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EVALUATION OF INORGANIC/ORGANIC SEPARATORS

by C. Philip Donnel III

YARDNEY ELECTRIC DIVISION YARDNEY ELECTRIC CORPORATION

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

NASA Lewis Research Center

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SUMMARY

Thirty-six (36) experimental 40AH Sealed Silver-Zinc Cells were constructed during Phase I of this two (2) phase program. These cells were divided into six (6) groups of six (6) cells each. Each group of six (6) cells was evenly divided into two batches of three (3) cells each. Groups 1 through 4 each featured a different inorganic filler material in the slurry used to coat the separator substrate. Groups 5 and 6 featured an alternate method of separator bag construction. With the exception of the various separator materials, the parts and processes used to produce these thirty-six (36) cells were the same as those used to make the HS40-7 cell. The two (2) batches of cells in each cell group differed only in the lots of solutions and other separator slurry components used. Each cell was given two formation charge/discharge cycles prior to being shipped to NASA Lewis Research Center.

Phase II of the program consisted of constructing another thirty-six (36) 40AH experimental cells in six (6) groups of six (6) cells each. Each group was distinguished by the type of precoated separator material used to fabricate separator bags. A new method of separator bag construction was used in this phase of the program. These cells were given two (2) formation cycles and shipped to NASA Lewis Research Center.

INTRODUCTION

NASA Lewis Research Center has funded programs, over the past several years, which were aimed at the development of the Sealed Silver-Zinc Cell and the refinement of its components.

The initial program conducted at McDonnell-Douglas Corporation's Astropower Laboratory fostered the development of the HS40-7 cell which utilized the semi-flexible inorganic separator 3420-25 FMA.

A later program involved the establishment of a facility at Yardney Electric Division equipped to produce the sealed silverzinc cell. Subsequent programs carried out in this facility have dealt primarily with variations in electrode components and the testing of cells containing these variations.

NASA Lewis Research Center has continued to fund programs in the battery and related fields to investigate inorganic separator materials. It has also conducated an in-house program which has produced a series of inorganic/organic (I/O) separators and separator components for both silver-zinc and nickel-zinc applications. In order to evaluate the performance of the experimental separators for silver-zinc cell applications, NASA Lewis Research Center funded a program to incorporate the separator components and separator materials in a cell of established configuration and familiar characteristics, namely the HS40-7 Sealed Silver-Zinc Cell.

It is the purpose of this report to describe the incorporation of these experimental separator components and materials in a standard configuration cell fabricated in NASA's facility at Yardney Electric Division under Contract NAS3-18530 funded by NASA Lewis Research Center.

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PHASE I - CONSTRUCT THIRTY-SIX (36) EXPERIMENTAL 40AH CELLS

1. Objective of Phase

The objective of this program phase was to introduce experimental inorganic filler materials and fabrication techniques into the standard HS40-7 sealed silver-zinc battery cell configuration for the purpose of evaluating these variables. Six (6) groups of six (6) cells were required, each group containing one variation from the standard configuration.

2. Task I - Electrode Fabrication

2.1 Silver positive electrodes were fabricated for the thirty-six (36) cells of Phase I. These electrodes were made by the mold-press method described in the procedures BFDO #0005 through BFDO #0013 furnished by NASA Lewis Research Center. Each electrode conformed to Drawing No. 1D12571.

2.2 Zinc-oxide negative electrodes were fabricated for the Phase I cells in accordance with the procedures BFD(/ #1009 through BFDO #1017 and BFDO #1001 furnished by NASA Lewis Research Center. Each electrode conformed to Drawing No. 1D12560.

3. Task II - Separator Slurry Preparation

The separator slurries prepared in this task were formulated in accordance with the applicable portions of the proprietary documentation furnished by NASA Lewis Research Center. The separator slurry filler material variations which characterized the cells of Groups 1 through 4 were introduced at this point. The four (4) filler materials were designated 3420-25, CAZR, DPCASR, and MGTI. The 3420-25 filler is used in the standard formulation. The other fillers were used in amounts equal to the 3420-25 filler on a volume percent basis. Due to the different specific gravities of the three non-standard inorganic fillers as compared to the 3420-25 powder, and because the slurry formulation is based on additions of filler by weight, the non-standard slurry formulations required different weights of filler. The following gives the desired reductions in filler weights as a fraction of the amounts of 3420-25 powder used in the standard formulation.

CELL GROUP	FILLER	SPECIFIC GRAVITY	FRACTION
1	3420-25 (Standard)	5.40	1.00
2	CAZR	4.38	0.81
3	DPCAZR	4.38	0.81
4	MGT1	3.36	0.62

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Sufficient slurry of each type was compounded to coat the separator bags required for the first three (3) cells of each group. The cells made using these separators were designated Batch 1. With the exception of the inorganic fillers all other materials used in Batch 1 slurries were the same. The processing of the slurries from formulation to use were identical.

4. Task III - Separator Bag Fabrication

4.1 Cell Groups 1 through 4

4.1.1 Sufficient separator substrate material was prepared and processed as a lot to meet the requirements for the Batch 1 cells of Groups 1 through 4. This material was cut to size and made into sealed separator bags which conformed to Drawing No. 1D12568.

4.1.2 After inserting electrodes into the separator bags each bagged electrode was dipped in the appropriate slurry to cast a coating on the bag. This operation was done in accordance with Procedure BFDO #2036 furnished by NASA Lewis Research Center.

4.2 Cell Groups 5 and 6

4.2.1 The separator used in Group 5 cells was a 3420-25 FMA material slurry coated by a continuous process. Pieces of this material were cut from the material furnished by NASA Lewis Research Center.

4.2.2 The separator used in Group 6 cells was a 3420-25 FMA material with the slurry coatings applied by the dipping process. An oversized sealed separator bag was made from substrate material and dipped in slurry containing 3420-25 filler. The sealed areas of the dipped bag were cut away leaving two (2) pieces of dip coated separator material.

4.2.3 Separator bag fabrication for Group 5 and 6 cells was accomplished by gluing cut pieces of the materials to form a bag around the electrode using an epoxy cement.

4.2.4 Three (3) cells worth of bagged positive and negative electrodes were made for Group 5 and Group 6 cells and were designated Batch #1.

5. Task IV - Batch Variations

5.1 The work accomplished in Task II and Task III was repeated to produce a second three (3) cell batch of bagged positive and negative electrodes of each group type. Since the eventual

aim of this task was to evaluate the repeatability of slurry formation and separator bag fabrication processes, the various slurries for Groups 1 through 4 were formulated with new and different lots of solutions and materials with the exception of inorganic fillers. The glued bags for Groups 5 and 6 were made of different batches of slurry coated 3420-25 FMA separator material. The parts produced in this task were designated Batch #2.

6. Task V - Cell Assembly and Formation

6.1 Cell Groups 1 through 4

6.1.1 Cells were assembled for these groups using the standard complement of six (6) bagged positive electrodes and five (5) bagged negative electrodes. Each cell was vacuum filled with 110 ml of the 45% solution of potassium hydroxide, allowed to soak for a minimum of 24 hours, and given the first of two (2) formation cycles. The cell formation charge, discharge and drain parameters were as follows:

Charge: Constant current of 1.5A to an end voltage of 1.98-2.00 volts or 45AH maximum input, whichever occurred first.

Discharge: Constant current of 6.0A to an end voltage of 1.00 volt.

Drain: Constant current of 2.0A to an end voltage of 1.00 volt.

The results of the first formation cycle are given in Tables I and II.

The cells were then sealed in a pressure vessel and subjected to an elevated temperature heat treatment for 24 hours at 100°C. Following the heat treatment the cells were sealed and given a second formation cycle using the same regime followed in the first formation cycle. The results of the second formation cycle are given in Tables III and IV.

6.2 Cells Groups 5 and 6

5.2.1 The cells assembled for Groups 5 and 6 contained five (5) bagged positive electrodes and five (5) bagged negative electrodes. The deletion of one (1) bagged positive electrode from the standard cell complement was necessitated by a thickness buildup in the bag seal areas. With the exception of formation cycle parameters, the processing of Group 5 and 6 cells was identical to that followed for Groups 1 through 4. The formation cycle charge, discharge and drain parameters used on the cells of Groups 5 and 6 were as follows: Charge: Constant current of 1.25A to an end voltage of 1.98-2.00 volts or 37.5 AH maximum, whichever occurred first.

Discharge: Constant current of 5.0A to 1.00 volt.

Drain: Constant current of 1.60A to 1.00 volt.

The results of the first formation cycle are given in Tables I and II. The results of the second formation cycle are given in Tables III and IV.

6.2.2 During the preparation of the cells for shipment, it was noted that the cells of Group 5 had a marked tendency to expand when the steel restraining plates were loosened indicating the presence of considerable internal pressure. One of the cells ruptured at the case to cover seal area. At the direction of NASA Project Manager, the Group 5 cells were not shipped to NASA Lewis Research. A thorough review of all fabrication, processing and formation data records gave no indication as to the cause of this high internal pressure condition in Group 5 cells.

6.2.3 The six (6) cells of Groups 1 through 4 and Group 6 were shipped to NASA Lewis Research Center.

7. Task VI - Reliability and Quality Assurance

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7.1 A Reliability and Quality Assurance Plan was developed by Yardney to cover the fabrication of the HS40-7 sealed silverzinc cell under Contract NAS3-16805. The applicable portions of this plan, entitled RQAPP #400, were utilized throughout the manufacture and processing of the cells produced during Fhase I of this program.

TABLE I

FORMATION CYCLE DATA EXPERIMENTAL 40AH CELLS PHASE I BATCH NO. 1 FORMATION CYCLE NO. 1

CELL	CELL	CHARGE	DISCHARGE	DRAIN	TOTAL
GROUP	S/N	INPUT	OUTPUT	OUTPUT	OUTPUT
NO.	NO.	AH	AH	AH	AH
. 1 .	001	44.25	37.37	0.56	37.93
	002	43.95	37.23	0.70	37.93
	003	43.27	38.52	0.69	37.21
-2	001	42.22	35.47	1.02	36.49
	002	42.07	35.58	0.76	36.34
	003	42.67	36.03	0.88	36.91
- 3	001	40.20	34.15	0.71	34.86
	002	39.45	33.53	0.90	34.43
	003	39.30	33.57	0.59	34.16
	001	39.45	34.09	0.74	34.83
	002	39.30	34.00	0.79	34.79
	003	40.20	33.90	1.17	35.07
* 5	001	37.5	30.91	0.55	31.46
	002	37.5	30.25	0.76	31.01
	003	36.87	32.38	0.66	33.04
* 6	001	28.12	24.71	0.66	25.37
	002	28.12	24.73	0.63	25.36
	003	28.12	24.69	0.67	25.36

*See Text paragraph 6.2.1 for charge, discharge and drain parameters used on this group of cells.

TABLE II

FORMATION CYCLE DATA EXPERIMENTAL 40AH CELLS PHASE I, BATCH NO. 2 FORMATION CYCLE NO. 1

CELL	CELL	CHARGE	DISCHARGE	DRAIN	TOTAL
GROUP	S/N	INPUT	OUTPUT	OUTPUT	OUTPUT
NO.	NO.	AH	АН	AH	AH
1.	004	40.05	34.56	1.07	35.63
	005	40.05	34.49	1.01	35.50
	006	40.05	34.37	1.16	35.53
2 .	004	40.50	34.86	0.89	35.75
	005	40.50	35.06	0.96	36.02
	006	40.50	34.73	1.28	36.01
3	004	40.80	34.92	1.07	35.99
	005	40.80	34.63	1.50	36.13
	006	40.80	35.11	0.99	36.10
4	004	40.80	34.83	1.15	35.98
	005	40.80	34.81	1.13	35.94
	006	41.25	35.19	1.23	36.42
* -5 *	004 005 006	37.5 37.5 37.5	31.26 31.03 31.07	0.55 0.77 0.72	31.81 31.80 31.79
*	004	28.12	24.71	0.58	25.29
	005	28.12	24.73	0.63	25.36
	006	28.12	24.73	0.59	25.32

*See Text paragraph 6.2.1 for charge, discharge and drain parameters used on this group of cells.

TABLE III

FORMATION CYCLE DATA EXPERIMENTAL 40AH CELLS PHASE I, BATCH NO. 1 FORMATION CYCLE NO. 2

CELL	CELL	CHARGE	DISCH'G	PLATEAU	DRAIN	TOTAL
GROUP	S/N	INPUT	OUTPUT	VOLTAGE	OUTPUT	OUTPUT
NO.	NO.	AH	AH	v	AH	AH
1	001	43.05	39.83	1.473	0.63	40.46
	002	43.50	40.28	1.464	0.63	40.91
	003	45.00	42.23	1.482	0.54	42.77
2	001	36.00	32.74	1.458	1.03	33.77
	002	33.75	30.46	1.457	1.09	31.55
	003	36.00	32.67	1.452	1.34	34.01
3	001	37.12	34.51	1.466	1.23	35.74
	002	40.35	37.92	1.474	0.81	38.73
	003	37.12	34.72	1.469	1.04	35.76
4	001	45.00	43.25	1.470	0.75	44.00
	002	45.00	43.43	1.465	0.72	44.15
	003	45.00	43.42	1.475	0.81	44.23
* 5	001 002 003	36.25 36.25 35.0	33.03 32.76 31.99	1.458 1.460 1.470	0.63 0.69 0.45	33.66 33.36 32.44
* 6	001 002 003	36.87 37.5 37.5	34.33 35.46 35.48	1.475 1.482 1.482	0.39 0.41 0.41	34.72 35.87 35.89

*See Text paragraph 6.2.1 for charge, discharge and drain parameters used on this group of cells.

TABLE IV

FORMATION CYCLE DATA EXPERIMENTAL 40AH CELLS PHASE I, BATCH NO. 2 FORMATION CYCLE NO. 2

CELL GROUP	CELL S/N	CHARGE INPUT	DISCH'G OUTPUT	PLATEAU VOLTAGE	DRAIN OUTPUT	TOTAL OUTPUT
NO.	NO.	AH	AH ····	on V era i	AH	AH ···
1.	004 005 006	45.00 45.00 45.00	41.91 41.96 41.94	1.483 1.487 1.490	0.65 0.64 0.59	-42.56 42.60 42.53
. 2	004 005 006	45.00 45.00 45.00	41.53 41.68 41.82	1.480 1.481 1.479	0.86 0.75 0.74	42.39 42.43 42.61
. 3 .	004 005 006	45.00 45.00 45.00	41.65 41.67 41.73	1.478 1.479 1.479	0.82 0.89 0.88	42.47 42.56 42.56
4	004 005 006	45.00 45.00 45.00	42.10 42.28 42.40	1.485 1.483 1.472	0.77 0.56 0.69	42.87 42.84 43.09
* 5	004 005 006	37.5 37.5 37.5	33.45 33.44 33.60	1.423 1.460 1.456	0.79 0.68 0.75	34.24 34.12 34.35
* - 6 *	004 005 006	37.5 37.5 37.5	35.39 35.31 35.47	1.482 1.476 1.472	0.39 0.49 0.60	35.78 35.80 36.07
				·		· · · ·

*See Text paragraph 6.2.1 for charge, discharge and drain parameters used on this group of cells. PHASE II - CONSTRUCT THIRTY-SIX (36) EXPERIMENTAL 40AH CILLS

1. Objective of Phase

The objective of program Phase II was to introduce experimental inorganic/organic (I/O) separator materials into the standard HS40-7 sealed silver-zinc battery cell configuration for the purpose of separator evaluation. Six (6) groups of six (6) cells were required, each group containing a different separator material.

2. Task VII - Electrode Fabrication

2.1 Silver positive electrodes were fabricated in accordance with the drawings, specifications and procedures supplied by NASA Lewis Research Center. These electrodes were identical to those fabricated in Task I of the program.

2.2 The zinc-oxide negative electrodes fabricated for this program phase differed from those used in Phase I cells in that the absorber mat used was Yardney Type YIFL-II. This was necessitated by the fact that the Government furnished absorber mat normally used was no longer available. All other aspects of negative electrode fabrication were in accordance with drawings, specifications and procedures supplied by NASA Lewis Research Center.

3. Task VIII - Separator Preparation

The separator materials used in this program phase were supplied by NASA Lewis Research Center. Six (6) different materials were supplied and were designated to be used as follows:

CELL GROUP	SEPARATOR MATERIAL					
NO.	DESIGNATION					
1	3420-25 (Standard)					
2	X-10					
3	X-39					
4	X-47W					
5	X-104					
6	X-107					

The Government also furnished a supply of two-faced tape made with a closed cell polyethylene foam. This tape was designed to be used to seal the edges of electrode bags made with inorganic/ organic separator materials.

4. Task IX - Separator Bag Fabrication

4.1 Pieces of the various types of separator materials were cut to size. The substrate side of each piece of separator wes treated with a solution of PPO in chloroform so as to reinforce the edges of each piece for a width of approximately 0.6 cm.

4.2 The separator bags were fabricated by positioning two pieces of the separator material, slurry coated side outboard, and sealing the sides and bottom of the bag with the polyethylene tape. After curing the tape adhesive at an elevated temperature, any excess tape was trimmed away flush with the edge of the separator pieces.

5. Task X - Cell Assembly

5.1 Each of the cells assembled during Phase II of the program contained six (6) bagged positive and five (5) bagged negative electrodes. Certain of the separator types were thinner than others and necessitated shimming to achieve a uniform fit of cell stack to cell case. This shimming was done with layers of the Teflon film normally used in the cells as an assembly strip.

5.2 The balance of the assembly operations were accomplished by the methods normally used to assemble the HS40-7 cell.

6. Task XI - Cell Formation

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6.1 Each cell was vacuum filled with 110 ml of 45% solution of potassium hydroxide. Following a room temperature soaking period of 24 hours minimum, each cell was given the first of two (2) formation cycles using the constant current rates and voltage limits listed below.

Charge: 1.50 amperes to an end voltage, while charging, of 2.0 volts or a maximum input of 45 ampere-hours, whichever occurred first.

Discharge: 6.0 amperes to 1.00 volt.

Drain: 2.0 amperes to 1.00 volt.

The results of this first formation cycle are given in Table V.

6.2 Following the normal heat treatment and sealing of the cells, each cell was to be given a second formation cycle similar to the first. Group 2 cells were the first cells to complete the

second formation cycle. Based on the results obtained with Group 2 cells, direction was given by the NASA Project Manager to use lower charging current rates, as necessary, on the balance of the cell groups to realize an input of at least 40 ampere hours. The desired input was accomplished by charging the remaining cells at 1.50A, 1.0A and 0.50A to an end voltage of 1.99-2.02 volts or until the total input reached a minimum of 40 or a maximum of 45 ampere-hours. Each cell was then discharged at 6.0A to 1.00 volt and drained at 2.0A to 1.00 volt.

6.3 The results of the formation cycle No. 2 charge, discharge and drain of each cell of the six cell groups are given in Table V. The plateau voltage noted was the cell voltage at the 3 hour (18 ampere-hour output) point of the discharge.

6.4 The thirty-six (36) cells of Phase II were shipped to NASA Lewis Research Center.

7. Task XII - Reliability and Quality Assurance

The applicable portions of Document RQAPP #400 were applied throughout the fabrication and processing of the cells produced during Phase II of this program.

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TABLE V

FORMATION CYCLES DATA

EXPERIMENTAL 40AH CELLS

PROGRAM PHASE II CELLS

	Ê.			TION CYCLE	LE :. 1				FORMATION CYCLE NO. 2					
GRP.	SEPARATOR	CELL	CHARGE	DISCH'G	DRAIN	1		RGE INPUT		TOTAL	DISCH	ARGE	DRAIN	TOTAL
NO.	TYPE	S/N	INPUT	OUTPUT	OUTPUT	OUTPUT	1.5A	1.0A	0.54	INPUT	OUTPUT	Vn	OUTPUT	OUTPUT
	[·		(AH)	(AH)	(AH)	(AH)	(AII)	(AH)	(AH)	(AE)	(AH)	(V)	(AE) 1.17 1.07 1.05 0.95 0.95 0.78 2.06 1.96 2.12 1.56 1.82 1.82 1.82 1.42 0.98	(AH)
	1	1	44.25	34.97	1.49	36.46	45.00		Į	45.00	42.02	1.466	1, ,-	43.19
		2	44.25	35.14	1.38	36.52	45.00	ļ	Į .	41.00	42.31	1.461		
1	3420-25	3	44.25	35.24	1.20	36.44	45.00		ł	45.00	42,40	1.462		43.38
-	5420-25	4	44.25	35.61	0.98	36.59	45.00			45.00	42,40			43.35
		5	44.25	35.41	1,30	36.71	45.00	1	· ·			1.462		43.31
	1	6						1		45.00	42.37	1.468		43.42
		0	44.25	35.49	1.19	36.68	45.00		[45.00	42.42	1.469	0.78	43.20
		1	27.00	22.62	1.55	24.17	29.25			29.25	24.00	1.350	2.06	26.06
		2	27.00	22.65	1.4~	24.09	29.25			29.25	24.15	1.354	1.96	26.11
2	X-10	3	27.00	22.82	1.28	24.10	29.25	}	j :	29.25	24.17	1.361		26.29
•		4	27,00	22.59	1.51	24.10	29.25			29.25	24.29	1.368		26.25
		5	27.00	22.87	1.22	24.09	29.25			29.25	24.47	1.354		26.29
		6	27.00	22.59	1.38	23.97	29.25			29.25	24.85	1.350		26.67
									}	<u>}</u>		}	<u> </u>	
		1	36.00	33.56	0.91	34.47	45.00		1	45.00	40.48	1.446		41.90
-		2	36.00	33.98	0,68	34.60	45.00			45.00	40.74	1.455		41.72
3	X-39	3	36.00	34.29	0.90	35.19	45.00			45.00	40.59	1.452	0.93	41.52
	1	4	36.00	34.11	1.24	35.35	45.00			45.00	40.59	1.449	0.98	41.57
		5	36.00	32.18	1.97	34.15	45.00			45.00	40.51	1.447	1.63	42.34
		6	36.00	32.16	1.86	34.02	45.00			45.00	40,51	1.449	1.77	42.28
		1	22.87	19.0B	1.53	20.61	24.00	13.00	8.00	45.00	36,24	1.349	2.35	38.59
	1 1	2	21.37	17,60	1.53	19.13	24.00	13.00	8.00		36.24	1.349	2.35	38.81
4	X-478	3	23.25	18.97	2.04		24.00	13.00	E.00	45.00	36.32	1.345	2.58	38.90
	A-410	4	22.87	18.89	1.69	20.58	24.00	13.00	8.00	45.00	36.32	1.341		
		5	21.37	16.48	2.62	19.10	24.00	13.00					2.53	38.95
	1	6	21.75	17.97	1.53				8.00		36.17	1.350	2.77	38.94
	<u> </u>		21.75	17.97	1.53	19.50	24.00	13.00	8.00	45.00	36.45	1.354	2.30	38.75
		1	24.00	21.19	0.95	22,14	33.75	10,50		44.25	35.28	1.393	2,50	37.78
	1	2	24.00	21.19	1,06	22.25	33.75	9.00		42.75	33.86	1.392	2.65	36.51
5	X-104	3	24.00	21.37	0.81	22.18	33.75	9.00	·	42.75	33.77	1.395	2.76	36.52
	1	4	24.00	21.49	0.96	22.45	33.75	8.00		41.75	33.95	1.392	2.63	36.58
	1	5	24.00	21.57	0.90	22.47	33.75	10.00		43.75	35.64	1.396	2.54	38.18
	Į į	6	24.00	21.19	1.16	22.35	33.75	11.25		45.00	36.61	1.397	2.61	38.92
<u> </u>	<u> </u>	1	27.00	23,32	0.77	24.09	33.75	10.50		44.25	35.17	1.382	3.05	38.22
		2	27.00	23,42	0.66	24.08	33.75	11.25		45.00	35.17	1.384	2.72	36.22
6	x-107	3	27.00	25	0.92	24,17	33.75	11.25		45.00	35.58	1.388	3.10	38.68
v	1	4	27.00	23.1:	0.52	23.95	33.75	10.00		43.75	35.72	1.388		
	f (27.00	23.25	0.81	23.95	33.75	10.00		43.75	34.85	1.387	2.41	38.13
	1	5	27.00	23.62	1.09	24.11	33.75	10.00		43.75	34.85	1.386	2.25	37.10
			·					10.00		13.13	34.45	1.300	2.74	37.03

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