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DEVELOPMENT OF A LOW TEMPERATURE BATTERY FOR SPACE PROBE APPLICATIONS

SECOND QUARTERLY REPORT

William F. Meyers, Principal Investigator

September 24, 1964 to December 23, 1964

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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SUMMARY

21276

The three phases of this work are to obtain: (I) 72-hour battery performance from liquid ammonia batteries over the temperature range -40 °C to -73°C; (II) limited wet stand capabilities to +70°C; and (III) cell performance at -90°C.

Reproducible low temperature cell performance has been accomplished this quarter at -63°C by the addition of small quantities of sulfur into the cathode of the magnesium/mercuric sulfate bobbin cell. Performance at the upper temperature is presently limited by aspects of Phase II.

The low temperature performance goal has also been exceeded with the magnesium/meta-dinitrobenzene couple by the use of a spiraled flat plate configuration. This cell also performed for the required time at +20°C to a slightly lower end voltage.

Difficulties encountered with suitable cell containers or vehicles and environmental requirements of Phases I and II have been largely overcome, and an approach to reproducibility has been established.

Author

INTRODUCTION

The first objective of this program is to obtain 2.1-watt, 72-hour reserve battery performance over the temperature range -40° C to -73°C by modification of existing Livingston Ammonia Battery designs.

The second objective is to develop limited wet stand capability to an upper temperature of +70°C.

During the first quarter, cell test chambers were obtained and tested. Numerous difficulties associated with hermetic seals on these test chambers below -55°C were encountered. Cell tests were made in spite of these difficulties, while other test chambers were being studied for possible use.

The first quarter's approach was to obtain operation in the moderate temperature region of +20°C to -55°C and then to test similar cells at -73°C. Three cathode reactants, mercuric sulfate, meta-dinitrobenzene, and sulfur were examined. Magnesium/mercuric sulfate cells were tested in a bobbin structure due to the naturally high current handling capability of mercuric sulfate. Meta-dinitrobenzene and sulfur were similarily tested in flat-plate cell structures in order to enhance their current handling capabilities by geometric means.

In the second quarter, considerable effort was focused upon improvement of the cell vehicles; and three useful cell containers evolved. Difficulties associated with continuous operation at the lower temperatures were overcome early in the quarter by providing -63°C within test tubes and -73°C testing in A-622 and A-624 vehicles. The availability of the test tube vehicle has been extremely helpful in permitting observation and modifications to cells during operation. The approach during the second quarter was re-oriented towards obtaining operation at the lower temperatures, -63°C and -73°C, with subsequent evaluation at +20°C.

The work is still largely an evolution in monovariant steps in order to meet at 72-hour life requirement from +20°C to -73°C. The data which are included in the report infer a satisfactory improvement in performance as follows:

	Cathode		
1.	Mercuric sulfate + sulfur	>72 hours at -63°C	Bobbin Cell
2.	Meta-dinitrobenzene	>72 hours at -73°C	Spiraled Flat Cell

Some difficulty in obtaining the 72-hour life to the arbitrarily selected cut-off 1.5 volts at room temperature with magnesium/ meta-dinitrobenzene was observed. However, the capacity-potential data shows a substantial degree of reproducibility to 1.3 volts.

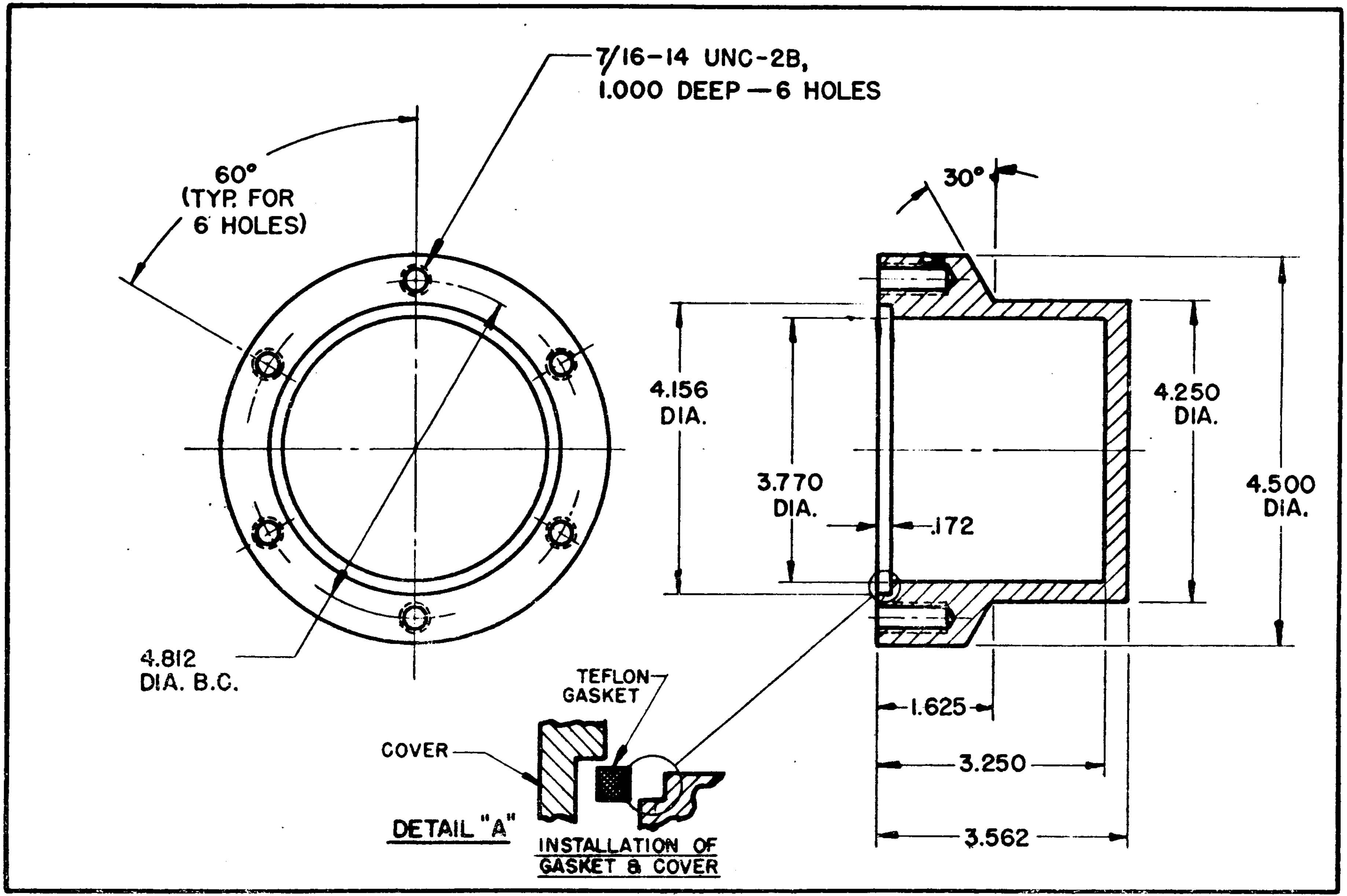
It is interesting to note that sulfur was shelved at the end of the first quarter due to the relatively poor low temperature performance in flat plate cells. However, the addition of relatively small amounts of sulfur to the mercuric sulfate bobbin has served to extend performance to the required period at low temperature.

TEST HARDWARE

During the course of the fifth month, an improved seal was developed for the A-622 test chamber, Figure I, page 4. The cells sealed in this chamber can be discharged in the carbon dioxide refrigerator at -73°C.

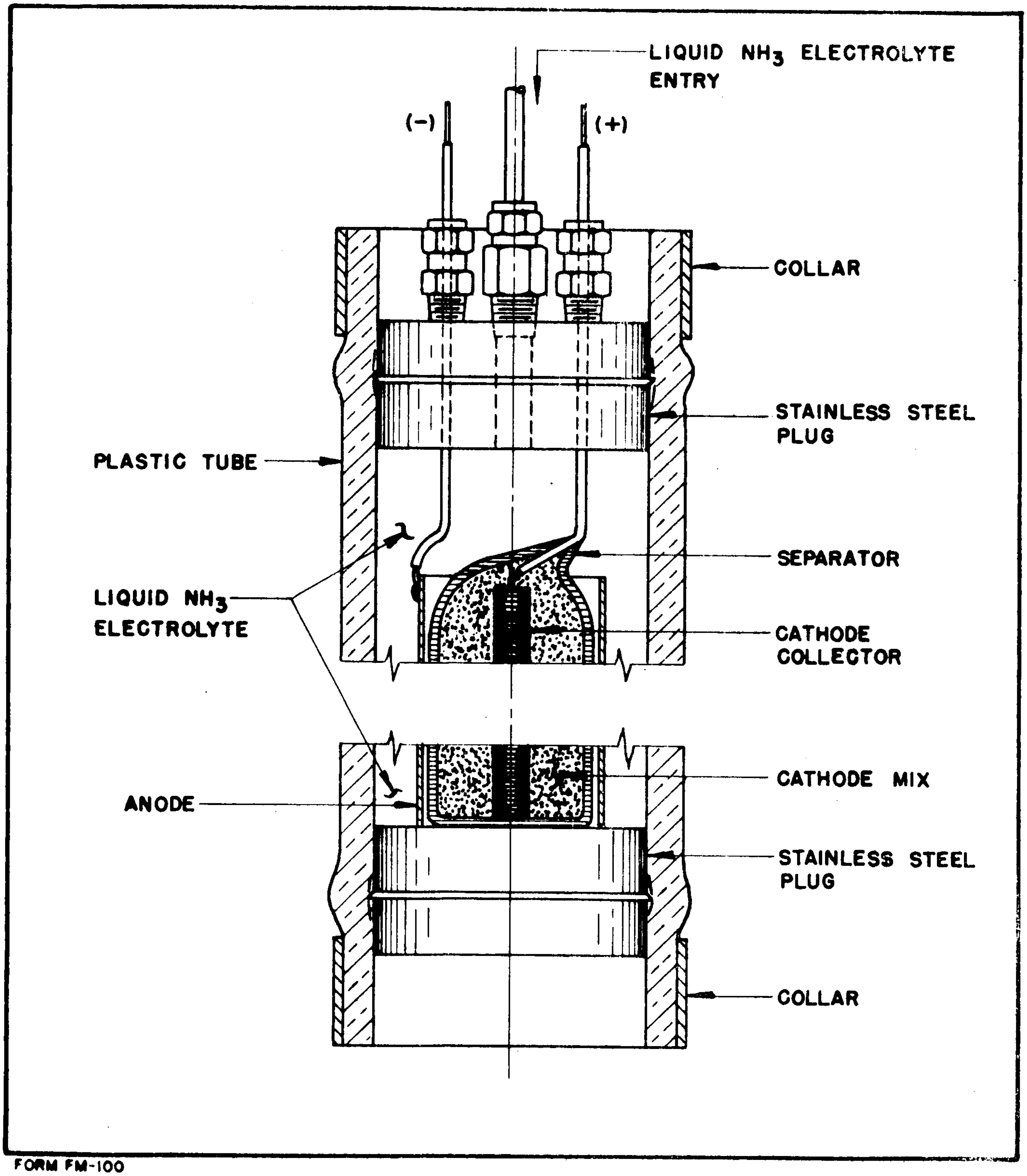
A second test chamber was also evolved during this quarter. This latest test vehicle makes use of disposable, cross-linked polyethylene pipe and re-usable stainless steel end plugs, Figure 2, page 5. Preliminary hydraulic and pneumatic tests conducted on this container indicated that it could withstand pressures in excess of 1,000 pounds per square inch at room temperature. The versatility of this plastic pipe makes changing the cell length an easy task. This container may also be used at -73°C without fear of contamination in the CO_2 refrigerator.

One of our mechanical refrigeration units was re-designed and rebuilt and now provides a continuous operating temperature of -63°C. In this refrigerator, open pyrex test tube cells can be tested continuously without sealing and without loss of electrolyte.



A-622 TEST CHAMBER

FIGURE 1



FORM FM-100

A-624 TEST VEHICLE

FIGURE 2

MAGNESIUM/MERCURIC SULFATE BOBBIN CELLS

Refer to Table I

The mercuric sulfate bobbin cells tested during the second quarter are listed in Table I, page 16. Many of the cells were enclosed in a test tube 1.8 inches in diameter by 16 inches in length. This test tube was immersed in cold circulating alcohol at -63°C, providing an operating cell of the full diameter which could be examined periodically throughout discharge. Many tests were also conducted within the stainless steel and plastic A-624 vehicle.

The majority of these cells were comprised of a bobbin containing $HgSO_4 \cdot 2NH_3$ and carbon (in the ratio of 7 : 3) with a silver cathode collector and a sheet of magnesium alloy wrapped around it. After construction, most of the bobbins were subjected to vacuum to remove moisture and air from the cathode mix. Following this, the cell was inserted in a vehicle and activated with a 25 weight percent solution of KSCN in liquid ammonia.

The cells were discharged under cyclic resistive loads to provide a nominal 0.3 watt for 54 minutes and 1.5 watts for 6 minutes based upon 2 volts/cell.

Description of Cell Tests

Cell number T2 was placed in a Pyrex test tube so that the corrosion effects and gas evolution could be observed. It performed very well for 44 hours down to 2.0 volts, at which time the voltage dropped suddenly. It is probable that failure was caused by corrosion at the lead wire connection to the anode.

Number T3 was essentially the same as T2, except that the A-624 vehicle (without top closure) was used to determine whether the plastic case had a detrimental effect on the cell performance. After 39.5 hours, the lead connection was lost due to corrosion of the expanded metal anode (Exmet); as a result, a new solid sheet anode was installed. The discharge was then continued until the voltage under the heavy load dropped to 1.5 after 70 hours.

In Number T4 the anode was made of a solid sheet to prevent loss of contact with the anode by corrosion as was observed in T2 and T3. The cell performed continuously for 63 hours to an end voltage of 1.5 at the heavy load.

In T5 the top plug of the A-624 case was inserted to determine the effect of retaining the gas produced during discharge. The life at -63°C was reduced from 63 hours to 57 hours. The discharge was resumed after three days on open circuit, and then it ran an additional 3.5 hours. It recovered again at room temperature and continued for 6.5 hours more to 1.5 volts. The increments in Faradays per mol were 1.11, 0.07, and 0.13, respectively, for a total of 1.31.

The weight of the cathode in Number T6 was increased substantially with no increase in life. The fact that the apparent density of the cathode mix was increased about 20 per cent over that of T5 may have some bearing on the results obtained.

A blockage in the filling tube caused the loss of Cell Number T7.

Both T8 and T9 were "slaked" with ammonia gas to convert $HgSO_4 \cdot 2NH_3$ to $HgSO_4 \cdot 4NH_3$ and to liberate the heat of this reaction prior to activation. After slaking, the cells were activated with electrolyte from lecture bottles under a pressure of 200 pounds per square inch gauge. The pressure was maintained during discharge with compressed argon.

In an attempt to gain the required life at +20°C, the cathode of Cell T8 was not only "slaked," but its diameter was increased from the usual 1-1/8" to 1-5/8" in order to increase its porosity. In 16 hours, the voltage decreased under heavy load to 1.5. At the same time, however, the light load voltage was 2.17.

T9 was similar to T6 except for the slaking, the pressure activation, and a reduction in cathode apparent density from 16 to 11 grams per cubic inch. The performance of T9 was only about half of that obtained from T6.

Cell T10 was an inverse bobbin in which the center anode was enclosed in separator paper. The cathode mixture was packed between the anode envelope and the test tube wall. The cathode density was so great that it absorbed only a small amount of electrolyte solution. Therefore, the cell voltage under load was less than 1.5 and did not increase after standing for 24 hours.

Cells T11 and T17 were tested primarily to determine a suitable geometry and anode to cathode spacing for conventional bobbin cell construction. The first three cathodes were 1-1/2" in diameter and did not perform well; so the diameter was decreased to 1-3/16" and then to 1-1/8". The anode to cathode annular spacing was increased to about 1/4". These changes appeared to have increased life at each temperature.

Other factors influenced the room temperature tests. For example, T12 developed a minor electrolyte leak and appeared to have a poor connection. T17 vented after 16 hours, at which time the voltage was 1.97 under the heavy load.

In T11 and T16, the temperature was increased from -73°C to ambient after the end voltage under maximum load had reached 1.50. In each case, the voltage recovered to approximately the original peak and additional discharge time was obtained: 46 hours from T11 and 36 hours from T16.

T18 and T19 were similar except for anode area. A 16 per cent reduction in area yielded 10 per cent fewer hours. Here again, an increase in temperature resulted in a sharp recovery in voltage.

T20 and T21 were similar to T18 and T19 except that they were treated with ammonia vapor (slaked) to convert $HgSO_4 \cdot 2NH_3$ to $HgSO_4 \cdot 4NH_3$ and dissipate the heat of reaction prior to activation. Since the average discharge time of T18 and T19 was 56 hours and that of T20 and T21 was 53 hours, there seems to be no advantage in slaking when activation is carried out at low temperature.

Shawinigan acetylene black (dried and micronized) was used in T22 (5 grams) and in T25 (9 grams) to improve the porosity and conductivity of the cathodes. The average life of T18 and T19 with no additive was 56 hours as compared with 55 hours for T22. T25, containing 9 grams of acetylene black, yielded only 39 hours in contrast with an average life of 53 hours in the case of T20 and T21 which contained none. The results demonstrate no advantage at -63°C through the use of acetylene black. However, it was noted that the rate of absorption of the electrolyte was improved; and this may be of significance.

In the following three tests, other means of improving access of electrolyte to cathode were tried. In cell T23, thirty discs of separator paper were inserted at intervals in the cathode cup as it was being filled. Fifteen discs of blotter paper replaced the rayon paper in T24. In T28, three grams of paper pulp were blended with the cathode mixture. These tests were conducted at -63°C and are summarized below.

Test No.	Variable Kind	Variable Amount	Slaked	Hours to End Voltage
T-23	Rayon	30 discs	yes	46
T-24	Blotter	15 discs	yes	48
T-20 & 21	NONE		yes	53 ave.
T-28	Pulp	3 grams	no	62.5
T-18 & 19	NONE		no	56 ave.

Nothing was gained by the inclusion of either the rayon paper or the blotter paper, but the addition of paper pulp may be significant.

It should be noted that in T23, T24, and T28 straight silver wire cathode collectors were used in place of silver coils because of construction difficulties. It is indicated that this change had a negligable effect on cell performance.

T29 and T34 were four-day active stand tests in KSCN-ammonia electrolyte at -63° C. The former was a complete cell; the latter, a cathode only. In the case of T29, the anode was badly corroded and covered with scale. The cell did not function with this anode or with a new one. After the active stand of T34, and anode was placed around the cathode; at which time there was an immediate and violent evolution of gas; and the anode turned black. After the application of the cyclic load, the cell ran only ten hours at -63°C to an end voltage under maximum load of 1.5.

The cathode of cell T30 was saturated with a solution of 17 grams of NH_4SCN in 53 grams of liquid ammonia at -63°C for 18 hours. Then, a magnesium sheet anode was wrapped around it; and the cell was activated as usual. A life of 31.5 hours was obtained which is only about half of that demonstrated by cells T3, T4, T18, and T19 but substantially greater than T34 above.

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 below. All tests were run at -63°C under cyclic loads of 2.7 and 13.6Ω.

Test No.	Sulfur gms.	$HgSO_4 \cdot 2NH_3$ gms.	Carbon gms.	NH_4SCN gms.	Total Cathode gms.	Cathode Collector	Life in Hours to 1.5V	Life in Hours to 1.3V
T-26	44.1	19.3	49.6	22.0	135	C rod	50	*
T-27	44.1	19.3	49.6	22.0	135	C rod	44	56
T-31	39.2	34.2	44.1	19.5	137	C rod	47.5	55
T-32	39.2	34.2	44.1	19.5	137	Steel rod	3	45
T-33	10	133	57	0	200	Ag wire	73	78
T-4	0	119	51	0	170	Ag coil	63	70
T-18	0	147	63	0	210	Ag coil	59	—
T-19	0	147	63	0	210	Ag coil	53	—

*50 hours under cyclic load plus an additional 64 hours under a 13.6Ω load to 1.30 volts

A comparison of T27 and T31 shows that a substantial increase in $HgSO_4 \cdot 2NH_3$ in the presence of sulfur did not increase the cell life significantly. The cells which did not contain any NH_4SCN (T4, T18, and T19) performed better than any in the group T26 to T32.

However, cell T33, which contained only ten grams of sulfur and no NH_4SCN, out-performed all of the others with a life of 73 hours.

MAGNESIUM/META-DINITROBENZENE CELLS

Refer to Table II

The magnesium/meta-dinitrobenzene cells tested during the second quarter are summarized in Table II, starting on page 23.

Test Number 2396, which was one of the best flat plate cells run at -73°C, yielded about one Faraday per mol in 44 hours which is well below the required life.

Since then, a number of cells have been constructed in the spiral configuration shown in Figure 3, page 14. That is, the various components in sheet form were laid one upon the other and then rolled into a solid unit of cylindrical shape. These cells can be inserted into various containers such as the A-606, the A-622, or beakers. The prefix "H" indicates disposable cell containers.

Cell Number B2 ran well for 49.5 hours at -73°C to 1.70 volts, at which time one of the leads was broken.

Number B3, a full size version, ran at -73°C for 116 hours to 1.5 volts and for another 15 hours to 1.4 volts. The yields were 0.76 and 0.84 Faradays per mol of oxidant, respectively,

Numbers B4, B5, and B6 were similar to B3 except that they were tested at +20°C. The results were very poor down to an end voltage of 1.5. However, B4 and B6 were allowed to continue discharging to 1.3 volts. Since the voltage curves were quite flat, it required about 50 hours for the voltage to decrease from 1.5 to 1.3.

	Faradays per mold of Oxidant		
	To 1.5v	To 1.4v	To 1.3v
B4	0.15	0.25	0.46
B6	0.15	0.31	0.45

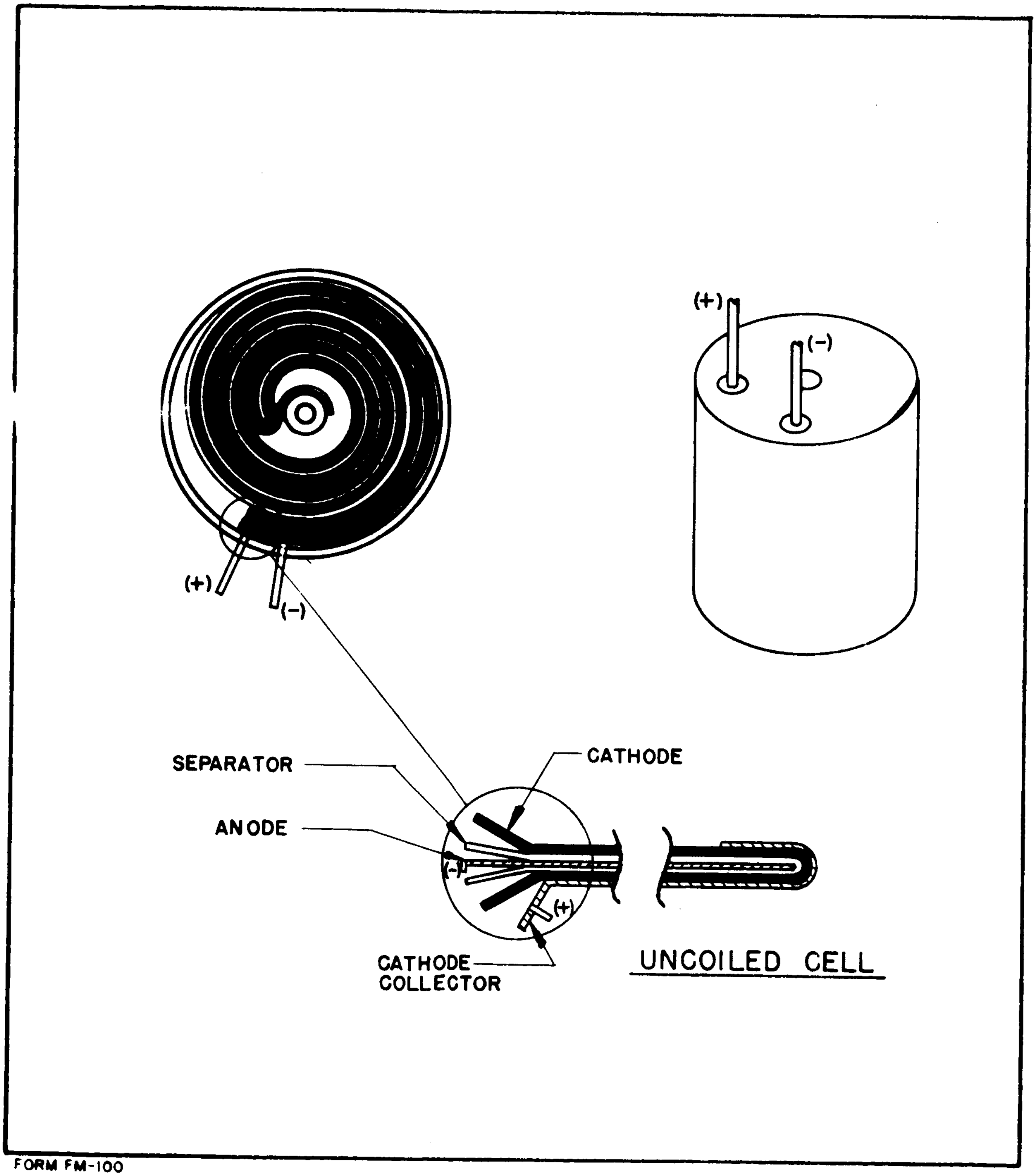
Cell Number B7 was built into an A-622 quick-coupler chamber and activated. Because of leakage of ammonia from the container, it was not discharged and was allowed to stand on open circuit for two weeks. After this, the cell was transferred from the chamber to a beaker, reactivated, and discharged under the required cyclic load at -63°C. It performed for 110 hours to 1.5 volts and for an additional 11 hours to 1.4 volts. It is interesting to note that this cell performed well at -63°C after a partially activated stand.

Cell B8 failed because of an electrolyte leak.

B9 was placed on active stand for seven days, after which an inspection revealed that the Mg anode had corroded completely.

B11 was on active stand for 8 days at -63°C, after which the cyclic load was applied. The peak closed circuit voltage was only 1.82 volts under the light load and 1.45 volts under the heavy load. One hundred two hours were required to discharge the cell to 1.67 volts under 13.6Ω and to 1.00 volt under 2.7Ω.

The results of test at -73°C on B10, as given in Table II, page 25, show an 88-hour life to 1.50 volts and a 107-hour life to 1.30 volts under heavy load. In addition, the cell was allowed to discharge another 15 hours to 1.15 volts, after which its temperature was increased slowly to ambient. During a ten-hour period, the voltage recovered to 1.90 at 2.7Ω. The discharge was continued for another 32 hours to an end voltage of 1.65 at 2.7Ω and 1.90 at 13.6Ω. It is probable that this cell could have run much longer.



SPIRAL CONFIGURATION

FIGURE 3

NOTES TO TABLES I AND II

ELECTROMOTIVE CELL TESTS

[1] T. T. = Pyrex Test Tube

[2] Loads shown as A/B represent cyclic loads of AΩ for 6 minutes and BΩ for 54 minutes

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

[4] After 23 hours, added KSCN

[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.

[a] 126 grams $HgSO_4$

[b] 106 grams $HgSO_4$

±anode lead sheared at 49.5 hours on cell B-2.

TABLE I

ELECTROCHEMICAL

	Magnesium/	
Test Number:	T2	T3
Major Variables:		open top
Reference (Page No.):	6	6
Vehicle[1]:	TT	A-624
Configuration - Bobbin:		
Net Cell Volume (in.^{3}):	30	34
Load, Ω[2]:	3/15	3/15
Temperature °C:	-63°	-63°
Hrs to End Voltage (Cumulative) 1.5/1.3V.** :	44	70/72
Anode (AZ31B Mg Sheet), Area (cm^2):	280	354
Cathode, gm. $HgSO_4 \cdot 2NH_3$:	119	119
Cathode, gm. Sulfur:		
Cathode, gm. Carbon:		
Cathode Collector:	Ag Coil	Ag Coil
Cathode Collector Area (cm^2):	60	60
Separator Thickness[3], inches:	0.028	0.028
$HgSO_4 \cdot NH_3$ Coordination No.[5]:	2	2
Initial Open Circuit Voltage:	2.30	2.30
Initial Closed Circuit Voltage*:	2.20	2.25
Peak Closed Circuit Voltage*:	2.22	2.25
End Voltage (under Max. Load)	2.00	1.50
End Voltage (under Light Load):	2.21	2.10
Initial Anode C.D. (mA/cm^2)*;	0.52	0.42
Initial Cathode Collector C.D. (mA/cm^2)*;	2.44	2.50
Observed Coulombs/gram oxidants;	281	398
Observed F/mol Cathode Reactant;	0.96	1.37
Watt Hours/Inch^3 of Net Cell;	0.65	0.73

CELL TESTS

Mercuric Sulfate Couples

T4	T5	T6	T8	T9
	closed top	cathode dia.	slaked cathode dia.	slaked cathode dia.
7	7	7	7	7
TT	A-624	A-624	A-624	A-624
30	34	34	34	34
3/15	3/15	3/15	3/15	2.67/15
-63°	-63°	-73°	+20°	-73°
63/70	57/61	57/59	16/18	33/34
354	354	248	337	373
119	119	140	161	147
Ag Coil	Ag Coil	Ag Coil	Ag Coil	Ag Coil
60	60	60	60	60
0.028	0.028	0.028	0.028	0.028
2	2	2	4	4
2.45		2.40	2.38	2.36
2.25	2.20	2.20	2.32	2.30
2.25	2.25	2.20	2.32	2.30
1.50	1.50	1.50	1.50	1.50
2.20	2.05	2.10	2.17	2.08
0.42	0.42	0.59	0.46	0.41
2.50	2.44	2.44	2.58	2.56
359	323	271	68	154
1.25	1.11	0.93	0.24	0.53
0.74	0.59	0.57	0.17	0.35

TABLE I

ELECTROMOTIVE

Magnesium/Mercuric

Test Number:	T-10	T-11		T-12	T-13
Major Variables:	Center Anode	geometry & temperature			
Reference (Page No.):	8	8		8	8
Vehicle[1]:	T. T.	A-624		A-624	T. T
Configuration - Bobbin:	Inverse	Conventional			
Net Cell Volume (in.^{3}):	30	34		34	30
Load, Ω^2:	3/15	3/15		3/15	3/15
Temperature °C:	-63°	-73°	+20°	+24°	-63°
Hrs to End Voltage (Cumulative) 1.5/1.3V. ** :	0	10.5	56.5	17	32.5
Anode (AZ31B Mg Sheet), Area (cm^2):		226		242	271
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
$HgSO_4 \cdot NH_3$ Coordination No.[5]:	2	4		4	4
Initial Open Circuit Voltage:		2.35		2.35	2.30
Initial Closed Circuit Voltage*:		2.30	2.35	2.30	2.15
Peak Closed Circuit Voltage*:		2.30	2.35	2.32	2.23
End Voltage (under Max. Load):		1.50	1.50	1.50	1.50
End Voltage (under Light Load):		1.75	1.78	1.80	2.00
Initial Anode C. D. (mA/cm^2)*:		0.68	--	0.64	0.55
Initial Cathode Collector C. D. (mA/cm^2)*:		2.56	--	2.58	2.48
Observed Coulombs/gram oxidants:		41	224	67	125
Observed F/mol Cathode Reactant:		0.14	0.77	0.23	0.43
Watt Hours/Inch^3 of Net Cell:		0.11	0.61	0.18	0.38
	failed to activate			leaked s.circuit	

Continued

CELL TESTS

Sulfate Couples

T-14	T-15	T-16		T-17	T-18		T-19	
geometry & temp.		temperature		temperature	ohmic load temperature		ohmic load temperature	
anode to cathode spacing $\sim\frac{1}{4}$"								
8	8	9		8	9		9	
A-624	T. T.	A-624		A-624	T. T.		T. T.	
Conventional								
68	30	37		37	30		30	
3/15	3/15	3/15		3/15	2.7/13.6		2.7/13.6	
-73°	-63°	-73°	+15°	20°	-63°	~-30°	-63°	~-30°
41	47	47	83	16	59	71	53	64
725	371	465		465	430		362	
175	175	147		164	147		147	
0	0	0		0	0		0	
75	75	63		71	63		63	
Ag Coil								
60	60	60		60	60		60	
0.028	0.016	0.016		0.028	0.028		0.028	
4	2	2		4	2		2	
2.35	2.35	2.35	--	2.44	2.22	--	2.40	--
2.30	2.25	2.25	--	2.30	2.05	--	2.10	--
2.35	2.28	2.25	2.27	2.32	2.25	2.30	2.30	2.37
1.50	1.50	1.50	1.50	1.97	1.50	1.50	1.50	1.88
2.00	1.90	1.70	1.75	2.25	1.88	1.82	1.95	2.29
0.22	0.41	0.32	--	0.33	0.38	--	0.47	--
2.61	2.54	2.50	--	2.58	2.76	--	2.82	--
163	183	216	383	76	300	362	272	335
0.56	0.63	0.74	1.31	0.26	1.03	1.24	0.93	1.15
0.23	0.56	0.45	0.80	0.20	0.77	0.93	0.70	0.88
	anode connection broken			"Vented"				

TABLE I Continued

ELECTROMOTIVE CELL TESTS

Magnesium/Mercuric Sulfate Couples

Test Number:	T-20	T-21	T-22	T-23	T-24
Major Variables:	Slaking	Slaking	Acetylene Black 5 gms.	Webril 30 discs	Blotter 15 discs
Reference (Page No.):	9	9	9	9	9
Vehicle[1]:	T. T.	T. T.	T. T.	T. T	
Configuration - Bobbin:	Conventional				
Net Cell Volume (in.^{3}):	30	30	30	30	30
Load, Ω[2]:	2.7/13.6				
Temperature °C:	-63°	-63°	-63°	-63°	-63°
Hrs to End Voltage (Cumulative) 1.5/1.3V.** :	47	59	55	46	48
Anode (AZ31B Mg Sheet), Area (cm^2):	354	354	354	354	354
Cathode, gm. $HgSO_4 \cdot 2NH_3$:	147	140	122	140	140
Cathode, gm. Sulfur:	0	0	0	0	0
Cathode, gm. Carbon:	63	60	52	60	60
Cathode Collector:	Ag Coil			24"	12"
Cathode Collector Area (cm^2):	60	60	60	16	8
Separator Thickness[3], inches:	0.028	0.028	0.028	0.028	0.028
$HgSO_4 \cdot NH_3$ Coordination No.[5]:	4	4	2	4	4
Initial Open Circuit Voltage:	2.35	2.28	2.35	2.25	2.32
Initial Closed Circuit Voltage*:	2.20	2.05	2.22	2.20	2.25
Peak Closed Circuit Voltage*:	2.30	2.21	2.25	2.22	2.30
End Voltage (under Max. Load):	1.50	1.50	1.50	1.50	1.50
End Voltage (under Light Load):	1.95	1.98	2.00	2.10	1.96
Initial Anode C. D. (mA/cm^2)*:	0.48	0.46	0.47	0.46	0.48
Initial Cathode Collector C. D. (mA/cm^2)*:	2.82	2.71	2.76	10.2	21.2
Observed Coulombs/gram oxidants:	242	311	337	220	258
Observed F/mol Cathode Reactant:	0.83	1.07	1.15	0.76	0.89
Watt Hours/Inch^3 of Net Cell:	0.62	0.75	0.72	0.58	0.64

TABLE I

ELECTROMOTIVE

Magnesium/Mercuric Sulfate and

Test Number:	T-25	T-26		
Major Variables:	Acetylene Black 9 gms.	S: $HgSO_4 \cdot 2NH_3$ with NH_4SCN		
Reference (Page No.):	9	11		
Vehicle[1]:	T. T.	T. T		
Configuration - Bobbin:	Conventional			
Net Cell Volume (in.^{3}):	30	30		
Load, Ω[2]:	2.7/13.6	2.7/13.6	∞/13.6	∞/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 rod - 3/16" x 11"		
Cathode Collector Area (cm^2):	60	38		
Separator Thickness[3], inches:	0.028	0.028		
$HgSO_4 \cdot NH_3$ 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	580	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

Continued

CELL TESTS

Magnesium/Mercuric Sulfate - Sulfur Couples

T-27	T-28	T-29	T-30	T-31	T-32		T-33	T-34
	Paper Pulp 3 gms.	Active Stand w/anode	cathode soaked in NH_4SCN/NH_3	S:$HgSO_4 \cdot 2NH_3$ with NH_4-SCN	steel cathode collector		$HgSO_4 \cdot 2NH_3$ / S	Active Stand wo/anode
11	9	10	10	11	11		11	10
T. T.	T. T.	T. T.	T. T.	T. T.	T. T.		T. T.	T. T.
Conventional								
30	30	30	30	30	30		30	30
2.7/13.6								
-63°	-63°	-63°	-63°	-63°	-63°		-63°	-63°
44/56	62.5/63.5	0	31.5/32.5	47.5/55	3/45	59	73/78	10/10.5
354	372	304	347	352	337		337	368
19.3	140	119	119	34.2	34.2		133	140
44.1	0	0	0	39.2	39.2		10	0
49.6	60	51	51	44.1	44.1		57	60
	Ag wire	Ag wire	Ag wire	C rod	Steel rod		Ag wire	Ag wire
38	13.4	13.4	13.4	38	31		14.7	16.7
0.028	0.028	0.028	0.028	0.028	0.028		0.028	0.028
2	2	2	2	2	2		2	2
2.45	2.36	0.93	2.25	2.52	2.57	--	2.35	2.24
2.20	2.00		2.12	2.00	2.06	--	2.10	2.15
2.20	2.30		2.20	2.23	2.13	2.01	2.25	2.24
1.50	1.50		1.50	1.50	1.50	1.25	1.50	1.50
1.95	1.75		1.95	1.95	2.01	1.66	1.75	1.87
0.46	0.45		0.47	0.47	0.47	--	0.49	0.45
4.26	12.6		12.1	4.32	5.06	--	11.25	9.90
510	337		194	481	30	524	382	53
0.23	1.16		0.67	0.28	0.02	0.30	0.79	0.15
0.55	0.83		0.40	0.61	0.04	0.59	0.95	0.13

TABLE II

ELECTROCHEMICAL

Magnesium/

Test Number:	B2	B3	
Major Variables:	Configuration	Size	
Reference (Page No.):	12	12	
Vehicle^1:	A-606	H-622	
Configuration -	spiral wrap	spiral wrap	
Net Cell Volume (in.^{3}):	6	33	
Load, Ω^2:	25/120	3/15	
Temperature °C:	-73°	-73°	
Hrs to End Voltage (Cumulative) **:	49.5	116	131
Anode (Mg Sheet), Area (cm^2):	77	671	
Cathode, gm m-DNB:	16	172	
Cathode, gm. Carbon:	16	172	
Cathode Collector:	Ag Sheet	Ag Sheet	
Cathode Collector Area (cm^2):	77	710	
Separator Thickness^3, inches:	0.012	0.012	
Initial Open Circuit Voltage:			
Initial Closed Circuit Voltage*:	2.34	2.10	
Peak Closed Circuit Voltage*:	2.34	2.10	
End Voltage (under Max. Load):	1.70$^{\pm}$	1.50	1.40
End Voltage (under Light Load):	1.93	1.82	1.67
Initial Anode C.D. (mA/cm^2)*:	0.25	0.21	
Initial Cathode Collector C.D. (mA/cm^2)*:	0.25	0.20	
Observed Coulombs/gram oxidants:	282	437	480
Observed F/mol Cathode Reactant:	0.49	0.76	0.84
Watt Hours/Inch^3 of Net Cell:	0.42	1.14	1.22

CELL TESTS

meta-Dinitrobenzene Couples

B4			B5	B6			B7		2396	B8
Temperature			Replicate	Replicate			Vehicle		Configuration	Vehicle
	12		12		12		13		12	13
	H-622		H-622		H-622		1L beaker		A-622	H-606
	Spiral Wrap		Sp.Wrap		Spiral Wrap		Spiral Wrap		Flat Plate	Sp.Wrap
	33		33		33		33		6.9	6
	3/15		3/15		3/15		3/15		10/50	25/120
	+20°		+20°		+20°		-63°		-73°	+20°
21	37	70+	5	22	49	73	110	121	44.4	0
	671		671		671		851		135	77
	172		172		172		206		16.2	16
	172		172		172		206		16.2	16
	Ag Sheet		AgSheet		Ag Sheet		S.S.[6]		Ag Exmet	Ag Sheet
	710		710		710		851		135	77
	0.012		0.012		0.012		0.012		0.007[7]	0.012
	2.40								2.25	
	2.28		2.12		2.12		2.05		2.25	
	2.28		2.12		2.12		2.05		2.25	
1.50	1.40	1.30	1.50	1.50	1.40	1.30	1.50	1.40	1.50	
1.90	1.87	1.84		1.83	1.77	1.70	1.80	1.73		
	0.23		0.21		0.21		0.16		0.33	
	0.21		0.20		0.20		0.16		0.33	
83	143	262	19	83	180	261	342	366	558	
0.15	0.25	0.46	0.03	0.15	0.31	0.45	0.60	0.64	0.97	
0.23	0.38	0.68	0.05	0.22	0.46	0.65	1.06	1.10	0.68	

TABLE II Continued

ELECTROCHEMICAL CELL TESTS

Magnesium/meta-Dinitrobenzene Couples

Test Number:	B-9	B-10		B-11
Major Variables:	active stand 7 days at 20°C	Temperature		active stand 8 days at -63°C
Reference (Page No.):	13	13		13
Vehicle[1]:	H-606	H-622		1L beaker
Configuration -	Sp. Wrap	Spiral Wrap		Sp. Wrap
Net Cell Volume (in.^{3}):	6	33		33
Load, Ω[2]:	25/120	2.7/13.6		2.7/13.6
Temperature °C:	+20°	-73°		-63°
Hrs to End Voltage (Cumulative) **:	0	88	107	102
Anode (Mg Sheet), Area (cm^2):	77	671		671
Cathode, gm. m-DNB:	16	172		172
Cathode, gm. Carbon:	16	172		172
Cathode Collector:	Ag Sheet	Ag Sheet		Ag Sheet
Cathode Collector Area (cm^2):	77	710		710
Separator Thickness[3], inches:	0.012	0.012		0.012
Initial Open Circuit Voltage:		2.25		
Initial Closed Circuit Voltage*:		2.10		1.60
Peak Closed Circuit Voltage*:		2.10	1.80	1.82
End Voltage (under Max. Load):		1.50	1.30	1.00
End Voltage (under Light Load):		1.80	1.65	1.67
Initial Anode C.D. (mA/cm^2)*:		0.23	--	0.20
Initial Cathode Collector C.D. (mA/cm^2)*:		0.22	--	0.19
Observed Coulombs/gram oxidants:		366	420	332
Observed F/mol Cathode Reactant:		0.64	0.73	0.58
Watt Hours/Inch^3 of Net Cell:		0.95	1.03	0.68

EFFECTS OF CHROMATE COATING ON MAGNESIUM ANODES

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

(1) To determine the passivation effect of chromate films on the corrosion rate of pure magnesium anodes

(2) 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 $Mg/25\% KSCN-NH_3/HgSO_4 \cdot 2NH_3-C: Ag$ 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 capactiy. The exact relationship and interplay of the anode (Mg) and cathode ($HgSO_4$) 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 $KSCN-NH_3$ 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 [$Mg(NH_3)_6{}^{++}$] and those in which it becomes part of a solid phase [$Mg(NH_2)_2$]. 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) or magnesium anodes (2" width, 4" length, .008" thickness) were degreased in acetone and treated with a special chromate solution (CrO_3 - 12 grams/liter; $CaSO_4 \cdot 2H_2O$ - 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 opened-circuit potentials were determined in 25 per cent KSCN - NH_3 solution. The cells were placed in propylene bottles, covered with 25 per cent KSCN-NH_3 solution, and allowed to stand for twenty-one days. Next, the cells were removed and placed in freshly prepared 25 per cent KSCN-NH_3 solutions. Negligable opened-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 under- lying 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 per cent 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 $KSCN-NH_3$ 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 (H_2).

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 per cent Cu, 0.001 per cent Ni, and 0.20 per cent other impurities.

FUTURE WORK

A mercuric sulfate/sulfur bobbin cell was tested at the end of the second quarter. This cell exceeded the 72-hour requirement at -63°C. Additional experiments are planned in which the ratio of sulfur to mercuric sulfate will be varied in order to determine an optimum. Various coordination (NH_3) levels of mercuric sulfate will be evaluated by activating cells at low temperature. Additions of sulfur will be made to coordinated mercuric sulfate. The effects of cathode and anode geometry will be further examined. $HgSO_4$ and sulfur mixtures will be treated with $KSCN/NH_3$ electrolyte, rather than the formerly established procedure of ammoniating the $HgSO_4$ alone.

Bobbin cells comprising meta-dinitrobenzene: carbon and sulfur: carbon will also be tested at -63°C.

Experiments are planned in which pure magnesium and other alloys such as AZ-10 and AZ-21 will be employed. We also contemplate coating the side away from the cathode to prevent corrosion in this area. An analysis of the gas generated during discharge at room temperature will be made. The effects of re-using spent electrolyte will be studied.

Arrangements are being made to coat magnesium anodes with lead in various thicknesses by means of vapor deposition under vacuum.

The wet stand capacity of meta-dinitrobenzene/carbon bobbin cathodes in KSCN/ammonia electrolyte will be examined.

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