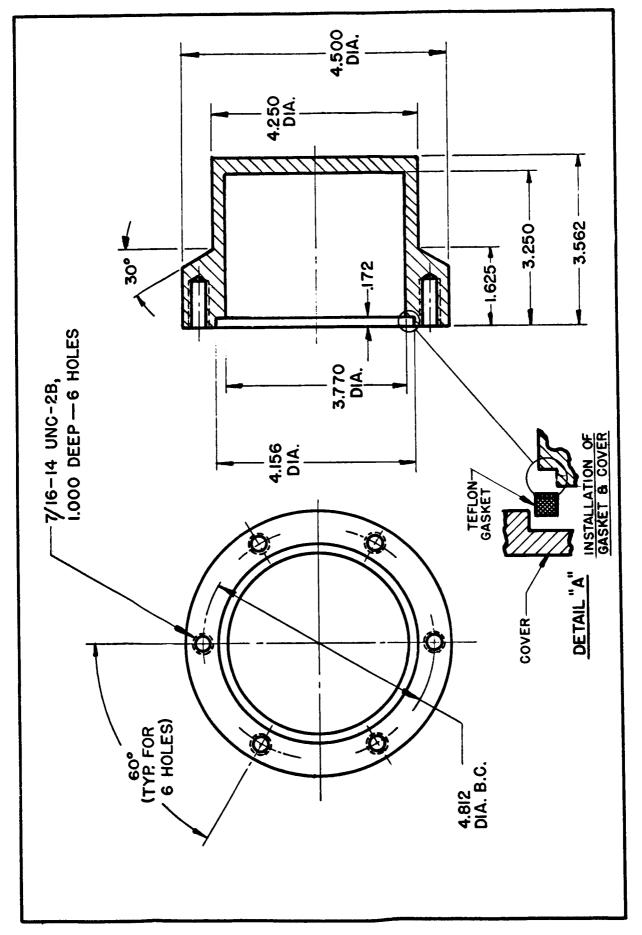


A_4



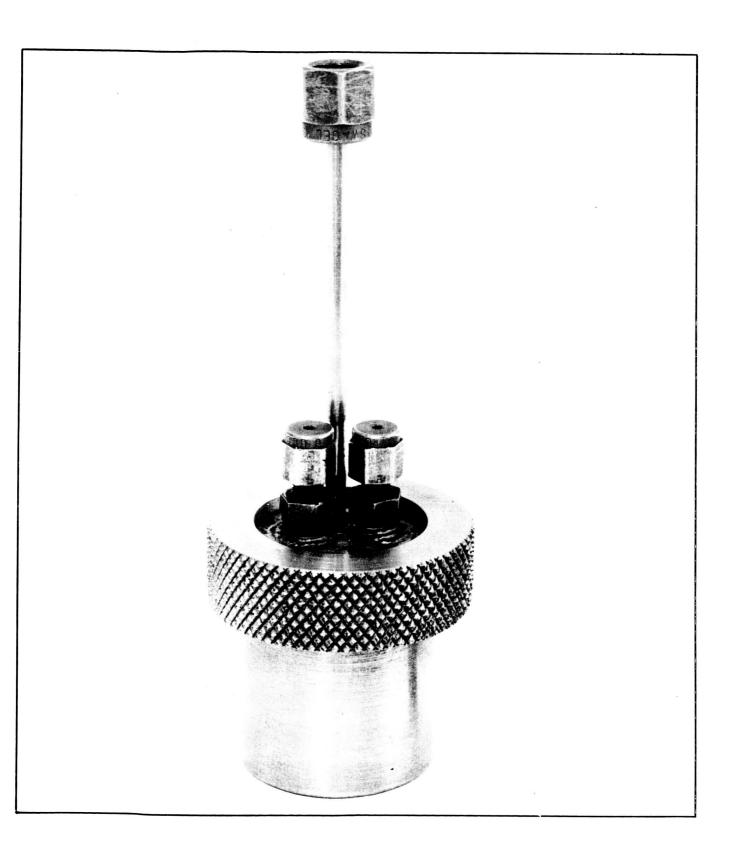
A-5

FIGURE 2

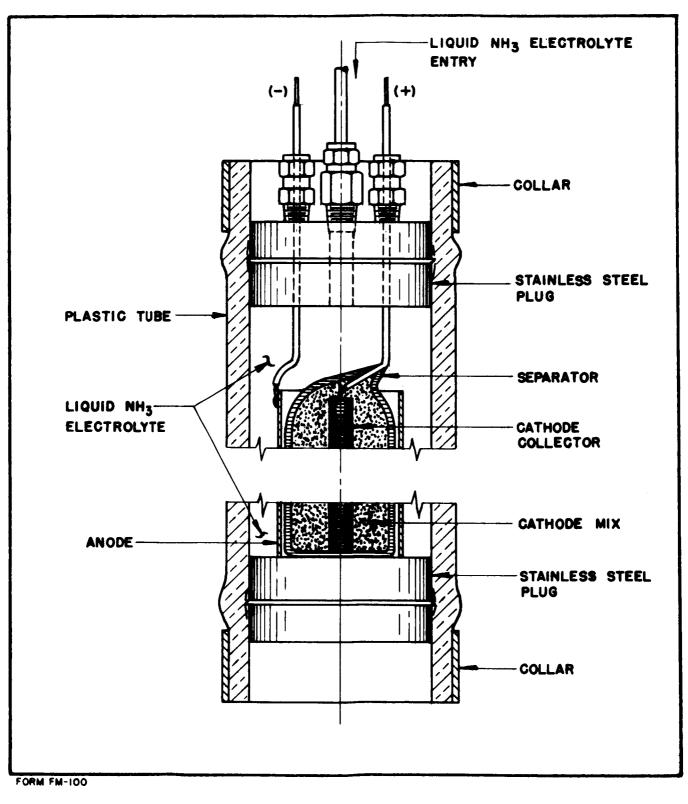


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A-622 TEST CHAMBER



A-606 TEST CHAMBER FIGURE 4



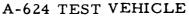
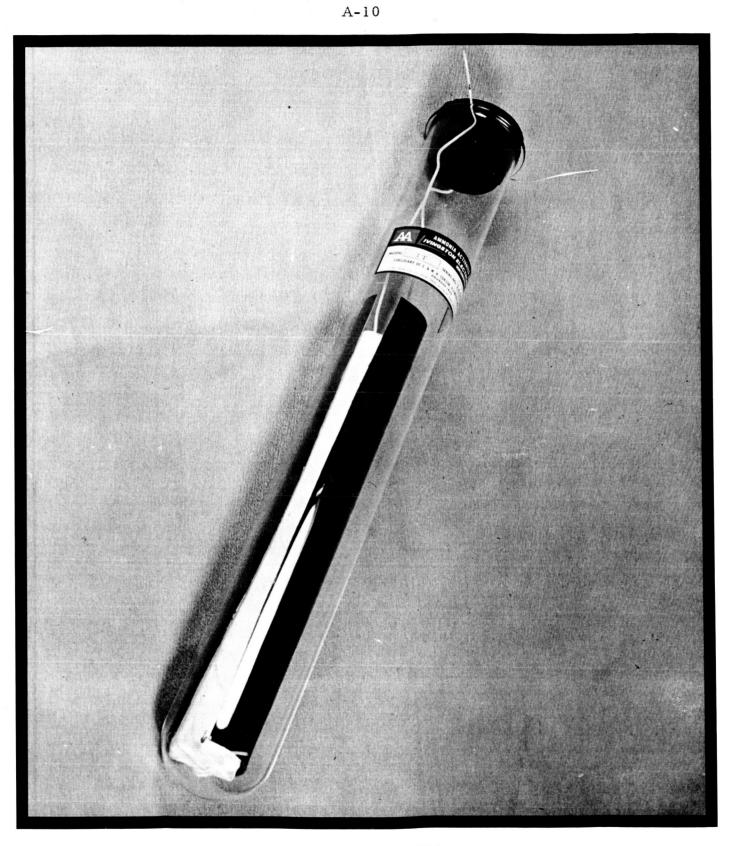


FIGURE 6



H-622 TEST VEHICLE FIGURE 6



TEST TUBE CELL FIGURE 7

Refrigeration

The tests conducted during the first two months used Livingston Electronic Corporation's alcohol bath mechanical refrigerators which are capable of maintaining -55°C. This temperature was adequately low for preliminary studies.

During this start-up period reliable methods of refrigeration capable of -73° C were studied and the installation of a liquid CO₂ system appeared to be the best immediate answer. During this time an alcohol bath unit was modified to maintain -63° C for additional low temperature studies. The liquid CO₂ system performed as anticipated but leakage problems with test vehicle sealing resulted in contamination of some test cells and loss of electrolyte.

Investigation of mechanical refrigeration capable of maintaining -90°C was also being conducted at this time and after careful consideration a custom built unit was ordered and delivered during the tenth month. This refrigerator proved capable of maintaining -90°C indefinitely. A photograph of this unit is shown in Figure 8, page A13.

Instrumentation

All test cell results were recorded by Esterline-Angus or Rustrak recorders. The proper load was applied to cells by a Livingston-built cyclic loader (Figure 9, page A14). This loader consisted of a timer which applied heavy drain for 6 minutes and light drain for 54 minutes.

A second loader was built for 5-cell operation. This device is a five-position scanner with a voltmeter recorder across each of the five cell positions for a period of 6 minutes for each cell. The scan of all cells is continuous and the cells are loaded for the low drain rate and recorded in one complete scan of 30 minutes. The alternate scan of all cells places a high current load across the cells that are being recorded. This high load function continues for a complete scan of five cells. The scanner then repeats the entire program, resulting in a 6 minutes high load and a 54 minutes low load for all cells.

The single cell resistive loads required to obtain power at the proper levels were computed from the formula, $R = E^2/P$, where

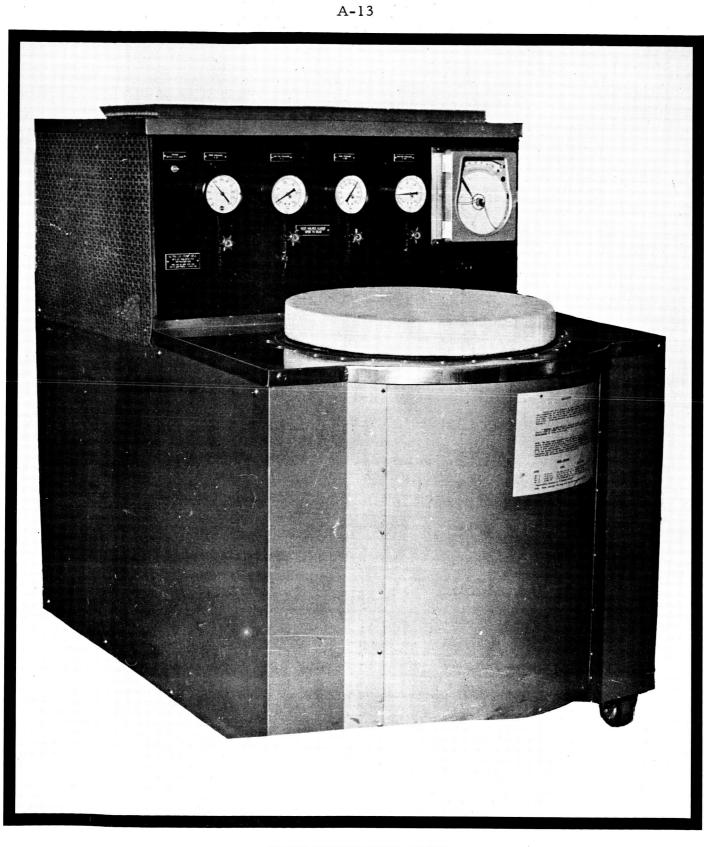
R =	the resistive load in ohms
E =	a nominal potential difference of 2 volts
	$(Mg/HgSO_4, S, and m-DNB)$
P =	power to be delivered in watts
$R_1 =$	$(2)^2/0.3 = 13.33$ ohms

 $R_2 = (2)^2 / 1.5 = 2.67$ ohms

Example:

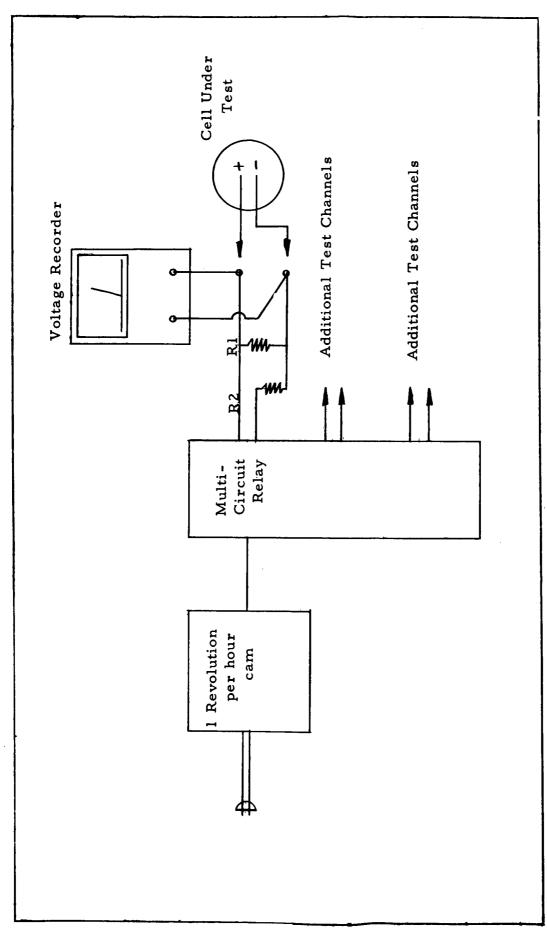
Standard commercial resistors of 2.7 ohms and five of 68 ohms in parallel were selected.

A cut-off of 75 percent, based upon the nominal 2 volts giving 1.5 volts, was taken as a good goal. Many tests were continued to 1.3 volts to obtain additional insight.



-90°C REFRIGERATOR

FIGURE 8



CYCLIC LOAD PROGRAMMER

FIGURE 9

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A-14

APPENDIX B

ELECTROCHEMICAL CELL TESTS

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NOTES TO TABLE I

l Loads shown as A/B represent cyclic loads of A Ω for 6 minutes and B Ω for 54 minutes.

²S.S. = stainless steel case Ag. P.S. = silver-plated steel case

³M-1365 Webril; #50 Whatman filter paper; or Whatman Extraction Thimble

⁴ Based on two electron change for meta-Dinitrobenzene

⁵ Finned and fluted, See Figure 1, page B-3

⁶ Finned

TABLE I - Supplement

Cell No.

2078	Failure caused by internal short circuit					
1 C	Butyl ''O'' ring chamber seal leaked at -73°C					
6 C	Silicone "O" ring leaked less than butyl at -73°C					
8C	Silicone "O" ring plus Teflon tape on threads provided satisfactory seal					
2273	4 percent silver powder added to cathode mixture					
2300	4 percent silver powder added to cathode mixture					
2290	Chamber leaked at -73°C					
		Hrs. to 1.5V				
2145	Ratio m-DNB:C:paper = 45:45:10	59				
2169	Ratio m-DNB:C:paper = 38:58:4	40				
2190	Ratio m-DNB:C:paper = 54:40:6	33				
2195	Ratio m-DNB:C:paper = 59:36:5	34				
2224	Ratio m-DNB:C:paper = 59:36:5	41				

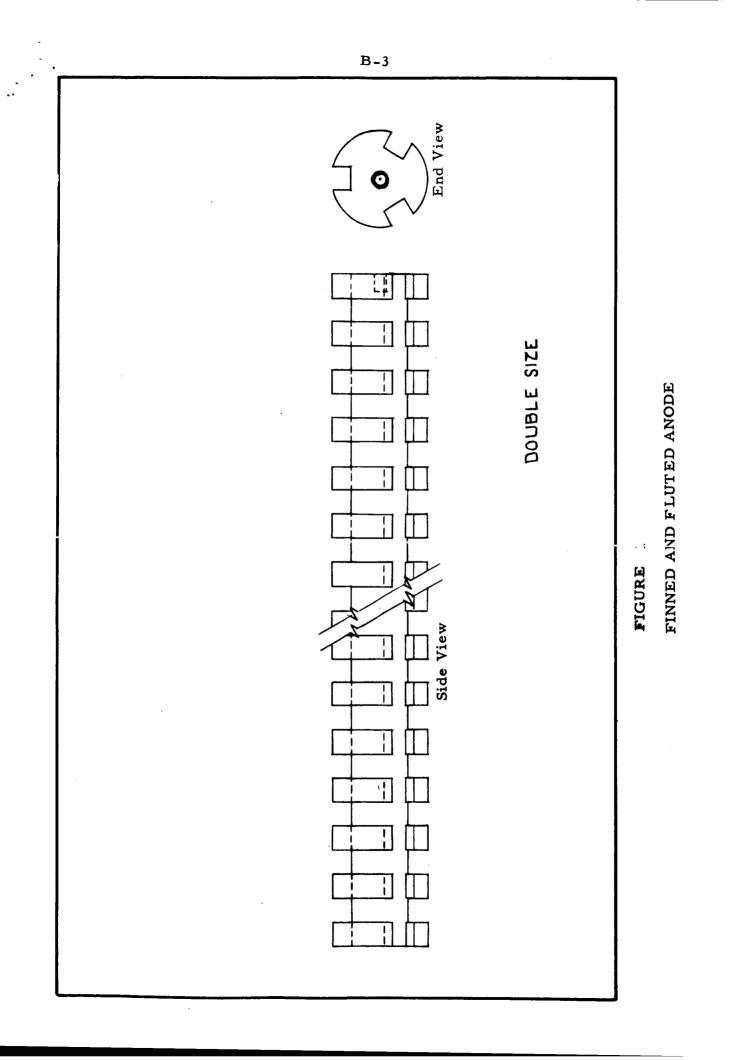


TABLE I - ELECTROCHEMICAL

Magnesium	-
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			8
Test Number:	А	2051	2078
Reference (Page No.):			
Vehicle:	A-601	A-601	A-601
Percent of Vehicle Volume Utilized:	60	60	60
Configuration:	Bobbin	Bobbin	Bobbin
Net Cell Volume (inches ³):	14.4	14.4	14.4
Load $\boldsymbol{\Omega}^{l}$:	10	10	10
Temperature:	24°C	-40° to -73°C	-73°C
Hours to End Voltage:	54	17	0
Anode (AZ31B Magnesium):	Rod	Rod	Rod
Anode Area cm ² :	71	71	71
Cathode:	HgSO ₄ **	HgSO ₄ **	HgSO ₄ **
Cathode Collector ² :	S. S.	S. S.	S. S.
Separator ³ :	Webril	Webril	Webril
Ion Exchange Membrane:			
Initial Open Circuit Voltage:	2.29	2.35	1.85
Initial Closed Circuit Voltage:	2.08	2.05	1.82
End Voltage:	1.50	1.80	1.80
Initial Anode Current Density (mA/cm ²)*:	2.9	2.9	
Initial Cathode Collector Current Density (mA/cm ²)*:	1.0	1.0	
Theoretical Cathode Coulombs/gram ⁴ :	580	580	580
Observed Cathode Coulombs/gram:	205	50	
Cathode Coulombic Efficiency:	35%	9%	
Watt Hours/Inch ³ of Net Cell:	1.21	0.44	0
,			·

*Based on the lighter load if cyclic **175 grams $HgSO_4 \cdot 2NH_3 + 75$ grams carbon B-4

i

CELL TESTS

<u>Group A</u>

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4

Mercuric Sulfate Couples

	2096	2166	7C	1C	6C	8C	2C	3C
								1
	A-601	A-601	A-601	A-601	A-601	A-601	A-622	A-622
	60	60	60	60	60	60	46	46
	Bobbin	Bobbin	Bobbin	Bobbin	Bobbin	Bobbin	Bobbin	Bobbin
	14.4	14.4	14.4	14.4	14.4	14.4	16.4	16.4
	10	3/15	3/15	10	10 -	50/250	10	10
	24°C	24°C	-73°C	-73°C	-73°C	-73°C	-55°C	-73°C
_	87	33	103	24	72	168	9	24
	Note ⁵	Note ⁵	Note ⁶	Note ⁶				
	142	142	142	142	142	142	25.6	25.6
	HgSO ₄ **	HgSO ₄ **	HgSO ₄ **	HgSO₄**				
	S. S.	S. S.	Ag P.S.	Ag P.S.				
	Webril	Webril	Webril	Webril	Webril	Webril	Webril	Webril
	2.24	2.37	2.20	2.20	2.34	2.30	2.30	2.24
	2.19	2.25	2.15	2.04	2.25	2.28	2.13	1,72
	1.50	1.50	1.50	1.50	1.50	1.80	1.50	1.50
	1.5	1.1	1.0	1.4	1.6	0.06	8.3	6.7
ļ	1.0	0.7	0.7	1.0	1.1	0.04	1.9	1.6
	580	580	580	580	580	580	580	580
ŀ	322	122	360	89	266	40	33	89
ļ	55%	21%	62%	15%	46%	7%	6%	15%
	2.06	0.83	2.39	. 52	1.77	0.29	0.33	0.43
		<u> </u>		L	· · · · · · · · · · · · · · · · · · ·			

TABLE I Continued

ELECTROCHEMICAL CELL TESTS

<u>Objective I Group A</u> Continued Magnesium-Mercuric Sulfate Couples

Test Number:	9C	10C	4C [#]	5.C
Reference (Page No.):				
Vehicle:	A-622	A-622	open cup	open cup
Percent of Vehicle Volume Utilized:	46%	46%		
Configuration:	Bobbin	Bobbin	Bobbin	Bobbin
Net Cell Volume (inches ³):	16.4	16.4		
Load Ω^1 :	3/15	50/250	200	200
Temperature:	-73°C	-73°C	-55°C	-55°C
Hours to End Voltage:	42		7	18
Anode (AZ31B Magnesium):	Note ⁵	Nuie ⁵	Exmet	Exmet
Anode Area cm ² :	25.6	25.6		
Cathode:	HgSO ₄ **	HgSO ₄ **	HgSO ₄ ***	HgSO ₄ ***
Cathode Collector ² :	Ag P.S.	Ag P.S.	Graphite	Graphite
Separator ³ :	Thimble	Thimble	Webril	Webril
Ion Exchange Membrane:				
Initial Open Circuit Voltage:	2.28	2.30	2.30	2.38
Initial Closed Circuit Voltage:	2.15	2.25	1.45	2.24
End Voltage:	1.5		1.0	1.0
Initial Anode Current Density (mA/cm ²)*:	5.60		2.98	4.35
Initial Cathode Collector C. D. (mA/cm^2) *:	1.30		3.28	4.84
Theoretical Cathode Coulombs/gram 4 :	580	580	580	580
Observed Cathode Coulombs/gram:	134		21	80
Cathode Coulombic Efficiency:	23%		4%	14%
Watt Hours/Inch ³ of Net Cell:	0.85			
*Based on the lighter load if cyclic				

175 grams $HgSO_4$ · $2NH_3$ + 75 grams carbon *7 grams of $HgSO_4$ · $2NH_3$ #94% liquid NH_3 + 6% Toluene

TABLE I

ELECTROCHEMICAL

Magnesium-

	1		6		
	Group B-1				
Test Number:	2057	2074	2089		
Reference (Page No.):					
Vehicle:	A-622	A-622	A-622		
Percent of Vehicle Volume Utilized:	< 20 %	< 20%	< 20%		
Configuration:	Flat Cell	Flat Cell	Flat Cell		
Net Cell Volume (inches ³):	5.5	5.5	5.5		
Load Ω^1 :	20/100	20/100	20/100		
Temperature:	25°C	25°C	25°C		
Hours to End Voltage:	42	50	117		
Anode (AZ31B Magnesium):	Sheet	Sheet	Sheet		
Anode Area cm ² :	135	135	135		
Cathode grams of $HgSO_4 \cdot 2NH_3$:					
Cathode, grams of meta-Dinitrobenzene:	16.2	16.2	16.2		
Cathode, grams of Sulfur:			-		
Cathode Collector ² :	S. S.	S. S.	S. S.		
Separator ³ :	Whatman	Whatman	Whatman		
Ion Exchange Membrane:					
Initial Open Circuit Voltage:	2.18	2.24			
Initial Closed Circuit Voltage:	2.15	2.20	2.16		
End Voltage:	1.70	1.70	1.50		
Initial Anode Current Density (mA/cm ²)*:	0.16	0.16	0.16		
Initial Cathode Collector Current Density (mA/cm ²)*:	0.16	0.16	0.16		
Theoretical Cathode Coulombs/gram ⁴ :	1150	1150	1150		
Observed Cathode Coulombs/gram:	210	280	600		
Cathode Coulombic Efficiency:	18%	24%	52%		
Watt Hours/Inch ³ of Net Cell:	0.43	0.43	1.04		

*Based on lighter load if cyclic.

Continued

CELL TESTS - Objective I

meta-Dinitrobenzene Couples

meta-Dinitrobenzene Couples							
I	Group	o B-2	Grou	Group B-3		up B-4	Group B-5
1	2128	2145	2273	2300	2258	2290	2319
		29					24
r 1	A-622	A-622	A-622	A-622	A-622	A-622	A-622
	< 20%	< 20%	<20%	< 20%	<20%	< 20%	< 20%
	Flat Cell	Flat Cell	Flat Cell				
	4.0	4.0	4.0	4.0	4.0	4.0	4.0
	20/100	10/50	10/50	10/50	10/50	10/50	10/50
	-55°C	-55°C	-73°C	-73°C	-73°C	-73°C	-55°C
	94	59.3	32.5	30.0	15.0	8.7	69.8
	Sheet	Sheet	Sheet	Sheet	Sheet	Sheet	Sheet
	135	125	135	135	135	135	135
				an 11 m			
	16.2	16.2	19.0	19.0	16.2	21.4	16.2
,	S. S.	S. S.	S. S.	S. S.	S. S .	S. S.	Ag Exmet
L.	Whatman	Whatman	Whatman	Whatman	Whatman	Whatman	Whatman
	SB6407	SB6407	SB6407	SB6407	SB6407	SB6407	SB6407
,			2.25	2.31	2.18	2.36	2.28
, i	2.22	2.20	2.24	2.28	2.11	2.20	2.27
	1.50	1.50	1.50	1.50	1.50	1.50	1.50
	0.16	0.3	0.3	0.3	0.3	0.3	0.3
	0.16	0.3	0.3	0.3	0.3	0.3	0.3
	1150	1150	1150	1150	1150	1150	1150
	518	628	285	285	155	83	784
(45%	55%	25%	25%	15%	7%	68%
	1.20	1.52	0.85	0.80	0.35	0.17	1.90

TABLE I

2224

A-622

<20%

4.0

10/50

-55°C

41.5

Sheet

135

_ _ _ _

21.4

- - -

S. S.

Whatman

SB6407

2.25

2.19

1.50

0.3

0.3

1150

299

26% 0.63

Flat Cell

ELECTROCHEMICAL

Magnesium-meta-Dinitrobenzene Couples

	Group B-6			
Test Number:	2169 -	2190	2195	
Reference (Page No.):				
Vehicle:	A-622	A-622	A-622	-
Percent of Vehicle Volume Utilized:	< 20%	< 20%	< 20%	•
Configuration:	Flat Cell	Flat Cell	Flät Cell	
Net Cell Volume (inches ³):	4.0	4.0	4.0	•
Load Ω^1 :	10/50	10/50	10/50	
Temperature:	-55°C	-55°C	-55°C	
Hours to End Voltage:	40.3	33.3	33.6	
Anode (AZ31B Magnesium):	Sheet	Sheet	Sheet	
Anode Area cm ² :	135	135	135	
Cathode, grams of $HgSO_4 \cdot 2NH_3$:				
Cathode, grams of meta-Dinitrobenzene:	13.7	19.3	21.4	
Cathode, grams of Sulfur:				
Cathode Collector ² :	S. S.	S. S.	S. S.	
Separator ³ :	Whatman	Whatman	Whatman	
Ion Exchange Membrane:	SB6407	SB6407	SB6407	
Initial Open Circuit Voltage:	2.03	2.17	2.19	
Initial Closed Circuit Voltage:	2.05	2.07	2.10	
End Voltage:	1.50	1.50	1.50	
Initial Anode Current Density (mA/cm^2) *:	0.3	0.3	0.3	
Initial Cathode Collector C. D. (mA/cm^2) *:	0.3	0.3	0.3	
Theoretical Cathode Coulombs/gram ⁴ :	1150	1150	1150	
Observed Cathode Coulombs/gram:	392	313	314	
Cathode Coulombic Efficiency:	33%	27%	27%	
Watt Hours/Inch ³ of Net Cell:	0.78	0.66	0.66	

*Based on lighter load if cyclic.

Continued

CELL TESTS - Objective I

Magnesium-Sulfur Couples

Magnesium-Mixed Cathode Couples

		Group C					
	2205	2204	2209	2056	2129	2136	2179
	9	9	9				29
	A-606	A-606	A-606	A-601	A-601	A-601	A-622
	< 20%	<20%	<20%	60%	60%	70%	< 20%
	Flat Cell	Flat Cell	Flat Cell	Bobbin	Bobbin	Bobbin	Flat Cell
	0.66	0.66	0.66	14.4	14.4	14.4	4.0
	50/250	50/250	50/250	10	10	10	10/50
	-55°C	-55°C	-55°C	24°C	24° C	24°C	-55°C
	12.5	9.5	7.7	1.0	0	26.1	9.2
	Sheet	Sheet	Sheet	Rod	Rod	Rod	Sheet
[[29	20	2 <u>0</u>	<u>71</u>	<u>71</u>	71	135
	200 00 m	ao ao ao	000 MM gay	26.3	26.3	26.3	*
í	162 1880 172	* * *	atunin an	400 000 000	au ao mo		13.0
	3.23	3.23	3.23	60	60	60	7.2
	S. S.	S. S.	S. S.	S. S.	S. S.	Ag wire	S. S.
	Whatman	Whatman	Whatman	Whatman	Webril	Webril	Whatman
		SA6404	SB6407	1400 CRF 1886	mu anto aco		SB6407
	2.22	2.11	2.16	caso entr life	0.4	2.12	2.18
	2.21	2.10	2.19	2.28	0.1	2.50	2.00
	1.50	1.50	1.50	1.50	1.50	1.50	1.50
	0.3	0.3	0.3	000 LBS 100	ດວ່າຫວັດພະ	3.5	0.3
	0.3	0.3	0.3	ue an eo	60 m m		0.3
	6030	6030	6030		and and an		≈2890
	154	114	95		~ ~ -	84	87
	2.6%	1.9%	1.58%	aar 000 900			8%
	0.39	0. 28	0.24		~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~	0.64	0.21

NOTES TO TABLES II AND III

ELECTROCHEMICAL CELL TESTS

- ¹ T. T. = Pyrex Test Tube
- 2 Loads shown as A/B represent cyclic loads of A Ω for 6 minutes and B Ω for 54 minutes
- ³ M-1365 Webril in multiple layers, 0.004"/layer
- ⁵ Coordination No. $0 = HgSO_4$ No. $2 = HgSO_4 \cdot 2NH_3$ (ammoniated) No. $4 = HgSO_4 \cdot 4NH_3$ (slaked)
- 6 S.S. = stainless steel
- ⁷ Whatman No. 50 filter paper
- * Based on lighter load** Based on heavy load
- ± Anode lead sheared at 49.5 hours on Cell B-2

B-12

TABLE II - ELECTROCHEMICAL

N/1 つ	an	001	11m /	
TATO	<u> 811</u>	C 21	ium/	

Test Number:	Т2	Т3	Τ4	T5
Major Variables:		open top		closed top
Reference (Page No.):			12	
Vehicle ¹ :	TT	A-624	ТТ	A-624
Configuration-Bobbin:				
Net Cell Volume (in. ³):	30	34 ·	30	34
Load, $\boldsymbol{\Omega}^2$:	3/15	3/15	3/15	3/15
Temperature, °C;	-63°	-63°	-63°	-63°
Hrs. to End Voltage (Cumulative) 1.5/1.3V**:	44	70/72	63/70	57/61
Anode (AZ31B Mg Sheet), Area (cm ²) :	280	354	354	354
Cathode, gm. HgSO ₄ ·2NH ₃ :	119	119	119	119
Cathode, gm. Sulfur:				
Cathode, gm. Carbon:	51	51	51	51
Cathode Collector:	Ag Coil	Ag Coil	Ag Coil	Ag Coil
Cathode Collector Area (cm^2) :	60	60	60	60
Separator Thickness ³ , inches:	0.028	0.028	0.028	0.028
$HgSO_4$ NH ₃ Coordination No. ⁵ :	2	2	2	2
Initial Open Circuit Voltage :	2.30	2.30	2.45	080 CC1 184
Initial Closed Circuit Voltage* :	2.20	2.25	2.25	2.20
Peak Closed Circuit Voltage*:	2.22	2. 25	2.25	2.25
End Voltage (under Max, Load):	2.00	1.50	1.50	1.50
End Voltage (under Light Load) :	2.21	2.10	2.20	2.05
Initial Anode C. D. (mA/cm^2) * :	0.52	0.42	0.42	0.42
Initial Cathode Collector C. D. (mA/cm^2) * :	2.44	2.50	2.50	2.44
Observed Coulombs/gram oxidants:	281	398	359	323
Observed F/mol Cathode Reactant :	0.96	1.37	1.25	1.11
Watt Hours/Inch ³ of Net Cell :	0.65	0.73	0.74	0.59

B-13

CELL TESTS

Mercuric Sulfate Couples

		-				
	Т6	Т8	Т9	T-10	T-	11
-	cathode dia.	slaked cathode dia.	slaked cathode dia.	center anode	geomet	ry & temperature
	13, 15		15		6	
	A-624	A-624	A-624	ΤT	A-	624
				Inverse	Co	onventional
	34	34	34	30	34	
	3/15	3/15	2.67/15	3/15	3/1	15
	-73°	+20°	-73°	-63°	-73°	+20°
	57/59	16/18	33/34	0	10.5	56.5
	248	337	373		226	6
	140	161	147		17	5
					0	
	60	69	63		75	
	Ag Coil	Ag Coil	Ag Coil		A	ng Coil
	60	60	60		60	
	0.028	0.028	0.028		0.(028
	2	4	4	2	4	
	2.40	2.38	2.36		2. 3	35
	2.20	2.32	2.30		2.30	2.35
	2.20	2.32	2.30		2.30	2.35
	1.50	1.50	1.50		1.50	1.50
	2.10	2.17	2.08		1,75	1.78
	0.59	0.46	0.41		0.68	
	2.44	2.58	2.56		2.56	
	271	68	154		41	224
	0.93	0.24	0.53		0.14	0.77
	0.57	0.17 🦿	0.35	مهر د ا	0.11	0.61
1				f a iled to activate		

TABLE II - ELECTROCHEMICAL

Magnesium/

Test Number:	T-12	T-13	T-14	
Major Variables:	geometry & temperature			
Reference (Page No.):	6	6	6, 15	
Vehicle ¹ :	A-624	TT	A-624	
Configuration-Bobbin:		Conven	tional	
Net Cell Volume (in. ³):	34	30 ·	68	
Load, $\boldsymbol{\Omega}^2$:	3/15	3/15	3/15	
Temperature, °C;	+24°	-63°	-73°	
Hrs. to End Voltage (Cumulative) 1.5/1.3V**:	17	32.5	41	
Anode (AZ31B Mg Sheet), Area (cm ²) :	242	271	725	
Cathode, gm. $HgSO_4 \cdot 2NH_3$:	175	175	175	
Cathode, gm. Sulfur:	0	0	0	
Cathode, gm. Carbon:	75	75	75	
Cathode Collector:	Ag Coil			
Cathode Collector Area (cm ²) :	60	60	60	
Separator Thickness ³ , inches:	0.028	0.028	0.028	
$HgSO_4$ NH ₃ Coordination No. ⁵ :	4	4	4	
Initial Open Circuit Voltage :	2.35	2.30	2.35	
Initial Closed Circuit Voltage* :	2.30	2.15	2.30	
Peak Closed Circuit Voltage*:	2.32	2.23	2.35	
End Voltage (under Max, Load) :	1.50	1.50	1.50	
End Voltage (under Light Load) :	1.80	2.00	2.00	
Initial Anode C. D. (mA/cm ²)* :	0.64	0.55	0.22	
Initial Cathode Collector C. D. $(mA/cm^2)*$:	2.58	2.48	2.61	
Observed Coulombs/gram oxidants:	67	125	163	
Observed F/mol Cathode Reactant :	0.23	0.43	0.56	
Watt Hours/Inch ³ of Net Cell :	0.18	0.38	0.23	
	leaked s.circuit			

B**-**15

CELL TESTS (Continued)

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Mercuric Sulfate Couples

T-15	a T-1	6	T-17	л. Г	-18	
geometry & te	emp. temper node to cathode spa	ature acing ≈ ¹ / ₄ ''	temperature	oh temperature te		
6	6, 1		6	1	2	
TT	A-624		A-624	г	`T	
	Co	onventional		4. a		
30				3	0	
3/15	3/1	5	3/15		.7/13.6	
-63°	-73°	+15°	20°	-63°	≈-30°	
47	47	83	16	59	71	
371	465	<u></u>	465		30	
175	147		164	1	47	
0	0		0	0		
75	63		71	63		
	- <u></u>	Ag Coil				
60	60	<u></u>	60	6	0	
0.016	0.0	16	0.028	· 0	. 028	
2	2		4	2		
2.35	2.35		2.44	2.22		
2.25	2.25	- 180-	2.30	2.05	.	
2.28	2.25	2.27	2.32	2.25	2.30	
1.50	1.50	1.50	1.97	1.50	1.50	
1.90	1.70	1.75	2.25	1.88	1.82	
0.41	0.32	ан айн	0.33	0.38	do tão	
2.54	2.50		2.58	2.76	ಕ್ಷತ್ರೆ ಹೆಲ	
183	216	383	76	300	362	
0.63	0.74	1.31	0.26	1.03	1.24	
0.56	0.45	0.80	0.20	0.77	0.93	
anode connec- tion broken			"Vented"			
n negoti nego negoti ne	، منه ^{الس} ليد الم المراجع من المراجع من المراجع المراجع المراجع من المراجع المراجع المراجع من المراجع من المراجع م 	، بي المان الم 	in a bha an Anna an Anna an Anna an Anna an Anna ann ann		anna a shaif isani ta kata 1995, <u>a sha ana an</u> anna 2009.	

TABLE II - ELECTROCHEMICAL

Magnesium/

. . .

Test Number:	
Major Variables:	
Reference (Page No.):	
Vehicle ¹ :	
Configuration-Bobbin:	
Net Cell Volume (in. ³):	
Load, Q ² :	
Temperature, °C;	
Hrs. to End Voltage (Cumulative) 1.5/1.3V**:	
Anode (AZ31B Mg Sheet), Area (cm ²):	
Cathode, gm HgSO ₄ ·2NH ₃ :	
Cathode, gm. Sulfur:	
Cathode, gm. Carbon:	
Cathode Collector:	
Cathode Collector Area (cm ²) :	
Separator Thickness ³ , inches:	
$HgSO_4$ NH ₃ Coordination No ⁵ :	
Initial Open Circuit Voltage :	
Initial Closed Circuit Voltage* :	
Peak Closed Circuit Voltage*:	
End Voltage (under Max, Load):	
End Voltage (under Light Load) :	
Initial Anode C. D. (mA/cm ²)* :	
Initial Cathode Collector C.D. $(mA/cm^2)*$:	
Observed Coulombs/gram oxidants:	
Observed F/mol Cathode Reactant :	
Watt Hours/Inch ³ of Net Cell :	

t		
r	- 19	T-20
1	hmic load emperature	slaking
]]	12	15
ב	T	TT
	Co	nventional
3	0	30
2	.7/13.6	2.7/13.6
-63°	≈-30°	-63°
53	64	47
3	62	354
1	47	147
0		0
6	3	63
	А	g Coil
6	0	60
0	0.028	0.028
2		4
2.40		2.35
2.10		2.20
2.30	2. 37	2.30
1.50	1.88	1.50
1.95	2.29	1.95
0.47		0.48
2.82		2.82
272	335	242
0.93	1.15	0.83
0.70	0.88	0.62

CELL TESTS (Continued)

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Mercuric Sulfate Couples

		· · · · · · · · · · · · · · · · · · ·	
T-21	T-22	Т-23	T-24
Slaking	Acetylene black 5 grams	Webril 30 discs	Blotter 15 discs
15			
TT	ŢT	TT	TT
	Conventional		
30	30	30	30
	2.7/13.6		
-63°	-63°	-63°	-63°
5 9	55	46	48
354	354	354	354
140	122	140	140
0	0	0	0
60	52	60	60
	Ag Coil	24" Ag Wire	12" Ag Wire
60	60	16	8
0.028	0.028	0.028	0.028
4	2	4	4
2.28	2.35	2.25	2.32
2.05	2.22	2.20	2.25
2.21	2.25	2.22	2.30
1.50	1.50	1.50	1.50
1.98	2.00	2.10	1.96
0.46	0.47	0.46	0.48
2.71	2.76	10.2	21.2
311	337	220	258
1.07	1.15	0.76	0.89
0.75	0.72	0.58	0.64
		.	

TABLE II - ELECTROCHEMICAL

Magnesium/Mercuric Sulfate and

		·····		
Test Number:	T-25]	r-26	
Major Variables:	Acetylene black 9 gms.	S:HgSO₄· 2	NH3 with	NH₄SCN
Reference (Page No.):			12	
Vehicle ¹ :	ТТ	7	ГТ	
Configuration-Bobbin:		Conventio	nal	
Net Cell Volume (in. ³):	30	. 3	30	
Load, $\mathbf{\Omega}^2$:	2.7/13.6	2.7/13.6	∞/ 1 3.6	∞/13.6
Temperature, °C;	-63°		-63°	
Hrs. to End Voltage (Cumulative) 1.5/1.3V**:	39	50	87	114
Anode (AZ31B Mg Sheet), Area (cm ²):	354		354	
Cathode, $gm HgSO_4 \cdot 2NH_3$:	115		19.3	
Cathode, gm. Sulfur:	0		44.1	
Cathode, gm. Carbon:	50		49.6	
Cathode Collector:	Ag Coil	Carbon ro	od - 3/16	"x11"
Cathode Collector Area (cm ²) :	60		38	
Separator Thickness ³ , inches:	0.028		0.028	
$HgSO_4$ NH ₃ Coordination No. ⁵ :	4		2	
Initial Open Circuit Voltage :	2.33	2.45		
Initial Closed Circuit Voltage* :	2.00	2.12		
Peak Closed Circuit Voltage*:	2. 22	2.20	1.80	1.50
End Voltage (under Max. Load):	1.50	1.50		
End Voltage (under Light Load) :	2.00	2.00	1.50	1.30
Initial Anode C. D. (mA/cm ²)* :	0.46	0.46		
Initial Cathode Collector C. D. (mA/cm^2) * :	2.72	4.26		
Observed Coulombs/gram oxidants:	250	5.80	835	993
Observed F/mol Cathode Reactant:	0.86	0.26	0.38	0.45
Watt Hours/Inch ³ of Net Cell :	0.50	0.63	0.88	1.01

B-19

CELL TESTS (Continued)

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Magnesium/Mercuric Sulfate - Sulfur Couples

							·
	T-27	T-28	T-29	T-30	T-31	T-32	
		Paper Pulp 3 gm s .	Active stand w/anode	Cathode soaked in NH4SCN/NH3	S:HgSO ₄ · 21	NH ₃ with NH ₄ -SCN Steel cathode collector	
	12		49, 51		12		
	TT	ТТ	TT	ТТ	ΤΤ	TT	
				Conventional			
	30	30	30	30	30	30	
			2.7/13.6				
	-63°	-63°	-63°	-63°	-63°	-63°	
	44/56	62.5/63.5	0	31.5/32.5	47.5/55	3/45 59	
	354	372	304	347	352	337	
	19.3	140	119	119	34.2	34.2	
	44.1	0	0	0	39.2	39.2	
	49.6	60	51	51	44.1	44.1	
		Ag wire	Ag wire	Ag wire	C rod	Steel rod	
	38	13.4	13.4	13.4	38	31	
	0.028	0.028	0.028	0.028	0.028	0.028	
	2	2	2	2	2	2	
	2.45	2.36	0.93	2.25	2.52	2.57	
	2.20	2.00		2.12	2.00	2.06	
	2.20	2.30		2.20	2.23	2.13 2.01	
	1.50	1.50		1.50	1.50	1.50 1.25	
	1.95	1.75		1.95	1.95	2.01 1.66	
	0.46	0.45		0.47	0.47	0.47	
	4.26	12.6		12.1	4.32	5.06	
	510	337		194	481	30 524	
	0.23	1.16		0.67	0.28	0.02 0.30	
	0.55	0.83		0.40	0.61	0.04 0.59	
······································			••••••••••••••••••••••••••••••••••••••	<u>. </u>	• · · · · · · · · · · · · · · · · · · ·		

TABLE II - ELECTROCHEMICAL CELL TESTS (Continued)

Magnesium/Mercuric Sulfate and Magnesium/Mercuric Sulfate - Sulfur Couples

Test Number:	T-33	T-34
Major Variables:	HgSO4 · 2NH3 S	Active stand wo/anode
Reference (Page No.):	13, 15	49, 51
Vehicle ¹ :	ТТ	ТТ
Configuration-Bobbin:	(Conventional
Net Cell Volume (in. ³):	30	30
Load, $\boldsymbol{\Omega}^2$:	2.7,	/13.6
Temperature, °C;	-63°	-63°
Hrs. to End Voltage (Cumulative) 1.5/1.3V**:	73/78	10/10.5
Anode (AZ31B Mg Sheet), Area (cm ²) :	337	368
Cathode, gm. HgSO ₄ ·2NH ₃ :	133	140
Cathode, gm. Sulfur:	10	U
Cathode, gm. Carbon:	57	60
Cathode Collector:	Ag wire	Ag wire
Cathode Collector Area (cm ²) :	14.7	16.7
Separator Thickness ³ , inches:	0.028	0.028
$HgSO_4$ NH ₃ Coordination No. ⁵ :	2	2
Initial Open Circuit Voltage :	2.35	2.24
Initial Closed Circuit Voltage* :	2.10	2.15
Peak Closed Circuit Voltage*:	2.25	2.24
End Voltage (under Max. Load):	1.50	1.50
End Voltage (under Light Load) :	1.75	1.87
Initial Anode C. D. $(mA/cm^2)*$:	0.49	0.45
Initial Cathode Collector C.D. (mA/cm^2) * :	11.25	9.90
Observed Coulombs/gram oxidants:	382	53
Observed F/mol Cathode Reactant :	0.79	0.15
Watt Hours/Inch ³ of Net Cell :	0.95	0.13

TABLE III - ELECTROCHEMICAL

Magnesium/

•

Test Number:	B2
Major Variables:	Co
Reference (Page No.):	28
Vehicle ¹ :	A-
Configuration:	Sp
Net Cell Volume (in. ³):	6
Load, Ω^2 :	25
Temperature °C:	-7
Hrs. to End Voltage (Cumulative)**:	49
Anode (Mg Sheet), Area (cm²):	77
Cathode, gm. m-DNB:	16
Cathode, gm. Carbon:	16
Cathode Collector:	Ag
Cathode Collector Area (cm ²):	77
Separator Thickness ³ , inches:	0.
Initial Open Circuit Voltage:	
Initial Closed Circuit Voltage*:	2.
Peak Closed Circuit Voltage*:*	2.
End Voltage (under Max. Load):	1.
End Voltage (under Light Load):	1.
Initial Anode C. D. (mA/cm^2) *:	0.
Initial Cathode Collector G. D. (mA/cm^2) *:	0.
Observed Coulombs/gram oxidants:	28
Observed F/mol Cathode Reactant:	0.
Watt Hours/Inch ³ of Net Cell:	0.

B2	В3			
Configuration	Size			
28	28			
A-606	H-6	22		
Spiral wrap	Spira	d Wrap		
6	33			
25/120	3/15			
-73°	-73°			
49.5	116	131		
77	671			
16	172			
16	172			
Ag sheet	Ag sheet			
77	710			
0.012	0.0	12		
2.34	2.10			
2.34	2.1	0		
1.70±	1.50	1.40		
1.93	1.82	1.67		
0.25	0.21			
0.25	0.20			
282	437	480		
0.49	0.76	0.84		
0.42	1.14	1.22		

B-22

CELL TESTS

meta-Dinitrobenzene Couples

				· · · · · · · · · · · · · · · · · · ·				
B4			В5	В6			B7	
Temperature		Replicate	Replicate			Vehicle		
 28			28	4	28		28	
	H-622		H-622		H-622		lL b	eaker
Spi	ral wrap)	Spiral wrap	s	piral w	rap	Spira	l wrap
	33		33		33		33	
	3/15		3/15		3/15		3/1	15
	+20°		+20°		+20°		-63°	
21	37	70+	5	22	49	73	110	121
671		671	671		851			
	172		172	172 172		206		
	172		172			206 S. S. ⁶ 851		
Ag	sheet		Ag sheet	Ag sheet				
	710		710	710				
	0.012		0.012	0.012		0.0	012	
	2.40			Nay ina Up				
	2.28		2.12	2.12		2.05		
	2, 28		2.12		2.12		2. (05
1.50	1.40	1.30	1.50	1.50	1.40	1.30	1.50	1.40
 1.90	1.87	1.84	666 (dg) (des	1.83	1.77	1.70	1.80	1.73
	0.23		0.21		0.21		0.1	16
0.21		0.20		0.20		0.1	16	
83	143	262	19	83	180	261	342	366
0.15	0.25	0.46	0.03	0,15	0.31	0.45	0.60	0.64
0.23	0.38	0.68	0.05	0.22	0.46	0.65	1.06	1.10

TABLE III - ELECTROCHEMICAL

Magnesium/

•.

Test Number:	2396	B8
Major Variables:	Configuration	Vehicle
Reference (Page No.):	24	28
Vehicle ¹ :	A-622	H-606
Configuration:	Flat Plate	Spiral wrap
Net Cell Volume (in. ³):	6.9	6
Load, Ω^2 :	10/50	25/120
Temperature °C:	-73°	+20°
Hrs. to End Voltage (Cumulative)**:	44.4	0
Anode (Mg Sheet), Area (cm²):	135	77
Cathode, gm. m-DNB:	16.2	16
Cathode, gm. Carbon:	16.2	16
Cathode Collector:	Ag Exmet	Ag sheet
Cathode Collector Area (cm ²):	135	77
Separator Thickness ³ , inches:	0.007 ⁷	0.012
Initial Open Circuit Voltage:	2.25	
Initial Closed Circuit Voltage*:	2.25	
Peak Closed Circuit Voltage*:	2.25	
End Voltage (under Max. Load):	1.50	
End Voltage (under Light Load):		
Initial Anode C.D. (mA/cm^2) *:	0.33	
Initial Cathode Collector C. D. (mA/cm^2) *:	0.33	
Observed Coulombs/gram oxidants:	558	
Observed F/mol Cathode Reactant:	0.97	
Watt Hours/Inch ³ of Net Cell:	0.68	

CELL TESTS (Continued)

meta-Dinitrobenzene Couples

 			······································
 B9	B10)	B11
7 days active stand at 20°C	Temperature		active stand 8 days at -63°
28	28		28
н-606	H-622		lL beaker
Spiral wrap	Spiral wrap		Spiral wrap
6	33		33
25/120	2.7/13.6		2.7/13.6
+20°	-73°		-63°
0	88	107	102
77	671		671
16	172		172
16	172		172
Ag sheet	Ag sheet		Ag sheet
77	710		710
0.012	0.012		0.012
	2.25		
	2.10		1.60
	2.10	1.80	1.82
	1.50	1.30	1.00
	1.80	1.65	1.67
	0.23		0.20
	0.22		0.19
	366	420	332
	0.64	0.73	0.58
	0.95	1.03	0.68

NOTES AND CODE TO TABLES IV THROUGH VII

¹ T. T. = Pyrex Test Tube

² Loads shown as A/B represent cyclic loads of A Ω for 6 minutes and B Ω for 54 minutes.

³M-1365 Webril in multiple layers, 0.004"/layer

⁴ After 23 hours, added KSCN

⁵ Coordination No. 0 = $MSO_4 \cdot 0NH_3$ (non-ammoniated) No. 2 = $MSO_4 \cdot 2NH_3$ (ammoniated) No. 4 = $MSO_4 \cdot 4NH_3$ (slaked)

Where "M" indicates a heavy metal.

⁶ "Oxidants" includes major oxidant, sulfur and additive if an oxidant

^c anode coated on outside with K-M rubber cement

d pure magnesium anode

^e cell life = 3 hours to 1.3 volts; 75 hours to 0.80 volts, based on heavy load

^f AZ31B Mg Exmet (expanded thickness = 0.040")

^g M-1365 Webril (0.004"/layer) + #4965 Whatman thimble (0.025" thick)

^h SM-91 Webril polypropylene (0.005" thick/layer)

ⁱ Ag plated copper Exmet

^j Ag Exmet collector outside = 192 cm^2

* Based on lighter load

** Cumulative hours, based on heavy load Hours shown as A/B represent A = hours to 1.5 volts and B = hours to 1.3 volts. A single entry represents hours to end voltage under maximum load.

Electrolyte Code

- A 25 weight percent KSCN in liquid ammonia
- B 50 percent spent electrolyte from similar discharged cell, plus
 50 percent of (A)
- C Liquid ammonia
- D 80 percent spent electrolyte plus 20 percent liquid ammonia
- E 10 weight percent KSCN in liquid ammonia
- F 12.5 weight percent $Mg(SCN)_2$ in liquid ammonia; saturated with $MgSO_4$
- G 50 percent of (A) plus 50 percent of (F)
- H 50 percent of a 17 weight percent solution of Mg(SCN)₂ in liquid ammonia plus 50 percent of (A)
- I 25 weight percent KSCN in liquid ammonia was electrolyzed 3 minutes using two Mg electrodes at 0.75 volts.
- J 50 weight percent KSCN in liquid ammonia
- K 5.6 weight percent NaBr in liquid ammonia
- L 34 weight percent KSCN in liquid ammonia

Computation of Observed Coulombs per gram of Oxidants:

 $\frac{E}{R} / \frac{T}{gms.} = \text{coulombs per gram, where}$ E = average of peak closed circuit voltage and end voltage of 1.50 R = mean resistance in ohms between light and heavy loads T = discharge time in seconds to 1.50 volts under heavy load gms. = combined weight of oxidants

ELECTROCHEMICAL CELL TESTS-

Test Number:T-35T-36T-37Major Variables: $FulfurFulfurFulfurFulfurElectrolyte:AAAReference (Page No.):13, 151313, 15Vehicle1:TTTTTTNet Cell Volume (in.3):303030Wet Stand, Hrs/Temp. °C:-63^{\circ}-63^{\circ}-63^{\circ}Temperature °C, Discharge:-63^{\circ}-63^{\circ}-63^{\circ}Hrs. to End Voltage (1.5/1.3v)**:72/7867/7397/98Anode (AZ31B Mg) Sheet-Thickness, ins.:0.0160.0160.016Anode Area (cm2):377368368Cathode, Major Oxidant:HgSO4HgSO4HgSO4gm. Major Oxidant:133137126gm. Carbon:575854Additive; -MSO4: NH3 Coordination No. \frac{5}{2}22Cathode Collector Area (cm2) (Ag wire):14.714.714.70.0280.0280.0280.028$
Major Variables:Temp.+Sulfur+SulfurElectrolyte:AAAReference (Page No.):13, 151313, 15Vehicle1:TTTTTTNet Cell Volume (in. 3):3030Wet Stand, Hrs/Temp.*C:-63°-63°Temperature °C, Discharge:-63°-63°Hrs. to End Voltage (1.5/1.3v)**:72/7867/73Anode (AZ31B Mg) Sheet-Thickness, ins.:0.0160.016Anode Area (cm²):377368Gathode, Major Oxidant:HgSO4HgSO4HgSO,gm. Sulfur:10520gm. Carbon:5758Additive, gms.:MSO4: NH3 Coordination No. $\frac{5}{1}$:22Cathode Collector Area (cm²) (Ag wire):14.714.714.714.714.7
Reference (Page No.): Vehicle1:13, 151313, 15Vehicle1:TTTTTTNet Cell Volume (in. 3): 303030Wet Stand, Hrs/Temp.*C: Temperature °C, Discharge:-63°-63°Hrs. to End Voltage (1.5/1.3v)**: Anode (AZ31B Mg) Sheet-Thickness, ins.: Major Oxidant:0.0160.016Anode Area (cm²): gm. Major Oxidant:377368368gm. Major Oxidant: gm. Sulfur:13137126gm. Carbon: Additive: Additive; gm. Carbon:575854MSQ, NH3 Coordination No. 5: Separator Thickness ³ , inches:222Cathode Collector Area (cm²) (Ag wire): Separator Thickness ³ , inches:14.714.714.7
Vehicle ¹ : TT TT TT Net Cell Volume (in. ³): 30 30 30 Wet Stand, Hrs/Temp.*C: -63° -63° -63° Temperature °C, Discharge: -63° -63° -63° Hrs. to End Voltage (1.5/1.3v)**: 72/78 67/73 97/98 Anode (AZ31B Mg) Sheet-Thickness, ins.: 0.016 0.016 0.016 Anode Area (cm ²): 377 368 368 Cathode, Major Oxidant: HgSO ₄ HgSO ₄ HgSO ₄ gm. Major Oxidant: 133 137 126 gm. Sulfur: 10 5 20 gm. Carbon: 57 58 54 Additive: - - - MSO ₄ NH ₃ Coordination No. ⁵ : 2 2 2 Cathode Collector Area (cm ²) (Ag wire): 14.7 14.7 14.7 Separator Thickness ³ , inches: 0.028 0.028 0.028
Net Cell Volume (in. ³): 30 30 30 Wet Stand, Hrs/Temp.*C: -63° -63° -63° Temperature °C, Discharge: -63° -63° -63° Hrs. to End Voltage (1.5/1.3v)**: 72/78 67/73 97/98 Anode (AZ31B Mg) Sheet-Thickness, ins.: 0.016 0.016 0.016 Anode Area (cm ²): 377 368 368 Cathode, Major Oxidant: HgSO4 HgSO4 HgSO4 gm. Major Oxidant: 133 137 126 gm. Sulfur: 10 5 20 gm. Carbon: 57 58 54 Additive: - - - MSO4· NH3 Coordination No. ⁵ : 2 2 2 Cathode Collector Area (cm ²) (Ag wire): 14.7 14.7 14.7 Separator Thickness ³ , inches: 0.028 0.028 0.028
Wet Stand, Hrs/Temp.*C: Temperature °C, Discharge: Hrs. to End Voltage $(1.5/1.3v)^{**}$: -63° -63° Anode (AZ31B Mg) Sheet-Thickness, ins.: Anode Area (cm ²): 0.016 0.016 0.016 Anode Area (cm ²): 377 368 368 Cathode, Major Oxidant: gm. Major Oxidant: $HgSO_4$ $HgSO_4$ gm. Sulfur: 10 5 20 gm. Carbon: Additive: MSO ₄ · NH ₃ Coordination No. $\frac{5}{12}$ 2 2 Cathode Collector Area (cm ²) (Ag wire): 14.7 14.7 14.7 Separator Thickness ³ , inches: 0.028 0.028 0.028
Temperature °C, Discharge: -63° -63° -63° Hrs. to End Voltage (1.5/1.3v)**: 72/78 67/73 97/98 Anode (AZ31B Mg) Sheet-Thickness, ins.: 0.016 0.016 0.016 Anode Area (cm ²): 377 368 368 Cathode, Major Oxidant: HgSO4 HgSO4 HgSO4 gm. Major Oxidant: 133 137 126 gm. Sulfur: 10 5 20 gm. Carbon: 57 58 54 Additive: MSO4: NH3 Coordination No. ⁵ : 2 2 2 Cathode Collector Area (cm ²) (Ag wire): 14.7 14.7 14.7 Separator Thickness ³ , inches: 0.028 0.028 0.028
Hrs. to End Voltage (1.5/1.3v)**: 72/78 67/73 97/98 Anode (AZ31B Mg) Sheet-Thickness, ins.: 0.016 0.016 0.016 Anode Area (cm ²): 377 368 368 Cathode, Major Oxidant: HgSO4 HgSO4 HgSO4 gm. Major Oxidant: 133 137 126 gm. Sulfur: 10 5 20 gm. Carbon: 57 58 54 Additive: Additive, gms.: 2 Cathode Collector Area (cm ²) (Ag wire): 14.7 14.7 14.7 Separator Thickness ³ , inches: 0.028 0.028 0.028
Anode (AZ31B Mg) Sheet-Thickness, ins.: 0.016 0.016 0.016 Anode Area (cm ²): 377 368 368 Cathode, Major Oxidant: HgSO4 HgSO4 HgSO4 gm. Major Oxidant: 133 137 126 gm. Sulfur: 10 5 20 gm. Carbon: 57 58 54 Additive: MSO4· NH3 Coordination No. ⁵ : 2 2 2 Cathode Collector Area (cm ²) (Ag wire): 14.7 14.7 14.7 Separator Thickness ³ , inches: 0.028 0.028 0.028
Anode Area (cm^2): 377 368 368 Cathode, Major Oxidant: HgSO ₄ HgSO ₄ HgSO ₄ gm. Major Oxidant: 133 137 126 gm. Sulfur: 10 5 20 gm. Carbon: 57 58 54 Additive: 4dditive: 4dditive: 4dditive: MSO ₄ · NH ₃ Coordination No. ⁵ : 2 2 2 Cathode Collector Area (cm^2) (Ag wire): 14.7 14.7 14.7 Separator Thickness ³ , inches: 0.028 0.028 0.028
Cathode, Major Oxidant: $HgSO_4$ $HgSO_4$ $HgSO_4$ gm. Major Oxidant:133137126gm. Sulfur:10520gm. Carbon:575854Additive:Additive, gms.:MSO_4 · NH_3 Coordination No. 5 :222Cathode Collector Area (cm ²) (Ag wire):14.714.714.7Separator Thickness 3 , inches:0.0280.0280.028
gm. Major Oxidant: 133 137 126 gm. Sulfur: 10 5 20 gm. Carbon: 57 58 54 Additive: 4dditive: 10 10 MSO ₄ · NH ₃ Coordination No. ⁵ : 2 2 2 Cathode Collector Area (cm ²) (Ag wire): 14.7 14.7 14.7 Separator Thickness ³ , inches: 0.028 0.028 0.028
gm. Sulfur: 10 5 20 gm. Carbon: 57 58 54 Additive:
$gm. Carbon: 57 58 54$ $Additive: 57 58 54$ $Additive: 57 58 54$ $Additive: 57 58 54$ $MSO_4 \cdot NH_3 Coordination No. 5: 2 2 2 2$ $Cathode Collector Area (cm2) (Ag wire): 14.7 14.7 14.7$ $Separator Thickness3, inches: 0.028 0.028$
Additive:Additive, gms.:Additive, gms.:MSO4. NH3 Coordination No. 5:2Cathode Collector Area (cm²) (Ag wire):14.714.7Separator Thickness 3, inches:0.0280.028
Additive, gms.:Additive, gms.:MSO4· NH3 Coordination No. 5:22Cathode Collector Area (cm²) (Ag wire):14.714.7Separator Thickness 3, inches:0.0280.028
MSO ₄ · NH ₃ Coordination No. ⁵ : 2 2 2 Cathode Collector Area (cm ²) (Ag wire): 14.7 14.7 14.7 Separator Thickness ³ , inches: 0.028 0.028 0.028
Cathode Collector Area (cm ²) (Ag wire): 14.7 14.7 14.7 Separator Thickness ³ , inches: 0.028 0.028 0.028
Separator Thickness ³ , inches: 0.028 0.028 0.028
Initial Closed Circuit Voltage *: 2.15 2.10 2.15
Peak Closed Circuit Voltage*: 2.25 2.25 2.30
End Voltage (under maximum load) : 1.50 1.50 1.50
End Voltage (under light load): 1.70 1.70 1.70
Peak Anode C. D. (mA/cm ²)*: 0.44 0.45 0.46
Peak Cathode Collector C. D. (mA/cm ²)*: 11.3 11.3 11.5
Observed Coulombs/gram Oxidants ⁶ : 376 352 502
Observed F/mol Cathode Oxidants ⁶ : 0.78 0.91 0.76
Watt Hours/Inch ³ of Net Cell : 0.94 0.87 1.29

	T-38	T-39	T-40	T-41	T-42	T-43
	+Sulfur Temp.	+Sulfur Temp.	Cathode dia. 1.5"	Cathode dia. 1.13''	+Sulfur Temp	l. NH3 Activation Temp.
	А	А	А	А	A	С
	23	13	6	13,15	13,15	23
	A-624	A-624	ΤT	TT	A-624	A-624
	37	37	59	59	35	37
	:					
	+20	-73°	-63°	-63°	-73°	+20°
	11/19	68/77	48/72	84/107	63/85	0
	0.016	0.016	0.016	0.016	0.016	0.016
	368	368	248	368	368	368
	$HgSO_4$	$HgSO_4$	HgSO4	HgSO4	HgSO4	HgSO4
ł	133	133	126	126	126	ΙŹÓ
4 - 1	10	10	20	20	20	20
r	57	57	54	54	54	54
k						
1						
1	4	2	2	2	2	4
1	14.7	14.7	6.5	14.7	14.7	14.7
5	0.028	0.028	0.028	0.028	0.028	0.028
١	2.05	2.20	1.65	2.15	2.30	1.52
١	2.30	2.35	2.37	2.25	2.30	
•	1.50	1.50	1.50	1.50	1.50	
1	2.04	1.75	2.00	1.78	1.90	
	0.46	0.47	0.70	0.45	0.46	
ł	11.5	11.7	26.8	11.2	11.5	
1	58	365	253	430	326	
	0.12	0.76	0.38	0.65	0.49	
	0.12	0.76	0.34	0.55	0.72	

Bobbin Configuration - Load, $\Omega^2 = 2.7/13.5$

T-46

В-29

T-44

Test Number:

Major Variables:	+Sulfur	Temp. Mg tube anode	Active stand cathode only
Electrolyte:	А	А	А
Reference (Page No.):	13,17	5,23	51
Vehicle ¹ :	ТТ	A-624	TT
Net Cell Volume (in. ³):	40	35	40
Wet Stand, Hrs/Temp. °C:			64/-63°
Temperature °C, Discharge:	-63°	+20°	-63°
Hrs. to End Voltage (1.5/1.3v)**:	72/97	5.5/9.5	12/17
Anode (AZ31B Mg) Sheet-Thickness, ins.:	0.016	0.062	0.016
Anode Area (cm ²):	368	270	337
Cathode, Major Oxidant:	HgSO4	HgSO4	HgSO₄
gm. Major Oxidant:	112	126	126
gm. Sulfur:	40	20	20
gm. Carbon:	48	54	54
Additive:			
Additive, gms.:			
MSO_4 · NH ₃ Coordination No. ⁵ :	2	4	2
Cathode Collector Area (cm ²) (Ag wire) :	14.7	14.7	14.7
Separator Thickness ³ , inches :	0.028	0.028	0.028
Initial Closed Circuit Voltage * :	2.18	2.10	1.95
Peak Closed Circuit Voltage* :	2.27	2.10	2.05
End Voltage (under maximum load) :	1.50	1.50	1.50
End Voltage (under light load) :	1.90	1.84	1.67
Peak Anode C. D. $(mA/cm^2)*$:	0.45	0.57	0.45
	1	1	1

11.3

355

0.35

0.71

10.5

0.04

0.06

27

10.3

0.09

0.11

58

ELECTROCHEMICAL CELL TESTS-

T-45

Watt Hours/Inch³ of Net Cell :

Peak Cathode Collector C. D. $(mA/cm^2)*$:

Observed Coulombs/gram Oxidants⁶:

Observed F/mol Cathode Oxidants⁶:

Continued

	T-47	T-48	T-49	T-50	T-51	T-52	T-53
	HgSO₄ [·] 0NH₃ No S	Increased C & S	l. NH ₃ Activation Note 4	Activ. at - 63°	Slaked	Mg rod center anode	Center & outer anodes
	А	А	С	А	А	А	А
	14,15	17		15,23	15	5, 23	5,23
	TT	TT	ТТ	A-624	ТТ	A-624	A-624
	40	40	40	35	40	35	35
	-63°	-63°	-63°	+20°	-63°	+20°	+20°
	56/57	78/93	41	49/76	38	33/45	35/62
	0.016	0.016	0.016	0.016	0.016		
	368	368	368	368	368	140	508
ł	HgSO4	HgSO ₄	HgSO4	HgSO4	HgSO4	HgSO4	HgSO ₄
a a a	126	72	126	126	126	126	126
	0	36	20	20	20	20	20
ı	60	72	54	54	54	54	54
:							
,							
,	0	2	2	2	4	2	2
,	11.0	15.5	14.9	15.5	13.6	15.5	15.6
	0.028	0.028	0.028	0.028	0.028	0.028	. 032/. 028
¢	1.95	2.15	2.15	2.35	1.70	2.15	2.20
	2.00	2.25	2.20	2.37	2.15	2.22	2.20
	1.50	1.50	1.50	1.50	1.50	1.50	1.50
e e	1.92	1.82	1.82	1.70	1.92	1.97	2.00
1	0.40	0.45	0.44	0.47	0.43	1.17	0.32
	13.4	10.7	10.9	11.2	11.6	10.5	10.4
,	309	534	207	259	189	167	176
	0.95	0.44	0.31	0.39	0.28	0.25	0.27
	0.48	0.76	0.39	0.58	0.35	0.36	0.38
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ELECTROCHEMICAL CELL TESTS-

B-31

Test Number:	T-54	T - 55	T-56
Major Variables:	+Sulfur	Sulfur Only S:C = 1:3	Sulfur Only S:C = 1:3
Electrolyte:	А	A	A ,
Reference (Page No.):	13	17	17
Vehicle ¹ :	ΤТ	TT	TT
Net Cell Volume (in. ³):	40	40	40
Wet Stand, Hrs/Temp. °C:			
Temperature °C, Discharge:	-63°	-63°	-63°
Hrs. to End Voltage (1.5/1.3v)**:	71/92	41/45	33/45
Anode (AZ31B Mg) Sheet-Thickness, ins.:	0.016	0.016	0.016
Anode Area (cm ²):	368	368	368
Cathode, Major Oxidant:	HgSO4	S	S
gm. Major Oxidant:	122	30	30
gm. Sulfur:	25		
gm. Carbon:	53	90	90
Additive:			
Additive, gms.;			
MSO_4 · NH ₃ Coordination No. ⁵ :	2		
Cathode Collector Area (cm ²) (Ag wire) :	15.6	15.6	14.3
Separator Thickness 3 , inches :	0.028	0.028	0.028
Initial Closed Circuit Voltage* :	2.20	1.85	2.05
Peak Closed Circuit Voltage* :	2.25	2.05	2.06
End Voltage (under maximum load) :	1.50	1.50	1.50
End Voltage (under light load) :	1.85	1.80	1.90
Peak Anode C.D. (mA/cm ²)*:	0.45	0.41	0.41
Peak Cathode Collector C. D. (mA/cm^2) *:	10.6	9.7	10.6
Observed Coulombs/gram Oxidants 6:	361	968	775
Observed F/mol Cathode Oxidants ⁶ :	0.48	0.32	0.26
Watt Hours/Inch ³ of Net Cell :	0.69	0.36	0.29

Continued

				2				
Bobbin	Configuration	-	Load,	ິດ	Ξ	2.	7/13	3.5

			•			
	T-57	T-58	T-59	Т-60	_T-61	T-62
	m-DNB	m-DNB Active stand	Sulfur only S:C =1:2	HgSO4 · 0NH3	Coord. No. Temp.	Coord. No. Temp.
	A 29	A 29	A 17	A 10,11,	A	A
				14,15,17	15	15,23
	TT	TT	TT	TT	A-624	A-624
	40	40	40	40	35	35
		70/-63				
	-63°	-63°	-63°	-63°	-73°	+20°
	22/57	0/18	25/45	106/119	85/102	30/91
	0.016	0.016	0.016	0.016	. 040	. 040
	368	368	368	368	368	368
	m-DNB	m-DNB	S	HgSO4	HgSO4	HgSO ₄
	0C	30	12	106	106	106
a i	0	0		20	20	20
	90	90	84	60	60	60
•						
				0	0	0
	14.3	14.3	15.6	13.0	12.3	12.3
	0.028	0.028	0.028	0.028	0.028	0.028
	2.07	1.63	1.93	2.20	2.22	2.25
	2.07	1.83	2.13	2.25	2.28	2.35
	1.50	1.50	1.50	1.50	1.50	1.50
	1.85	1.73	1.85	1.72	1.88	1.82
	0.41	0.73	0.43	0.45	0.46	0.47
•	10.6	9.4	10.0	12.7	13.6	14.0
	524		431	628	506	187
1	0.91		0.14	0.84	0.67	0.25
l . • 1	0.19		0.23	1.03	0.96	0.37
		· · · · · · · · · · · · · · · · · · ·		.	· · · · · · · · · · · · · · · · · · ·	

ELECTROCHEMICAL CELL TESTS-

Test Number:	Т-63	T-64	Т-65		
Major Variables:	Wet stand Cathode Only	Thick Anode	Triple cathode,single anode		
Electrolyte:	А	А	А		
Reference (Page No.):	51	5	6		
Vehicle ¹ :	ТТ	TT	TT		
Net Cell Volume (in. ³):	40	40	40		
Wet Stand, Hrs/Temp. °C:	66/-63°				
Temperature °C, Discharge:	-63°	-63°	-63°		
Hrs. to End Voltage (1.5/1.3v)**:	10/16	71/88	63/74		
Anode (AZ31B Mg) Sheet-Thickness, ins.:	0.016	. 040	0.040		
Anode Area (cm ²):	368	368	245		
Cathode, Major Oxidant:	$HgSO_4$	$HgSO_4$	HgSO ₄		
gm. Major Oxidant:	120	120	106		
gm. Sulfur:	20	20	20		
gm. Carbon:	60	60	60		
Additive:					
Additive, gms.					
MSO_4 · NH ₃ Coordination No. ⁵ :	2	2	0		
Cathode Collector Area (cm ²) (Ag wire) :	15.6	15.6	27.2		
Separator Thickness ³ , inches :	0.028	0.028	0.016		
Initial Closed Circuit Voltage * :	2.15	2.17	2.25		
Peak Closed Circuit Voltage*:	2.15	2.25	2.25		
End Voltage (under maximum load) :	1.50	1.50	1.50		
End Voltage (under light load) :	1.85	1.78	1.65		
Peak Anode C. D. (mA/cm ²)*:	0.43	0.45	0.68		
Peak Cathode Collector C. D. $(mA/cm^2)*$:	10.1	10.6	6.1		
Observed Coulombs/gram Oxidants ⁶ :	52	379	374		
Observed F/mol Cathode Oxidants ⁶ :	0.08	0.56	0.50		
Watt Hours/Inch ³ of Net Cell :	0,09	0.69	0.61		

B-33

B-34

Continued

Bobbin Configuration - Load, $\Omega^2 = 2.7/13.5$

	т-66	Т-67	T-68	T-69	_T-70	T-71
	Pure Mg Anode	Spent electro- lyte	Spent electro- lyte	Cathode electro- lyte treated	Hg(SCN) ₂	HgS alone
	A	B	B	C	A	A
	5	33	23, 33	37	18,36	18
	TT	TT	A-624	TT	TT	ΤT
	40	40	35	40	40	40
	-63°	-63°	+20°	-63°	-63°	-63°
. <u> </u>	87/94	101/109	76/92	30/45	63/74	21/28
	0.014 ^d	. 040	0.040	0.040	0.040	0.040
	348	368	368 ^c	368	368	368
,	$HgSO_4$	HgSO ₄	HgSO ₄	$HgSO_4$	Hg(SCN) ₂	HgS
1	106	106	106	77	106	130
	20	20	20	15	20	0
,	60	60	60	45	60	56
,				KSCN		л
,				83		
	0	0	0	0	0	0
	11.7	11.7	11.7	14.2	14.2	14.2
	0.028	0.028	0.028	0.028	0.028	0.028
,	2.30	2.30	2.25	1.70	2.15	2.20
ł	2.30	2.34	2.25	2.25	2.25	2.20
	1.50	1.50	1.50	1.50	1.50	1.50
,	1.72	1.75	1.65	1.80	1.65	1.65
+	0.49	0.47	0.45	0.45	0.45	0.44
1	14.5	14.7	14.2	11.7	11.7	11.4
+	520	611	450	244	374	119
	0.69	0.81	0.60	0.32	0.51	0.29
r	0.87	1.03	0.85	0.29	0.61	0.20
	· · · · · · · · · · · · · · · · · · ·					

ELECTROCHEMICAL CELL TESTS-

	······		
Test Number:	T-72	T-73	T-74
Major Variables:	Spent electro- lyte	Hg(SCN) ₂ Temp.	Spent electro- lyte
Electrolyte:	В	А	D
Reference (Page No.):	33	18	33
Vehicle ¹ :	A-624	A-624	TT
Net Cell Volume (in. ³):	35	35	40
Wet Stand, Hrs/Temp. °C:			
Temperature °C, Discharge:	-73°	-73°	-63°
Hrs. to End Voltage (1.5/1.3v)**:	67/75	60/74	78/87
Anode (AZ31B Mg) Sheet-Thickness, ins.:	0.040	0.040	0.040
Anode Area (cm ²):	368	368	368 ^c
Cathode, Major Oxidant:	HgSO4	Hg(SCN)2	HgSO4
gm. Major Oxidant:	106	106	106
gm. Sulfur:	20	20	20
gm. Carbon:	60	60	60
Additive:			
Additive, gms.:			
MSO_4 · NH ₃ Coordination No. ⁵ :	0	0	0
Cathode Collector Area (cm ²) (Ag wire) :	11.7	14.2	11.0
Separator Thickness ³ , inches :	0.028	0.028	0.028
Initial Closed Circuit Voltage* :	2.20	2.24	2.00
Peak Closed Circuit Voltage* :	2.20	2.24	2.25
End Voltage (under maximum load) :	1.50	1.50	1.50
End Voltage (under light load) :	1.75	1.80	1.72
Peak Anode C.D. (mA/cm ²)*:	0.44	0.45	0.45
Peak Cathode Collector C. D. $(mA/cm^2)*$:	13.8	11.6	15.0
Observed Coulombs/gram Oxidants ⁶ :	390	354	462
Observed F/mol Cathode Oxidants :	0.52	0.48	0.62
Watt Hours/Inch ³ of Net Cell :	0.72	0.66	0.76 *

Continued

Bobbin Configuration - Load, $\Omega^2 = 2.7/13.5$

	T- 75	т-76	T-77	T-78	T-79	T-80			
	Cathode electrolyte treated	Amt. S	HgS + S	Hg(SCN) ₂ spent electrolyte	Wet stand Cath. only	Increased C & S			
	E	А	A	В	А	A			
	37	14,15	18	36	51	17			
	ТТ	TT	TT	TT	TT	TT			
	40	40	40	40	40	40			
					66/-63°				
	-63°	-63°	-63°	-63°	-63°	-63°			
	0/23	72/80	35/40	60/70	15/20	83/98			
	0 .040	0.040	0.040	0.040	0.040	0.040			
	368	258 ^c	368 ^c	368 ^c	258 ^c	310c			
	HgSO4	HgSO4	HgS	Hg(SCN) ₂	HgSO4	HgSO ₄			
، ۱	((116	106	106	106	75			
4	15	10	20	20	20	39			
e e	45	60	60	60	60	72			
	KSCN								
	83								
ţ	0	0	0	0	0	0			
•	14.2	12.3	15.6	15.6	11.6	13.6			
٢.	0.028	0.028	0.028	0.028	0.028	0.028			
	1.95	2.10	2.12	2.16	2.05	2.25			
t	1.95	2.23	2.20	2.34	2.15	2.25			
ŧ	1.50	1.50	1.50	1.50	1.50	1.50			
1	100 mm	1.73	1.78	1.75	1.70	1.85			
1	0.39	0.64	0.44	0.47	0.61	0.53			
(10.1	13.3	10.4	11.0	13.6	12.2			
۰.	05. mau	424	204	363	87	544			
	ann agu	0.79	0.25	0.49	0.12	0.44			
s		0.70	0.33	0.61	0.14	0.81			
				-					

ELECTROCHEMICAL	CELL	TESTS-

Test Number:	T-81	T-82	T-83
Major Variables:	electro- lyte and MgSO ₄	Triple cells	Amt. S electrolyte
Electrolyte:	F	А	G
Reference (Page No.):	22, 33	6	23, 36
Vehicle ¹ :	ТТ	ΤT	A-624
Net Cell Volume (in. ³):	40	40	35
Wet Stand, Hrs/Temp. °C:			
Temperature °C, Discharge:	-63°	-63°	+20°
Hrs. to End Voltage $(1.5/1.3v)$ **:	49/57	55/59	49/76
Anode (AZ31B Mg) Sheet-Thickness, ins.:	0.040	0.016	0.040
Anode Area (cm ²):	258 ^c	348 ^c	258 ^c
Cathode, Major Oxidant:	HgSO4	$HgSO_4$	HgSO4
gm. Major Oxidant:	106	106	116
gm. Sulfur:	20	20	10
gm. Carbon:	60	60	60
Additive:	MgSO ₄		
Additive, gms:	10		
MSO_4 · NH ₃ Coordination No. ⁵ :	0	0	0
Cathode Collector Area (cm ²) (Ag wire) :	13.0	23.3	12.3
Separator Thickness ³ , inches :	0.028	0.016	0.028
Initial Closed Circuit Voltage * :	1.92	2.13	
Peak Closed Circuit Voltage*:	2.20	2.25	2.33
End Voltage (under maximum load) :	1.50	1.50	1.50
End Voltage (under light load) :	1.87	1.68	1.65
Peak Anode C.D. (mA/cm²)*:	0.63	0.48	0.66
Peak Cathode Collector C. D. (mA/cm ²)*:	12.4	7.1	13.9
Observed Coulombs/gram Oxidants ⁶ :	286	326	297
Observed F/mol Cathode Oxidants ⁶ :	0.38	0.43	0.55
Watt Hours/Inch ³ of Net Cell :	0.46	0.54	0.57

Continued

T-84	T-85	T-86	T-87	T-88
 Amt. S electrolyte	Acetyl. Black	Increased C & S	Acetyl. Black electrolyte	Amt. S electrolyte
G	А	А	н	Н
 (Leaked)	17, 33	8,17	33	23, 36
A-624	TT	TT	TT	A-624
35	40	40	40	35
+20°	-63°	-63°	-63°	+20°
 12/13	79/95	83/96	85/93	67/78
 0.040	0.040	0.040	0.040	0.040
258 ^c	258 ^c	310 ^c	258 ^c	258 ^c
$HgSO_4$	HgSO4	HgSO4	HgSO ₄	HgSO ₄
106	106	50	106	116
20	20	50	20	10
60	54	80	54	60
	acet. blk.		acet. blk.	
	6		6	
0	0	0	0	0
11.3	12.3	14.9	13.0	12.3
0.028	0.028	0.028	0.028	0.028
	2.18	2.05	1.92	2. 27
2.35	2.25	2.25	2.24	2. 27
1.50	1.50	1.50	1.50	1,50
2.05	1.85	1.92	1.72	1.66
 0.67	0.64	0.53	0.64	0.65
15.3	13.5	11.1	12.7	13.6
73	468	620	501	400
0.10	0.62	0.37	0.67	0.74
0.14	0.77	0.81	0.82	0.75

ELECTROCHEMICAL CELL TESTS-

Test Number:	T-89	T-90	T-91
Major Variables:	Wet stand 3000 Ω	temp. coated anode	temp. electro- lyte
Electrolyte:	А	А	Н
Reference (Page No.):	51	23, 33	23, 33
Vehicle ¹ :	ТТ	A-624	A-624
Net Cell Volume (in. ³):	40	35	35
Wet Stand, Hrs/Temp. °C:	45/-63°		
Temperature °C, Discharge:	-63°	+20°	+20°
Hrs. to End Voltage (1.5/1.3v)**:	30/39	52/93	60/86
Anode (AZ31B Mg) Sheet-Thickness, ins.:	0.040	0.040	0.040
Anode Area (cm ²):	258 ^c	310 ^c	310c
Cathode, Major Oxidant:	HgSO4	HgSO4	HgSO ₄
gm. Major Oxidant:	106	106	106
gm. Sulfur:	20	20	20
gm. Carbon:	60	60	60
Additive:			
Additive, gms.:			
MSO_4 NH ₃ Coordination No. ⁵ :	0	0	0
Cathode Collector Area (cm ²) (Ag wire) :	11.0	11.7	13.0
Separator Thickness 3 , inches :	0.028	0.028	0.028
Initial Closed Circuit Voltage* :	2.13	2.23	2.41
Peak Closed Circuit Voltage*:	2.17	2.23	2.41
End Voltage (under maximum load) :	1.50	1.50	1.50
End Voltage (under light load) :	1.82	1.73	1.65
Peak Anode C. D. (mA/cm ²)*:	0.62	0.53	0.57
Peak Cathode Collector C. D. (mA/cm ²)*:	14.5	14.0	13.6
Observed Coulombs/gram Oxidants ⁶ :	174	307	370
Observed F/mol Cathode Oxidants ⁶ :	0.23	0.41	0.49
Watt Hours/Inch ³ of Net Cell :	0.28	0.57	0.73

B**-**39

B-40

Continued

	Bobbin Configuration - Load, $\Omega^2 = 2.7/13.5$										
	T-92	T-93	T-94	T-95	T-96	T-97					
	Wet stand 3000 Ω	25% wt. % KSCN electro- lyzed 3 mins.	Wet stand 645 Ω	Wet stand 3000 Ω High S & C	PbSO ₄ and Sulfur	Microporous rubber separator 0.045" Thk.					
	А	I	А	А	А	А					
	51	36	51	52	18,19,29	8					
	TT	ТТ	TT	ТТ	ΤT	TT					
	40	40	40	40	40	40					
	65/-63		65/-63	65/-63							
	-63°	-63°	-63°	-63°	-63°	-63°					
	41/58	75/89	41/50	45/54	34/53	107/119					
	0 .040	0.040	0.040	0.040	0.025	0.025					
	31 0 ^C	258 ^c	310 ^c	310 ^c	310 ^c	258c					
	HgSO4	$H_{g}SO_{4}$	HgSO4	$HgSO_4$	PbSO4	HgSO4					
•	106	106	106	50	108	106					
4 5 2	20	20	20	50	20	20					
t	60	60	60	80	60	60					
ı											
	0	0	0	0	0	0					
	11.7	11.7	11.7	14.3	13.6	11.7					
	0.028	0.028	0.028	0.028	0.028	0.036					
,	2.26	2.20	2.25	2.13	2.06	2.25					
	2.28	2.21	2. 25	2.20	2.06	2.28					
a	1.50	1.50	1.50	1.50	1.50	1.50					
	1.80	1.83	1.75	2.00	1.91	1.70					
4	0.54	0.63	0.53	0.52	0.49	0.65					
	14.3	13.9	14.1	11.3	11.1	14.3					
	244	440	243	331	188	637					
	0.32	0.58	0.32	0.20	0.25	0.85					
	0.40	0.72	0.40	0.42	0.30	1.05					

ELECTROCHEMICAL CELL TESTS-

Test Number:	T-98	Т-99	T-100
Major Variables:	3000 Ω MPR separator	High S & C	(NH ₄) ₂ - SO ₄
Electrolyte:	А	A	А
Reference (Page No.):	52	17	22
Vehicle ¹ :	ΤT	TT	TT
Net Cell Volume (in. ³):	40	40	40
Wet Stand, Hrs/Temp. °C:	65/-63		
Temperature °C, Discharge:	-63°	-63°	-63°
Hrs. to End Voltage (1.5/1.3v)**:	58/76	60/72	87/115
Anode (AZ31B Mg) Sheet-Thickness, ins.:	0.025	0.025	0.025
Anode Area (cm ²):	258 ^c	310 ^c	258 ^c
Cathode, Major Oxidant:	HgSO4	HgSO4	HgSO4
gm. Major Oxidant:	106	40	106
gm. Sulfur:	20	60	20
gm. Carbon:	60	80	60
Additive:			$(NH_4)_2SO_4$
Additive, gm s .:			10
$MSO_4 \cdot NH_3$ Coordination No. ⁵ :	0	0	0
Cathode Collector Area (cm ²) (Ag wire) :	13.0	15.5	12.3
Separator Thickness ³ , inches :	0.028	0.028	0.028
Initial Closed Circuit Voltage* :	2.27	2.25	2.20
Peak Closed Circuit Voltage* :	2.27	2.27	2. 25
End Voltage (under maximum load) :	1.50	1.50	1.50
End Voltage (under light load) :	1.75	2.00	1.75
Peak Anode C. D. (mA/cm ²)*:	0.65	0.54	0.64
Peak Cathode Collector C. D. (mA/cm^2) *:	12.8	10.8	13.5
Observed Coulombs/gram Oxidants ⁶ :	346	451	516
Observed F/mol Cathode Oxidants ⁶ :	0.46	0.23	0.69
Watt Hours/Inch ³ of Net Cell :	0.57	0.59	0.85

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Continued Bobbin Configuration - Load, $\Omega^2 = 2.7/13.5$

:	T-101	T-102	T-103	T-104	T-105	T-106
	50 wt. % KSCN	10 wt. % KSCN	MPR Sep. masked narrow 3000Ω	l'' dia. cathode	2700 Ω ½ spent electrolyte	MPR Sep. masked wide 2700 Ω
	J	E	А	А	В	Α
	35	35	52	6	51	52
	ТТ	ΤT	ТТ	ΤT	TT	TT .
	40	40	40	40	40	40
			65/-63		65/-63	65/-63
	-63°	-63°	-63°	-63°	-63°	-63°
	103/108	52/60	61/88	96/105	16/44	64/74
	0.025	0.025	0.025	0.025	0.025	0.025
	258 ^c	258 ^c	258 ^c	258c	258 ^c	258 ^c
2	HgSO ₄	HgSO4	$HgSO_4$	HgSO4	HgSO4	HgSO ₄
r T	106	106	106	106	106	106
	20	20	20	20	20	20
	60	60	60	60	60	60
	0	0	0	0	0	0
:	11.6	11.6	11.6	13.0	11.7	11.7
1	0.028	0.028	0.028	0.028	0.028	0.036
	2.28	2.26	2.25	2.22	2.15	2.21
I	2.28	2.26	2.25	2.30	2.18	2.22
i.	1.50	1.50	1.50	1.50	1.50	1.50
1	2.00	1.94	1.87	1.75	2.00	1.95
	0.65	0.64	0.64	0.66	0.62	0.63
1	14.5	14.3	14.3	13.0	13.7	13.9
r F	615	308	362	575	93	375
	0.82	0.41	0.48	0.77	0.12	0.50
I	1.01	0.51	0.59	0.96	0.15	0.61

ELECTROCHEMICAL CELL TESTS-

			_
Test Number:	T-107	T-108	T-109
Major Variables:	MPR double layer 2700 Ω	K ₂ SO ₄	Wet stand at +20°C,MPR .045'' thk.,3000Ω
Electrolyte:	А	А	А
Reference (Page No.):	52	22	52
Vehicle ¹ :	ΤΤ	ΤT	A-624
Net Cell Volume (in. ³):	40	40	35
Wet Stand, Hrs/Temp. °C:	65/-63		65/+20
Temperature °C, Discharge:	-63°	-63°	-63°
Hrs. to End Voltage (1.5/1.3v)**:	69/78	84/99	0/75 ^e
Anode (AZ31B Mg) Sheet-Thickness, ins.:	0.025	0.025	0.025
Anode Area (cm ²):	354 [°]	258 ^c	258 ^c
Cathode, Major Oxidant:	HgSO ₄	$HgSO_4$	HgSO ₄
gm. Major Oxidant:	106	106	106
gm. Sulfur:	20	20	20
gm. Carbon:	60	60	60
Additive:		K_2SO_4	
Additive, gms.:		10	
MSO_4 · NH ₃ Coordination No. ⁵ :	0	0	0
Cathode Collector Area (cm ²) (Ag wire) :	11.7	12.3	11.6
Separator Thickness ³ , inches :	0.028	0.028	0.036
Initial Closed Circuit Voltage* :	2.33	2.20	1.81
Peak Closed Circuit Voltage* :	2.33	2.24	1.81
End Voltage (under maximum load) :	1.50	1.50	0.80
End Voltage (under light load) :	1.70	1.70	0.98
Peak Anode C. D. (mA/cm ²)*:	0.48	0.64	0.52
Peak Cathode Collector C. D. (mA/cm ²)*:	14.6	13.4	11.5
Observed Coulombs/gram Oxidants:	417	496	307
Observed F/mol Cathode Oxidants ⁶ :	0.56	0.66	0.41
Watt Hours/Inch ³ of Net Cell :	0.70	0.81	0.40

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Continued

Bobbin Configuration -	Load,	$\Omega^2 = 2.7/13.5$
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	T-110	T-111	T-112	T-113		T-114		T-115	•
	m - DNB added	m-DNB replaced S	PbSO ₄ replaced HgSO ₄	MPR + <u>SA-6404</u> 3000Ω		MPR + SB-210 3000	Ω Ω	Incr. S & C MPR	
	A	А	А	А		А		А	
	29	29	19,29	52	- -	52		8	
	ТТ	ТТ	TT	A-624		A-624		TT	-
	40	40	40	35		35		40	
				65/-63		65/-63			w
	-63°	-63°	-63°	-63°	-63°		+20	-63°	
	106/121	89/102	34/104	69/84	0/3		36/82	85/94	
	0.025	0.025	0.025	0.025		0.025		0.025	
	258 ^c	258 ^c	310 ^c	258 ^C	•	258 ^c		387 ^c	
	HgSO4	HgSO4	PbSO ₄	$HgSO_4$		HgSO4		$HgSO_4$	U.
	106	106	108	106		106		50	
	20	0	20	20		20		50	
I	60	60	60	60		60		80	
:	m-DNB	m-DNB	m - DNB						
	10	20	10					-	- -
1	0	0	0	0		0		0	
	13. 0	11.6	15.5	11.6		11.6		14.3	
_	0.028	0.028	0.028	0.028		0.028		0.036	
	2.06	1.80	1.81	2.23	2.03		2.33	2.28	
	2.25	2.30	1.96	2.25	2.03		2.33	2.30	
	1.50	1.50	1.50	1.50	1.30		1.50	1.50	
	1.70	1.64	1.87	1.70	2.00		1.88	1.92	
	0.64	0.66	0.47	0.64	0.58		0.67	0.44	
	12.7	14.6	10.6	14.3	12.9		14.8	11.8	
	581	533	169	409			218	641	
	0.79	1.46	0.23	0.54			0.29	0.38	
	1.03	0.89	0.28	0.77			0.42	0.85	
	4			L	I			l	

CELL VOLTAGES ON SUCCESSIVE DAYS AND HOURS TO END VOLTAGES OF 1.5 AND 1.3

Test <u>No. T-</u>			oltage 3 days	After: <u>4 days</u>		Load Vo 2 days	0		Hrs. <u>1.5V</u>	1
50	1.85	1.50	1.35		2.00	1.70	1.60		49	76
51				Recor	der faile				38	
52	1.65	1.25		[2.02	1.80			33	45
53	1.70	1.40	1.20		1.90	1.60	1.45		35	62
54	2.05	1.85	1.49	1.24	2.25	2.10	1.85	1.60	71	92
55	1.70	1.25			2.00	1.60			41	45
56	1.62	1.23			2.00	1.75			33	45
57	1.47	1.23 1.41	0.90		1.82	1.73	1.14		22	57
58	1.17	1.11	0.70		1.02	1.13	1.11		0	
59	1.50	1.45	1.00		1.85	1.72	1.18		25	45
	2.15	2 10	1 05		2.24	2.25	2 15	1 0 0	1.0(110
60 61	2.15	2.10	1.95	1.55	2.26	2.25	2.15	1.80	106 85	119 102
62	1.98 1.74	$1.82 \\ 1.46$	1.76	1.42	2.20 1.96	2.16 1.73	2.00 1.65	1.70 1.42	30	91
63	0.85	1.40	1.44	1.15	1.90	1.75	1.05	1.42	10	16
64	2.05	1.86	1.48	1.10	2.25	2.12	1.77	1.40	71	87
04	2.05	1.00	1.40	1.10	2.25	2.12	1. / /	1.40		01
65	2.10	1.85	1.32	0.85	2.25	2.05	1.60	1.00	63	74
66	2,20	2.00	1.62	1.25	2.30	2.20	1.85	1.50	87	94
67	2.21	1.97	1.95	1.60	2.32	2.22	2.10	1.78	101	109
68	1.98	1.53	1.51	1.20	2.06	1.77	1.66	1.40	76	92
69	1.55	1.24			1.95	1.60			30	45
70	2.05	1.60	1.36	0.80	2.22	1.85	1.60	1.00	63	74
71	1.46	1.20			1.60	1.48			21	28
72	1.70	1.68	1.37		2.15	2.00	1.70		67	75
73	2.05	1,82	1.35		2.25	2.00	1.62		60	74
74	2.12	1.94	1.65	1.05	2.25	2.10	1.95	1.32	78	87
75	1.28	0.84			1.60	1.25			0	23
76	2.10	1.92	1.50	0.95	2.20	2.20	1.73	1.16	72	80
77	1.85	1.12	1.50	0.75	2.20	1.57	1.15	4.10	36	41
78	2.05	1.80	1.25		2.00	2.04	1.61		60	70
79	1.15	1.00	1.60)		1.42	D. U.			15	20
L					<u> </u>				<u> </u>	

* under heavy load

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TABLE V

(continued)

CELL VOLTAGES ON SUCCESSIVE DAYS AND HOURS TO END VOLTAGES OF 1.5 AND 1.3

Test <u>No. T-</u>			Voltage <u>3 days</u>	After: <u>4 days</u>			Voltage <u>3 days</u>		Hrs. <u>1.5V</u>	
80 81 82 83 84	2.12 1.87 2.08 2.05	1.95 1.51 1.73 1.50	1.65 1.00 1.35	1.35	2.25 2.10 2.20 2.20	2.15 1.90 1.92 1.65	1.98 1.30 1.60	1.67	83 49 55 49 Lea	98 57 59 76 ked
85 86 87 88 89	2.18 2.04 2.12 2.05 1.66	1.93 1.86 1.94 1.52	1.70 1.60 1.69 1.47	1.26 1.32 1.26 0.65	2.25 2.20 2.24 2.20 1.94	2.15 2.12 2.10 1.70	1.99 2.00 1.95 1.65	1.62 1.82 1.55 0.80	79 83 85 67 30	95 97 93 78 39
91 92 93 94	1.92 1.96 1.80 2.06 1.78	1.52 1.61 1.42 1.90 1.35	1.44 1.44 1.04 1.60	1.18	2.08 2.07 2.08 2.20 1.97	1.75 1.78 1.70 2.10 1.65	1.62 1.63 1.34 1.92	1.40	52 60 41 75 41	93 86 58 89 50
95 96 97 98 99	1.82 1.62 2.12 1.92 2.02	1.41 1.34 2.06 1.75 1.75	1.92 1.35 1.30	1.57	2.15 1.97 2.25 2.15 2.22	1.95 1.83 2.22 2.00 2.10	2.10 1.67 1.88	1.75	45 34 107 58 60	54 53 119 76 72
100 101 102 103 104	2.08 1.70 1.82 1.80 2.20	1.94 1.81 1.59 1.70 1.97	1.72 1.75 1.37 1.85	1.46 1.58 1.50	2.25 2.21 2.20 2.15 2.30	2.20 2.21 1.99 2.06 2.20	2.05 2.20 1.70 2.07	1.75 2.03 1.75	87 103 52 61 96	115 108 60 88 105
105 106 107 108 109 110	1.42 1.84 2.10 2.14 1.25 2.14	1.25 1.67 1.89 1.93 1.98	1.33 1.43 1.81 1.89	1.34	1.95 2.18 2.26 2.24 1.60 2.25	1.80 2.05 2.08 2. 16 2.14	1.84 1.68 2.04 2.05	1.63	16 64 69 84 0 106	44 74 78 99 3 121

* under heavy load

TABLE VI

ELECTROCHEMICAL CELL TESTS-

Test Number:	T-116	T-117	T-118		
Major Variables:	NaBr electrolyte 5.6 wt.%	PbSO4 no S Acet. Blk.	PbSO4 no S Acet. Blk.		
Electrolyte:	К	А	А		
Reference (Page No.):	37	19	19		
Vehicle ¹ :	ТТ	TT	A-624		
Net Cell Volume (in. ³):	40	40	35		
Wet Stand, Hrs/Temp. °C:					
Temperature °C, Discharge:	-63°	-63°	+20		
Hrs. to End Voltage (1.5/1.3v)**:	67/75	0/33	65/70		
Anode (AZ31B Mg) Sheet-Thickness, ins.:	0.025	0.025	0.025		
Anode Area (cm ²):	258 ^c	258 ^c	258 ^c		
Cathode, Major Oxidant:	$HgSO_4$	PbSO ₄	PbSO ₄		
gm. Major Oxidant:	106	120	120		
gm. Sulfur:	20	0	0		
gm. Carbon:	60	55	55		
Additive:		Acet. Blk.	Acet. Blk.		
Additive, gms.		5	5		
MSO_4 · NH ₃ Coordination No. ⁵ :	0	0	0		
Cathode Collector Area (cm ²) (Ag wire) :	12.9	11.6	11.6		
Separator Thickness ³ , inches :	0.028	0.028	0.020		
Initial Closed Circuit Voltage * :	1.83	1.75	2.26		
Peak Closed Circuit Voltage*:	2.10	1.75	2.26		
End Voltage (under maximum load) :	1.50	1.30	1.50		
End Voltage (under light load) :	1.82	1.70	1.72		
Peak Anode C.D. $(mA/cm^2)*$:	0.60	0.50	0.64		
Peak Cathode Collector C. D. (mA/cm^2) *:	12.0	11.1	14.3		
Observed Coulombs/gram Oxidants ⁶ :	380	167	370		
Observed F/mol Cathode Oxidants ⁶ :	0.51	0.53	1.16		
Watt Hours/Inch ³ of Net Cell :	0.60	0.21	0.61		

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Bobbin Configuration - Load, $\Omega^2 = 2.7/13.5$

 T-119	T-120	T-121	T-122	T-123	T-124
-90° electrolyte	NaBr electrolyte 5.6 w% No S	PbSO ₄ wet stand 3000 Ω	PbSO ₄ wet stand 3000 Ω	PbSO ₄ cathode wet stand	Zn plate in NH3
L	к	А	А	A	A
56,57	37	54	54	54	49
TT	A-624	A-624	A-624	ТТ	TT
40	35	35	35	40	40
		40/+20	65/20	65/20	
-90°	+20		+20	-63°	-50°
 21/27	0/0		77 t o 0. 52V	13/22	Mg Zn
0.025	0.025	0.025	0.025	0.025	0.040 0.025
258 ^c	258 ^c	258c	258	258c	232 250
HgSO4	$HgSO_4$	PbSO ₄	PbSO ₄	PbSO4	PbSO4
106	126	120	120	100	τŹÛ
20	0	0	0	0	0
60	60	55	55	50	60
		Acet. blk.	Acet. blk.		
		5	5		
0	0	0	0	0	0
11.6	10.4	11.6	11.6	10.4	11.6
0.020	0.020	0.020	0.020	0.020	0.020 0.020
 2.20	1.80		1.17	1.61	
2.20	1.80		1.17	1.75	(Refer to text)
1.50		. 	0.52	1.50	
2.15			0.65	1.75	
0.63	0.51		0.33	0.50	
13.9	12.7		7.4	12.4	
122			217	84	
0.16			0.68	0.27	
0.20			0.18	0.10	

Discharged Plates From

Test Number:	S-1	S-2	<u>S-3</u>
Major Variables:	2 Exmet anodes 1 disch. (+) cath.	l disch. (-) cath	l Exmet 2 (-) cath.
Electrolyte:	33% KSCN	33% KSCN	25% KSCN
Reference (Page No.):			
Vehicle ¹ :			<u> </u>
Net Cell Volume (in. ³) :			
Wet Stand, Hrs/Temp.°C:			
Temperature °C, Discharge:	-63	-63	-63
Hrs. to End Voltage (1.5/1.3V)** :	7.5/10.3	3/4	1/1.5
Anode (AZ31B Mg) Sheet-Thickness, ins. :	Exmet 2 anodes	Exmet 2 anodes	Exmet 1 anode
Anode Area (cm ²):	272	272	272
Cathode, Major Oxidant:	$PbSO_4$	PbSO ₄	PbSO4
gm. Major Oxidant:			
gm. Sulfur:			
gm. Carbon:			
Additive:			
Additive, gms.:			
MSO_4 NH ₃ Coordination No. $\frac{5}{2}$			
Cathode Area (cm ²) :	287	287	287
Separator Thickness ³ , inches:	. 052	052	0.52
Initial Closed Circuit Voltage*:	2.30	1.70	1.70
Peak Closed Circuit Voltage *:	2.30	1.75	1.76
End Voltage (under maximum load) :	1.50/1.30	1.52/1.36	1.57/1.57
End Voltage (under light load) :	1.62/1.43	1.74/1.65	1.77/1.65
Peak Anode C. D. (mA/cm ²)*:	2.6/35	2.1/12.3	2.1
Peak Cathode C. D. (mA/cm ²) * :			
Observed Coulombs/gram Oxidants ⁶ :			
Observed F/mol Cathode Oxidants ⁶ : Watt Hours/Inch ³ of Net Cell:			
S.C. Current (Total)-Amperes:	10	3.5	

ELECTROCHEMICAL CELL TESTS - Load, $\Omega^2 = 2.7/13.5$

Aqueous Lead-Acid Batteries

	S-4	S-5	S-6	S-10	S-13	S-14
				Working anode		
	l Pure	l dry chg.	2(S-4)	. 003'' Mg	No salt	-
	Mg anode	(+) plate	cells in	control anode	in electro-	Electrolyte
	2 (-) cath.	as cath.	parallel	. 009'' Mg	lyte	from S-13
	25%KSCN	25%KSCN	25%KSCN	25%KSCN	L.NH ₃	$MgSO_4$ in L. NH_3
			·			
					:	
	-55	- 55	-55	- 55	- 55	- 55
	11/11.5		9/10.3	1/2		
	.015 ^d	.015 ^d	. 015d	. 003 ^d	.015 ^d	. 015 ^d
	272	31	544	134	272	272
	PbSO ₄	PbO₂		PbSO₄	PbSO₄	PbSO₄
	F0504	FDO ₂	$PbSO_4$	FDSO4	F 0504	F 5504
				l		
				i		
i						
1						
i	287	31	574	287	287	287
	. 052	. 052			. 052	. 052
			. 052	. 052	. 052	
	1.80	1.70	1.81	1.72		1.58 o.c.
	1.83	1.74	1.83	1.81		1.62 o.c.
1	1.50/1.3		1.50/1.28	1.52/1.31		
· · ·	1.68/1.55		1.63/1.50	1.76/1.73		
	2.2/12.3	12/32.5	1.4/22.5	2.1		
1						
+ -						
	3.5 at end					max. cur-
	of run	1	13			rent.022

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TABLE VII

ELECTROCHEMICAL CELL TESTS-

Test Number:	T-125	T-126
Test Number.	1-125	1-120
Major Variables:	Coord. No.	Temp. Acet. blk.
Load, Ω^2 :	2.7/13.5	2,7/13.5
Electrolyte Wt. % KSCN:	25	35
Reference (Page No.):	19	56,
Vehicle ¹ :	TT	ТТ
Net Cell Volume (in. ³):	40	40
Wet Stand, Hrs./Temp. °C:		
Temperature °C, Discharge:	-50°	-90°
Hrs. to End Voltage (1.5/1.3v)**:	33/49	0
Anode (AZ31B Mg) Sheet-Thickness, ins.:	0.025	0.025
Anode Area (cm²):	232 ^c	232 ^c
Cathode, Major Oxidant:	PbSO ₄	HgSO ₄
gm. Major Oxidant:	100	100
gm. Sulfur:	0	20
gm. Carbon:	45	55
Additive:	Acet. blk.	Acet. blk.
Additive, gms.:	5	5
MSO_4 · NH ₃ Coordination No. ⁵ :	2	0
Cathode Collector Area (cm ²) (Ag wire):	11.6	11.0
Separator Thickness ³ , inches:	0.028	0.028
Initial Closed Circuit Voltage*:	1.63	2.17
Peak Closed Circuit Voltage*:	1.75	2.17
End Voltage (under maximum load):	1.50	1.27
End Voltage (under light load):	1.74	2.15
Peak Anode C. D. (mA/cm ²)*:	0.55	
Peak Cathode Collector C. D. (mA/cm ²)*:	11.1	
Observed Coulombs/gram Oxidants ⁶ :	213	
Observed F/mol Cathode Oxidants ⁶ :	0.67	
Watt Hours/Inch ³ of Net Cell:	0.24	

Bobbin Configuration

Contractor

j.

,								
	T-127		T-128		T-129	T-130 ·		
	Wet stand Zn anode	Dis- charge Mg anode	Te	empera	ture	Zinc plated anode	Size Temp. Ratio	
	0	2.7/13.5		2.7/13	3.5	47/220	47/220	
	25			34		25	35	
	54			58		47	56,59	
	A-601	TT		ΤТ		TT	TT	
	24	40		40		1.2	1.2	
	65/30							
		-50°	-90°	-50°	- 85°	-50°	-90°	
		0	35	79	193		2	
	0.015	0.025		0.025		0.015	0.016 ^f	
	206	232 ^c		232 ^c		14	14	
	Ph	SO <u>₄</u>	HgSO4		PbSO ₄	HgSO ₄		
	10	0	106		2.68	2.8		
	0		20		0	0		
	45		60		1.32	1.2		
	Acet.	Blk.						
	5		 					
	2		0		2	2		
	11	. 6	11.6		1.62	1.62		
, 	0.	028		0.020	· · · · · · · · · · · ·	0.008	0.008	
)		0.90	2.15	2.26	1.62	1.45	2.01	
		0.90	2.15	2.26	1.62	1.90	2.06	
ł		0.88	0.65	1.88	0.63	1.50	1.50	
		0.90	1.67	2.05	0.80	1.57	1.65	
			0.69	0.72	0.52	0.62	0.67	
			13.7	14.4	10.7	5.32	5.78	
			154	287			29	
			0.20	0.38			0.10	
			0.19	0.52			0.03	

TABLE VII

ELECTROCHEMICAL CELL TESTS-

-				
Test Number:	T-131	T-1	32	
Major Variables:	Size temp. ratio	Pasted P SM-91 Sep		
Load, Ω^2 :	47/220	2.7,	/13.5	
Electrolyte Wt. % KSCN:	25	34		
Reference (Page No.):	20	7,50	5,59	
Vehicle ¹ :	ТТ	P.E	. Bag	
Net Cell Volume (in. ³):	1.5	7.5		
Wet Stand, Hrs./Temp. °C:				
Temperature °C, Discharge:	-50°	-90°	-50°	
Hrs. to End Voltage (1.5/1.3v)**:	4.5/4.6	9/13	2.5/5.2	
Anode (AZ31B Mg) Sheet-Thickness, ins.	0.016 ^f	0.0	15 ^d	
Anode Area (cm ²):	18	232		
Cathode, Major Oxidant:	$PbSO_4$	HgSO ₄		
gm. Major Oxidant:	1.5	18.9		
gm. Sulfur:	0	3.8		
gm. Carbon:	1.5	11.3		
Additive:		Bin	der	
Additive, gms.:		0.5		
$MSO_4 \cdot NH_3$ Coordination No. ⁵ :	2	0		
Cathode Collector Area (cm ²)(Ag wire):	1.30	232 ⁱ		
Separator Thickness ³ , inches:	0.008g	0.0	10 ^h	
Initial Closed Circuit Voltage*:	1.84	2.22	1.87	
Peak Closed Circuit Voltage*:	1.84	2.22	1.87	
End Voltage (under maximum load):	1.50	1.50	1.50	
End Voltage (under light load):	1.54	1.98	1.70	
Peak Anode C.D. (mA/cm ²)*:	0.46	0.71	0.60	
Peak Cathode Collector C. D. (mA/cm^2) *:	6.43	0.71	0.60	
Observed Coulombs/gram Oxidants ⁶ :	117	293	74	
Observed F/mol Cathode Oxidants ⁶ :	0.41	0.38	0.10	
Watt Hours/Inch ³ of Net Cell:	0.06	0.46	0.10	

B-54

Continued

Bobbin Configuration

	T-133	T-134	T-135	T-	136	T-137
	Size temp. ratio	Wet stand, MPR .028" thk., No load	Size temp. ratio	0.02	stand,MPR 8'' thickness, 2 load	Size temp. ratio
	47/220	2.7/13.5	47/220	2.	7/13.5	47/220
	35	25	35	25		25
	20, 56	53, 54	20, 56	54		20
)	TT	ТТ	ТТ	A-	601	ТТ
	1.5	40	1.5	24		1.5
		65/30		18/-50+	48/+30	
	-85°	-50°	- 85°	- 5	0 °	-50°
	9/9	34/38	10/10	0/	28	6.8/6.9
	0.016 ^f	0.025	0.016 ^f	0.	025	0.016 ^f
,	18	232	18	23	2	18
i i	$PbSO_4$	$PbSO_4$	$PbSO_4$	$PbSO_4$		$PbSO_4$
,	1.5	86	2	94		2
	0	0	0	0		0
	1.5	42	1	46		1
	2	2	2	2		2
T	1.30	10.4	1.30	10	. 4	1.30
1	0.008 ^g	0.020	0.008 ^g	0.	020	0.008g
,	1.72	1.74	1.70	1.72 [,]	1.72	1.84
.	1.75	1.80	1.78	1.77	1.77	1.84
	1.50	1.50	1.50	1.50	1.30	1.50
	1.70	1.73	1.70		1.67	1.55
	0.44	0.57	0.45	0.56	0.56	0.47
	6.12	12.8	6.23	12.6	12.6	6.45
	229	259	194		182	133
	0.80	0.90	0.68		0.64	0.46
	0.11	0.26	0.12		0.31	0.09
·			·		L	

TABLE VII

ELECTROCHEMICAL CELL TESTS-

Test Number:	T-13	38	T-139					
Major Variables:	in cente:	collector r/Ag Exmet r outside	Webril M1365= Webril SM91=					
Load, $\mathbf{\Omega}^2$:	2.7/	13.5	2.7/13.5					
Electrolyte Wt. %KSCN:	34							
Reference (Page No.):	7,56	, 59						
Vehicle ¹ :	ТТ							
Net Cell Volume (in. ³):	40							
Wet Stand, Hrs./Temp.°C:								
Temperature °C, Discharge:	-90°	-85°	-85°					
Hrs. to End Voltage (1.5/1.3v)**:	7/19	0/28	22					
Anode (AZ31B)Mg) Sheet-Thickness, ins.:	0.02	25						
Anode Area (cm ²):	232 ^c							
Cathode, Major Oxidant:	HgSO4							
gm. Major Oxidant:	100							
gm. Sulfur:	20							
gm. Carbon:	60							
Additive:								
Additive, gms.:								
MSO_4 NH ₃ Coordination No. ⁵ :	0							
Cathode Collector Area (cm ²) (Ag wire) :	11.0/192 ^j							
Separator Thickness ³ , inches:	0.028							
Initial Closed Circuit Voltage*:	2.10		1.15					
Peak Closed Circuit Voltage*:	2.17	2.07	1.15					
End Voltage (under maximum load):	1.50	1.30	0.35					
End Voltage (under light load):	2.11	1.94	0.87					
Peak Anode C. D. (mA/cm^2) *:	0.69	0.69	0.37					
Peak Cathode Collector C.D. $(mA/cm^2)*:$	14.6	0.80	7.75					
Observed Coulombs/gram Oxidants ⁶ :	43	156						
Observed F/mol Cathode Oxidants ⁶ :	0.06	0.20						
Watt Hours/Inch ³ of Net Cell:	0.07	0.22						

Continued

Bobbin Configuration

	T-139	(Continued)	T-140	T-141	T-142	T-143	T-144
			KSCN conc.	KSCN conc.	Size load	Size load	Size load
	2.7/13.5	o.c./13.5	47/220	47/220	6.67	6.67	6.67
	34		34	34	25	25	25
	19, 20, 56		20	20	18	18	18
	TT		ТТ	TT	TT	TT	TT
	40		1.5	1.5	1.2	1.2	1.2
	-50°	-50°	-50°	~50°	-50°	-50°	-50°
	95	114	7.4/7.5	7.8/8.1	0.73	0.46	0.01
	0.025		0.016 [±]	0.016 ¹	0.016 ^f	0.016 [[]	0.016 ^f
	232		18	18	14	14	14
	$PbSO_4$		$PbSO_4$	PbSO ₄	HgSO4	PbSO ₄	$Sn(SO_4)_2$
	87		1.5	2	2.8	2.8	2.8
	0		0	0	0	0	0
	43		1.5	1	1.2	1.2	1.2
	2		2	2	2	2	4/6
	11.0		1.30	1.30	1.62	1.62	1.62
	0.016		0.008g	0.008g	0.008	0.008	0.008
	1.53	1.34	1.83	1.84	1.58	1.23	1.27
	1.62	1.34	1.85	1.87	1.98	1.50	1.42
	0.83		1.50	1.50	1.78	1.35	1.28
	1.30	1.22	1.55	1.74			
	0.52	0.43	0.47	0.47	21.2	16.0	15.2
	10.8	9.03	6.47	6.53	183	139	131
			198	1.59	265	127	2.60
			0.69	0.56	0.91	0.44	0.01
			0.10	0.11	0.32	0.12	0.00