HEAT STERILIZABLE Ni-Cd BATTERY DEVELOPMENT

Jet Propulsion Laboratory
Contract No. 951972

TEXAS INSTRUMENTS
INCORPORATED

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Report for Third Quarter
1 January to 31 March 1968

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This work was performed for the Jet Propulsion Laboratory, California Institute of Technology, sponsored by the National Aeronautics and Space Administration under Contract NAS-7-100; Task Order No. RD-26

metallurgical
materials division

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ABSTRACT

The work performed to date has established the technical feasibility of heat-sterilization of sealed nickel-cadmium cells. They maintain approximately 75% of the nominal capacity after heat-sterilization in fully discharged condition. The major problem areas identified and on which work is now being done are (1) changes in the oxygen-free capacity and morphology or crystal structure of the positive plate (2) oxidation of excess cadmium metal in the negative plate and (3) changes in the electrolyte absorption and distribution during heat-sterilization.

Electrochemical performance data for automatic continuous cycling of sealed control cells containing the two best available heat-sterilizable separators and various levels of electrolyte are presented. There is little change in the performance of these cells as a function of cycling. The delivered capacity increases as the rate of charge increases, other conditions being the same. Optimum electrolyte level appears to be between 70 and 80% of the free pore volume.

As a part of the factorial design experiment, preliminary data on the effect of cobalt on the positive and indium as well as thallium on the negative plate performance before and after sterilization are presented. There does not appear to be a major improvement in the post-sterilization performance characteristics of these cells as a result of these additions.

The relative amount of electrolyte appears to be the major factor in the performance of these cells. Wetting and KOH absorption characteristics of the separator, the positive and the negative plate change as a result of sterilization and cycling. These modify the electrolyte distribution in the starved cell and also affect the oxygen recombination rates at the negative plate. Data for the changes in the KOH pick up by individual components are given.

Further physico-chemical characterizations of the active cell components before and after heat-sterilization are underway. These include x-ray diffraction, DTA, TGA and electron microscopic studies. Typical data for DTA studies are presented. An attempt is underway to correlate and interpret these results.

Rectangular sealed cells for further factorial design studies have been built and are undergoing characterization cycles prior to heat-sterilization.

I. INTRODUCTION

This is the third quarterly progress report on the heat-sterilizable nickel-cadmium battery development under Jet Propulsion Laboratory Contract No. 951972, sponsored under NASA Contract NAS-7-100, Task Order No. RD-26. The object of this contract is to perform research and development work leading to the design, development, fabrication and testing of sealed, rechargeable, nickel-cadmium cells capable of heat sterilization.

The heat sterilization requirements include testing at 135°C for type approval, and 125°C testing for flight acceptance. At the 135°C sterilization temperature, the heating rate is 19°C/hour. The chamber is cooled at the same rate at which it was heated. Two such cycles are required. For preliminary testing one 120-hour cycle may be used.

The specific tasks under this contract are divided into three broad catagories: (1) electrochemistry involving statistical and other experiments for characterizing and optimizing electrodes, electrolyte and separators for heat-sterilizable Ni-Cd cell, (2) case design for hermetically sealed, heat sterilizable cells, and (3) fabrication and evaluation of rectangular, 4 AH sealed cells before and after heat sterilization.

The details of each specific task requirement are given in the First Progress Report and will not be repeated here. The work performed during the third quarter is described in the following sections.

II. ELECTROCHEMICAL INVESTIGATIONS

2.1 Behavior of Sealed Control Cells

As a part of the factorial design experiments for the development of hermetically sealed, heat sterilizable Nickel-Cadmium cells, it is necessary to develop a standard, reproducible control cell against which cells containing different design variables can be compared. Rectangular control cells containing heat-sterilizable polypropylene separator types Pellon 14019 and Pellon FT2140 and with varying amounts (60, 70, 80 and 90% of free pore volume) of 30% KOH have been fabricated according to the procedure described in the second quarter progress report and have been undergoing automatic charge discharge cycling tests to determine the performance on continued cycling prior to sterilization. Parameters measured include end of charge voltage and pressure, capacity delivered to 1.00 V cut off, end of charge and end of discharge resistances and in selected cells, oxygen recombination rates. One object of this continued cycling of control cells is to determine what if any degradation in capacity or increase of resistance occurs during life cycles. Cycle data for cells containingPellon 14019 separator for various rates of charge and discharge are given in Table I for 59 cycles. After the initial several characterization cycles, only selected, representative cycle data are presented for the sake of conciseness consistant with clarity and usefulness. Similar data for Pellon FT2140 are given in Table II. The efficiency data given in Tables I and II are not based upon so called "rated capacity" which is a variable quantity but are based upon theoretical capacity of the plates as determined by chemical analysis of the positive plates. This theoretical capacity for cells of Table I and II is 5.58 AH.

An examination of the data of Tables I and II lead to the following general observations and conclusions:

a. The rate of charge has a significant effect on the charge acceptance and on the delivered capacity for a given rate of discharge, in this case 2 ampere discharge rate to 1.0 V cut off. Greater the rate of charge, greater is the charge acceptance and the output efficiency. Thus, for instance, with the cells containing type 14019 separator and 80% pore fill, the efficiency values are approximately 55%, 68% and 78% for charge rates of 250 ma, 400ma and 1.0A respectively. For the cells containing FT2140 separator and 80% pore fill, the corresponding efficiency values are approximately 35%, 50% and 70% respectively. It is well established that the charge acceptance (or oxygen-free capacity) at the positive plate increases with the charge rate. This is related to the fact that the oxygen overvoltage increases more rapidly with increase in current density than does the overvoltage for the oxidation of the positive active material.

b. The optimum amount of the electrolyte with both types of separator is between 70 and 80% of the free volume. Cells with 60% pore fill exhibited high resistances, particularly with FT2140 separator. Cells with 90% pore fill exhibited excessive end of charge pressures and relatively slow rates of oxygen recombination as expected.

c. There is significant variation in the end of charge pressure data and more careful measurements of the pressure rise on charge and required to determine the effect of charge rate on pressure rise. Data for the pressure decay on stand are being analysed now. Preliminary analysis shows little difference in the rates of oxygen recombination with 70% and 80% pore fills in the cells containing 14019 and FT2140 separators.

d. There is no significant difference in the end of charge voltages between the two types of separators with 70% and 80% pore fills.

While, from these results 14019 separator appears to be superior to FT2140, the tendency of cells with 14019 to develop short circuits after second heat sterilization requires the inclusion of both separator materials in further factorial experiments.

TABLE I

Performance Data for Heat-Sterilizable, Sealed Rectangular, Ni-Cd Control Cells (Unsterilized) With Polypropylene 14019 Separator on Continuous Cycling at 22°C

Cell No	% Pore Fill	Cycle No.	Charge Data						Discharge Data; 10V Cut Off					Comments
			Amp.	Hrs.	AH Input	ECV Volts	ECP PSIA	ECR m	Amp	AH Output	EDP PSIA	EDR m	Eff. %	
1	60	1	0.250	24	6	1.405	26.7	3.24	2.0	2.634	15.7	4.42	46.6	
2	60	1	0.250	24	6	1.466	24.7	2.60	2.0	2.752	17.7	3.40	48.7	
3	70	1	0.250	24	6	1.409	29.7	2.49	2.0	3.218	18.2	2.63	57.0	
4	70	1	0.250	24	6	1.403	32.7	2.34	2.0	2.784	17.2	2.58	49.3	
5	80	1	0.250	24	6	1.403	59.7	2.32	2.0	3.184	41.7	2.47	56.4	
6	80	1	0.250	24	6	1.409	36.2	2.44	2.0	3.318	25.2	2.55	58.7	
7	90	1	0.250	24	6	1.405	79.7	2.09	2.0	3.284	68.7	2.21	58.1	
8	90	1	0.250	24	6	1.406	67.7	2.20	2.0	3.318	58.7	2.33	58.7	
1	60	2	0.250	24	6	1.405	36.7	3.27	2.0	2.734	17.2	4.36	43.4	
2	60	2	0.250	24	6	1.404	29.2	2.67	2.0	2.834	14.7	3.41	50.2	
3	70	2	0.250	24	6	1.406	42.7	2.52	2.0	3.152	23.7	2.79	55.8	
4	70	2	0.250	24	6	1.405	41.7	2.36	2.0	2.934	21.7	2.67	51.9	
5	80	2	0.250	24	6	1.409	70.7	2.37	2.0	3.334	51.2	2.62	59.0	#7 & 8 removed due
6	80	2	0.250	24	6	1.407	57.7	2.47	2.0	3.300	39.7	2.70	58.4	to high pressures.
1	60	3	0.250	24	6	1.401	39.7	3.28	2.0	2.134	16.7	3.74	37.8	
2	60	3	0.250	24	6	1.400	26.7	2.69	2.0	2.200	14.7	2.98	38.9	Lower efficiency probably due to
3	70	3	0.250	24	6	1.403	53.7	2.50	2.0	2.484	20.2	2.48	44.0	fluctuations in
4	70	3	0.250	24	6	1.402	50.7	2.31	2.0	2.334	16.7	2.35	41.3	charge current over-
5	80	3	0.250	24	6	1.408	74.7	2.31	2.0	2.866	38.7	2.27	50.7	night resulting in lower net input.

TABLE I - continued

Performance Data for Heat-Sterilizable, Sealed Rectangular, Ni-Cd Control Cells (Unsterilized)
With Polypropylene 14019 Separator on Continuous Cycling at 22°C

Cell No	% Pore Fill	Cycle No.	Charge Data Amp.	Hrs.	AH Input	ECV Volts	ECP PSIA	ECR m	Discharge Data; 10V Cut Off Amp	AH Output	EDP PSIA	EDR m	Eff. %	Comments
6	80	3	0.250	24	6	1.405	66.7	2.42	2.0	2.652	35.7	2.40	46.9	
1	60	4	0.250	24	6	1.405	43.7	3.38	2.0	2.318	18.7	3.76	41.0	
2	60	4	0.250	24	6	1.403	34.7	2.79	2.0	2.352	15.7	2.87	41.6	
3	70	4	0.250	24	6	1.405	62.7	2.54	2.0	2.552	35.2	2.44	45.2	
4	70	4	0.250	24	6	1.404	58.2	2.42	2.0	2.566	29.7	2.29	45.4	
5	80	4	0.250	24	6	1.414	76.7	2.41	2.0	3.266	56.7	2.29	57.8	
6	80	4	0.250	24	6	1.409	76.7	2.57	2.0	2.918	56.7	2.41	51.6	
1	60	6	0.400	16	6.4	1.410	61.2	3.29	2.0	2.384	14.7	3.80	42.2	
2	60	6	0.400	16	6.4	1.410	46.2	2.71	2.0	2.434	14.7	3.01	43.1	
3	70	6	0.400	16	6.4	1.414	75.7	2.60	2.0	2.866	26.7	2.56	50.7	
4	70	6	0.400	16	6.4	1.411	62.7	2.39	2.0	2.700	29.7	2.40	47.8	
5	80	6	0.400	16	6.4	1.423	74.7	2.36	2.0	3.718	41.7	2.33	65.8	
6*														cell out of circuit.
1	60	7	0.400	16	6.4	1.411	64.7	3.06	2.0	2.700	21.7	4.10	47.8	
2	60	7	0.400	16	6.4	1.410	48.7	2.53	2.0	2.734	18.7	3.06	43.4	
3	70	7	0.400	16	6.4	1.417	76.7	2.51	2.0	3.400	33.7	2.81	60.2	
4	70	7	0.400	16	6.4	1.412	63.2	2.26	2.0	3.066	43.7	2.69	54.3	
5	80	7	0.400	16	6.4	1.425	74.7	2.25	2.0	4.084	47.7	2.45	72.3	

TABLE I continued

Performance Data for Heat-Sterilizable, Sealed Rectangular, Ni-Cd Control Cells (Unsterilized) With Polypropylene 14019 Separator on Continuous Cycling at 22°C

Cell No	% Pore Fill	Cycle No.	Charge Data: Amp.	Hrs.	AH Input	ECV Volts	ECP PSIA	ECR mΩ	Discharge Data; 10V Cut Off: Amp	AH Output	EDP PSIA	EDR mΩ	Eff. %	Comments
1	60	10	0.400	16	6.4	1.393	57.7	3.27	2.0	2.552	26.7	4.60	45.2	cell #1, 2, 6 out of
2	60	10	0.400	16	6.4	1.413	59.7	2.69	2.0	2.752	23.7	3.74	48.7	circuit on charge
3	70	10	0.400	16	6.4	1.421	75.7	2.53	2.0	3.800	36.2	2.72	67.3	
4	70	10	0.400	16	6.4	1.419	63.2	2.33	2.0	3.552	46.2	2.61	62.9	
5	80	10	0.400	16	6.4	1.427	76.2	2.27	2.0	3.952	48.7	2.45	69.9	
6	30	10	0.400	16	6.4	1.323	87.7	1.58	2.0	3.200	77.7	2.45	56.6	both ECV and Eff. lower cause: unknown
1	60	12	0.400	16	6.4	1.379	68.7	3.24	2.0	2.384	13.2	4.48	42.2	cell #1, 2, 6 out of
2	60	12	0.400	16	6.4	1.380	50.7	2.71	2.0	2.434	2.2	3.56	43.1	circuit on charge
3	70	12	0.400	16	6.4	1.429	14.7	2.79	2.0	3.784	4.7	2.61	67.0	
4	70	12	0.400	16	6.4	1.418	64.7	2.33	2.0	3.434	36.2	2.50	60.8	
5	80	12	0.400	16	6.4	1.433	14.7	2.31	2.0	3.934	-7.3	2.29	69.6	
6	80	12	0.400	16	6.4	1.423	67.7	2.66	2.0	3.652	46.2	2.34	64.6	
1	60	13	1.0	5	5.0	1.382	74.7	2.96	2.0	3.384	23.7	4.20	59.9	
2	60	13	1.0	5	5.0	1.427	64.7	2.53	2.0	3.384	18.2	3.25	59.9	
3	70	13	1.0	5	5.0	1.444	52.7	2.35	2.0	4.066	19.7	2.50	72.0	
4	70	13	1.0	5	5.0	1.441	61.7	2.23	2.0	4.052	29.7	2.37	69.9	
5	80	13	1.0	5	5.0	1.462	49.7	2.13	2.0	4.052	27.2	2.15	71.7	
6	80	13	1.0	5	5.0	1.442	65.7	2.35	2.0	4.066	42.7	2.26	72.0	

TABLE I[illegible] continued

Performance Data for Heat-Sterilizable, Sealed Rectangular, Ni-Cd Control Cells (Unsterilized) With Polypropylene 14019 Separator on Continuous Cycling at 22°C

Cell No	% Pore Fill	Cycle No.	Charge Data: Amp.	Hrs.	AH Input	ECV Volts	ECP PSIA	ECR m[illegible]	Discharge Data; 10V Cut Off: Amp	AH Output	EDP PSIA	EDR m[illegible]	Eff. %	Comments
1	60	14	1.0	5	5.0	1.449	40.7	3.22	2.0	3.752	14.7	4.83	66.4	
2	60	14	1.0	5	5.0	1.448	36.7	3.22	2.0	3.752	14.7	4.82	66.4	
3	70	14	1.0	5	5.0	1.452	35.7	2.47	2.0	4.134	14.7	2.73	73.2	
4	70	14	1.0	5	5.0	1.449	47.2	2.32	2.0	4.052	14.7	2.52	71.7	
5	80	14	1.0	5	5.0	1.476	36.7	2.17	2.0	4.100	14.7	2.26	72.6	
6	80	14	1.0	5	5.0	1.451	42.7	2.41	2.0	4.134	14.7	2.48	73.2	
1	60	15	1.0	5	5.0	1.451	60.7	2.78	2.0	4.100	20.7	5.13	72.6	
2	60	15	1.0	5	5.0	1.450	47.2	2.34	2.0	4.066	16.2	3.72	72.0	
3	70	15	1.0	5	5.0	1.453	46.7	2.26	2.0	4.352	18.7	2.75	77.0	
4	70	15	1.0	5	5.0	1.451	57.7	2.06	2.0	4.266	24.7	2.58	75.5	
5	80	15	1.0	5	5.0	1.481	49.7	1.93	2.0	4.300	26.2	2.36	76.1	
6	80	15	1.0	5	5.0	1.452	57.7	2.33	2.0	4.352	37.7	2.57	77.0	
1	60	18	1.0	5	5.0	1.455	49.7	3.08	2.0	4.166	18.7	5.86	73.7	
2	60	18	1.0	5	5.0	1.457	44.7	2.60	2.0	4.218	16.2	2.36	74.7	
3	70	18	1.0	5	5.0	1.457	41.2	2.40	2.0	4.400	17.7	1.52	77.9	
4	70	18	1.0	5	5.0	1.455	47.2	2.25	2.0	4.352	21.7	1.56	77.0	
5	80	18	1.0	5	5.0	1.493	48.2	1.97	2.0	4.300	26.2	1.90	76.1	
6	80	18	1.0	5	5.0	1.457	49.7	2.38	2.0	4.418	32.2	1.31	78.2	

TABLE I — continued

Performance Data for Heat-Sterilizable, Sealed Rectangular, Ni-Cd Control Cells (Unsterilized) With Polypropylene 14019 Separator on Continuous Cycling at 22°C

Cell No	% Pore Fill	Cycle No.	Charge Data						Discharge Data; 10V Cut Off					Comments
			Amp.	Hrs.	AH Input	ECV Volts	ECP PSIA	ECR mΩ	Amp	AH Output	EDP PSIA	EDR mΩ	Eff. %	
1	60	21	1.0	5	5.0	1.451	37.7	3.54	2.0	3.784	17.2	6.39	67.0	
2	60	21	1.0	5	5.0	1.455	46.2	2.91	2.0	4.134	21.2	4.23	73.2	
3	70	21	1.0	5	5.0	1.451	33.2	2.77	2.0	4.284	16.2	2.97	75.8	
4	70	21	1.0	5	5.0	1.449	38.7	2.71	2.0	4.266	19.7	2.96	75.5	
5	80	21	1.0	5	5.0	1.464	40.7	2.64	2.0	4.218	23.7	2.78	74.7	
6	80	21	1.0	5	5.0	1.452	39.7	2.63	2.0	4.334	27.7	2.61	76.7	
7	90	21	1.0	5	5.0	1.444	45.7	2.40	2.0	4.218	39.7	2.45	74.7	
8	90	21	1.0	5	5.0	1.443	43.2	2.65	2.0	4.334	37.2	2.68	76.7	
1	60	22	1.0	5	5.0	1.460	48.7	3.35	2.0	3.818	26.2	7.98	67.6	
2	60	22	1.0	5	5.0	1.462	55.7	2.77	2.0	4.166	21.2	4.46	73.7	
3	70	22	1.0	5	5.0	1.461	42.7	2.66	2.0	4.400	17.7	3.00	77.9	
4	70	22	1.0	5	5.0	1.460	47.7	2.68	2.0	4.352	22.2	2.98	77.0	
5	80	22	1.0	5	5.0	1.475	52.2	2.62	2.0	4.252	27.2	2.84	75.3	
6	80	22	1.0	5	5.0	1.461	50.2	2.58	2.0	4.434	34.2	2.70	78.5	
7	90	22	1.0	5	5.0	1.451	60.7	2.39	2.0	4.334	53.7	2.59	76.7	
8	90	22	1.0	5	5.0	1.449	57.7	2.64	2.0	4.434	49.2	2.84	78.5	
1	60	23	1.0	5	5.0	1.459	52.2	3.55	2.0	3.900	28.7	8.53	69.0	
2	60	23	1.0	5	5.0	1.459	54.7	2.91	2.0	4.266	20.7	4.60	75.5	
3	70	23	1.0	5	5.0	1.457	39.7	2.75	2.0	4.434	16.2	3.11	78.5	

Performance Data for Heat-Sterilizable, Sealed Rectangular, Ni-Cd Control Cells (unsterilized) With Polypropylene 14019 Separator on Continuous Cycling at 22°C

Cell No	% Pore Fill	Cycle No.	Charge Data						Discharge Data; 10V Cut Off					Comments
			Amp.	Hrs.	AH Input	ECV Volts	ECP PSIA	ECR m Ω	Amp	AH Output	EDP PSIA	EDR m Ω	Eff. %	
4	70	23	1.0	5	5.0	1.457	44.7	2.70	2.0	4.384	20.2	3.09	77.6	
5	80	23	1.0	5	5.0	1.500	50.7	2.60	2.0	4.284	25.2	2.97	75.8	
6	80	23	1.0	5	5.0	1.458	47.2	2.65	2.0	4.452	30.2	2.80	78.8	
7	90	23	1.0	5	5.0	1.450	61.2	2.44	2.0	4.366	53.7	2.64	77.3	
8	90	23	1.0	5	5.0	1.447	58.7	2.71	2.0	4.400	49.2	2.81	77.9	
2	60	28	1.0	5	5.0	1.461	50.7	2.56	2.0	4.352	20.7	4.15	77.0	cell #1 removed from test
3	70	28	1.0	5	5.0	1.458	39.7	2.45	2.0	4.484	15.7	2.50	79.4	
4	70	28	1.0	5	5.0	1.457	43.2	2.37	2.0	4.466	19.7	2.54	79.0	
5	80	28	1.0	5	5.0	1.473	46.7	2.25	2.0	4.384	22.7	2.48	77.6	
6	80	28	1.0	5	5.0	1.458	45.7	2.23	2.0	4.518	27.7	2.29	80.0	
7	90	28	1.0	5	5.0	1.452	51.7	1.99	2.0	4.434	38.7	2.12	78.5	
8	90	28	1.0	5	5.0	1.451	50.2	2.33	2.0	4.384	35.7	2.44	77.6	

TABLE I (Contd)

PERFORMANCE DATA FOR HEAT STERILIZABLE, SEALED RECTANGULAR, Ni-Cd CONTROL CELLS (UNSTERILIZED) WITH POLYPROPYLENE 14019 SEPARATOR ON CONTINUOUS CYCLING AT 22°C

Cell No.	% Pore Fill	Cycle No.	Charge Data						Discharge Data; 10 V Cutoff					Comments
			Amp	Hrs	AH Input	ECV Volts	ECP psia	ECR mΩ	Amp	AH Output	EDP psia	EDR mΩ	Eff. %	
2	60	33	1.0	5	5.0	1.444	49.7	2.83	2.0	4.218	21.7	5.64	74.7	
3	70	33	1.0	5	5.0	1.457	37.7	2.57	2.0	4.384	17.2	2.85	77.6	
4	70	33	1.0	5	5.0	1.459	39.7	2.62	2.0	4.366	21.7	2.92	77.3	
5	80	33	1.0	5	5.0	1.508	44.7	2.67	2.0	4.334	23.2	2.77	76.7	
6	80	33	1.0	5	5.0	1.460	42.2	2.51	2.0	4.384	29.7	2.58	77.6	
7	90	33	1.0	5	5.0	1.457	39.7	2.29	2.0	4.318	31.7	2.40	76.4	
8	90	33	1.0	5	5.0	1.459	52.7	2.79	2.0	3.918	38.7	2.85	69.3	
2	60	43	1.0	5	5.0	1.429	49.7	2.73	2.0	4.218	11.7	6.61	74.7	
3	70	43	1.0	5	5.0	1.457	34.7	2.47	2.0	4.452	13.7	2.80	78.8	
4	70	43	1.0	5	5.0	1.463	39.7	2.58	2.0	4.418	17.2	2.95	78.2	
5	80	43	1.0	5	5.0	1.512	43.7	2.49	2.0	4.352	18.7	2.82	77.0	
6	80	43	1.0	5	5.0	1.463	40.7	2.46	2.0	4.500	25.2	2.59	79.6	
7	90	43	1.0	5	5.0	1.460	33.2	2.23	2.0	4.500	21.7	2.36	79.6	
8	90	43	1.0	5	5.0	1.458	45.7	2.82	2.0	4.400	24.7	3.08	77.9	

TABLE I (Contd)

PERFORMANCE DATA FOR HEAT STERILIZABLE, SEALED RECTANGULAR, Ni-Cd CONTROL CELLS (UNSTERILIZED) WITH POLYPROPYLENE 14019 SEPARATOR ON CONTINUOUS CYCLING AT 22°C

Cell No.	% Pore Fill	Cycle No.	Charge Data Amp	Hrs	AH Input	ECV Volts	ECP psia	ECR mΩ	Discharge Data; 10 V Cutoff Amp	AH Output	EDP psia	EDR mΩ	Eff. %	Comments
2	60	47	.250	24	6.0	1.407	42.7	2.98	2.0	3.352	4.7	6.58	59.3	
3	70	47	.250	24	6.0	1.411	44.7	2.37	2.0	3.918	4.7	2.77	69.3	
4	70	47	.250	24	6.0	1.412	50.7	2.48	2.0	3.852	44.7	2.91	68.2	
5	80	47	.250	24	6.0	1.409	31.7	2.47	2.0	4.300	24.7	2.80	76.1	
6	80	47	.250	24	6.0	1.413	64.7	2.27	2.0	4.134	164.7	2.44	73.2	
7	90	47	.250	24	6.0	1.414	60.7	2.13	2.0	4.118	249.7	2.25	72.9	
8	90	47	.250	24	6.0	1.411	69.7	2.77	2.0	4.818	184.7	3.10	67.6	
2	60	54	.400	16	6.4	1.422	57.7	2.98	2.0	3.634	11.7	6.68	64.3	
3	70	54	.400	16	6.4	1.432	57.7	2.61	2.0	4.334	16.7	2.65	76.7	
4	70	54	.400	16	6.4	1.439	59.7	2.70	2.0	4.518	23.7	2.79	80.0	
5	80	54	.400	16	6.4	1.249	49.7	2.63	2.0	4.484	17.7	2.47	79.4	
6	80	54	.400	16	6.4	1.441	64.7	2.47	2.0	4.618	35.7	2.27	81.7	
7	90	54	.400	16	6.4	1.445	61.7	2.27	2.0	4.484	47.7	2.15	79.4	
8	90	54	.400	16	6.4	1.435	52.7	2.52	2.0	4.500	14.7	2.43	79.6	

TABLE I (Contd)

PERFORMANCE DATA FOR HEAT STERILIZABLE, SEALED RECTANGULAR, Ni-Cd CONTROL CELLS (UNSTERILIZED) WITH POLYPROPYLENE 14019 SEPARATOR ON CONTINUOUS CYCLING AT 22°C

Cell No.	% Pore Fill	Cycle No.	Charge Data						Discharge Data; 10 V Cutoff					Comments
			Amp	Hrs	AH Input	ECV Volts	ECP psia	ECR mΩ	Amp	AH Output	EDP psia	EDR mΩ	EFF. %	
2	60	57	.400	16	6.4	1.420	57.2	2.92	2.0	3.452	14.7	5.58	61.1	
3	70	57	.400	16	6.4	1.429	56.7	2.50	2.0	4.252	16.7	2.47	75.3	
4	70	57	.400	16	6.4	1.434	62.7	2.62	2.0	4.434	22.7	2.66	78.5	
5	80	57	.400	16	6.4	1.435	44.7	2.49	2.0	4.552	18.7	2.53	80.6	
6	80	57	.400	16	6.4	1.435	65.7	2.34	2.0	4.552	33.7	2.27	80.6	
7	90	57	.400	16	6.4	1.437	68.7	2.15	2.0	4.552	46.7	2.15	80.6	
8	90	57	.400	16	6.4	1.430	60.7	2.48	2.0	4.384	25.7	2.48	77.6	
2	60	58	1.0	5	5.0	1.457	-	-	2.0	3.952	-	7.21	69.9	
3	70	58	1.0	5	5.0	1.461	-	-	2.0	4.418	-	2.50	78.2	
4	70	58	1.0	5	5.0	1.458	-	-	2.0	4.400	-	2.66	77.9	
5	80	58	1.0	5	5.0	1.479	-	-	2.0	4.384	-	2.54	77.6	
6	80	58	1.0	5	5.0	1.456	-	-	2.0	4.452	-	2.15	78.8	
7	90	58	1.0	5	5.0	1.447	-	-	2.0	4.352	-	2.02	77.0	
8	90	58	1.0	5	5.0	1.459	-	-	2.0	4.352	-	2.48	77.0	

TABLE I (Contd)

PERFORMANCE DATA FOR HEAT STERILIZABLE, SEALED RECTANGULAR, Ni-Cd CONTROL CELLS (UNSTERILIZED) WITH POLYPROPYLENE 14019 SEPARATOR ON CONTINUOUS CYCLING AT 22°C

Cell No.	% Pore Fill	Cycle No.	Charge Data						Discharge Data: 10 V Cutoff					Comments
			Amp	Hrs	AH Input	ECV Volts	ECP psia	ECR mΩ	Amp	AH Output	EDP psia	EDR mΩ	Eff %	
2	60	59	1.0	5	5.0	1.464	-	3.10	2.0	4.066	-	8.48	72.0	
3	70	59	1.0	5	5.0	1.462	-	2.64	2.0	4.500	-	2.79	79.6	
4	70	59	1.0	5	5.0	1.469	-	2.80	2.0	4.618	-	3.02	81.7	
5	80	59	1.0	5	5.0	1.490	-	2.81	2.0	4.434	-	2.94	78.5	
6	80	59	1.0	5	5.0	1.466	-	2.59	2.0	4.634	-	2.42	82.0	
7	90	59	1.0	5	5.0	1.470	-	2.32	2.0	4.700	-	2.29	83.2	
8	90	59	1.0	5	5.0	1.469	-	2.77	2.0	4.466	-	2.86	79.0	

TABLE II

Performance Data for Heat-Sterilizable, Sealed Rectangular, Ni-Cd Control Cells (Unsterilized) With Polypropylene FT2140 Separator on Continuous Cycling at 22°C

Cell No	% Pore Fill	Cycle No.	Charge Data						Discharge Data; 10V Cut Off					Comments
			Amp.	Hrs.	AH Input	ECV Volts	ECP PSIA	ECR mΩ	Amp	AH Output	EDP PSIA	EDR mΩ	Eff. %	
1	60	1	.250	24	6	1.412	16.7	8.57	2.0	2.066	15.7	24.75	36.6	
2	60	1	.250	24	6	1.409	10.7	9.51	2.0	1.966	2.7	26.53	34.8	
3	70	1	.250	24	6	1.410	14.7	3.36	2.0	2.752	4.7	3.35	48.7	
4	70	1	.250	24	6	1.403	17.7	2.50	2.0	2.484	-1.3	2.50	44.0	
5	80	1	.250	24	6	1.407	21.7	2.76	2.0	2.800	11.7	2.37	49.6	
6	80	1	.250	24	6	1.404	38.7	2.49	2.0	2.584	22.7	2.26	45.7	
7	90	1	.250	24	6	1.411	57.7	2.40	2.0	3.200	43.7	2.01	56.6	
8	90	1	.250	24	6	1.410	50.7	2.52	2.0	3.034	39.7	2.32	53.7	
1	60	2	.250	24	6	1.401	15.7	8.58	2.0	1.652	14.7	24.78	29.2	
2	60	2	.250	24	6	1.395	14.7	8.53	2.0	1.566	2.7	22.29	27.7	
3	70	2	.250	24	6	1.399	89.7	3.01	2.0	2.200	4.7	3.52	38.9	
4	70	2	.250	24	6	1.395	24.7	2.17	2.0	2.018	-4.3	2.63	25.7	
5	80	2	.250	24	6	1.395	32.7	2.26	2.0	2.234	11.7	2.60	39.5	
6	80	2	.250	24	6	1.395	61.7	2.07	2.0	2.066	24.7	2.47	36.6	
7	90	2	.250	24	6	1.390	77.2	1.99	2.0	2.466	56.7	2.23	43.6	
8	90	2	.250	24	6	1.353	74.7	2.17	2.0	2.334	42.7	2.52	41.3	
1	60	3	.250	24	6	1.397	15.7	8.17	2.0	1.534	14.7	24.80	27.2	
2	60	3	.250	24	6	1.392	15.7	7.75	2.0	1.484	3.7	22.30	26.3	
3	70	3	.250	24	6	1.396	26.7	3.00	2.0	2.052	8.7	3.29	36.3	

TABLE II

Performance Data for Heat-Sterilizable, Sealed Rectangular, Ni-Cd Control Cells (Unsterilized) With Polypropylene FT2140 Separator on Continuous Cycling at 22°C

Cell No	% Pore Fill	Cycle No.	Charge Data: Amp.	Hrs.	AH Input	ECV Volts	ECP PSIA	ECR mΩ	Discharge Data; 10V Cut Off: Amp	AH Output	EDP PSIA	EDR mΩ	Eff. %	Comments
4	70	3	.250	24	6	1.393	29.7	2.20	2.0	1.934	1.7	2.39	34.2	
5	80	3	.250	24	6	1.394	40.7	2.41	2.0	2.152	17.7	2.36	38.1	
6	80	3	.250	24	6	1.394	65.7	2.13	2.0	2.084	36.7	2.27	36.9	
7	90	3	.250	24	6	1.352	75.7	1.99	2.0	2.352	55.7	2.09	41.6	
8	90	3	.250	24	6	1.345	64.7	2.18	2.0	2.166	43.7	2.33	38.3	
1	60	4	.250	24	6	1.398	15.7	8.55	2.0	1.434	14.7	23.09	25.4	
2	60	4	.250	24	6	1.394	14.7	8.03	2.0	1.400	2.7	19.88	24.8	
3	70	4	.250	24	6	1.395	29.7	3.19	2.0	1.852	4.7	3.41	32.8	
4	70	4	.250	24	6	1.392	33.7	2.24	2.0	1.734	-3.3	2.48	30.7	
5	80	4	.250	24	6	1.393	44.7	2.45	2.0	1.934	14.7	2.42	34.2	
6	80	4	.250	24	6	1.393	70.7	2.13	2.0	1.784	29.2	2.42	31.6	
7	90	4	.250	24	6	1.354	62.7	1.91	2.0	2.000	35.7	2.25	35.4	
8	90	4	.250	24	6	1.354	51.7	2.14	2.0	1.984	29.7	2.52	35.1	
1	60	5	.250	24	6	1.398	15.7	8.23	2.0	1.484	14.7	30.23	26.3	
2	60	5	.250	24	6	1.397	15.7	7.90	2.0	1.466	2.7	25.55	25.9	
3	70	5	.250	24	6	1.397	33.7	2.95	2.0	1.934	7.7	3.20	34.2	
4	70	5	.250	24	6	1.393	34.7	2.16	2.0	1.834	-0.3	2.30	32.5	
5	80	5	.250	24	6	1.394	46.2	2.32	2.0	2.066	16.2	2.26	36.6	
6	80	5	.250	24	6	1.393	68.7	2.07	2.0	2.000	32.7	2.21	35.4	

TABLE II

Performance Data for Heat-Sterilizable, Sealed Rectangular, Ni-Cd Control Cells (Unsterilized) With Polypropylene FT2140 Separator on Continuous Cycling at 22°C

Cell No	% Pore Fill	Cycle No.	Charge Data: Amp.	Hrs.	AH Input	ECV Volts	ECP PSIA	ECR mΩ	Discharge Data; 10V Cut Off: Amp	AH Output	EDP PSIA	EDR mΩ	Eff. %	Comments
7	90	5	.250	24	6	1.359	77.7	2.00	2.0	2.466	14.7	6.36	43.6	
8	90	5	.250	24	6	1.376	56.7	2.19	2.0	2.200	33.7	2.24	38.9	
1	60	6	.400	16	6.4	1.409	15.7	7.20	2.0	1.634	15.7	29.74	28.9	
2	60	6	.400	16	6.4	1.405	19.7	7.16	2.0	1.518	4.7	25.37	26.9	
3	70	6	.400	16	6.4	1.409	46.7	2.81	2.0	2.252	16.7	3.10	39.9	
4	70	6	.400	16	6.4	1.404	48.7	2.06	2.0	2.118	14.7	2.27	37.5	
5	80	6	.400	16	6.4	1.405	65.7	2.25	2.0	2.434	28.7	2.15	43.1	
6	80	6	.400	16	6.4	1.386	35.7	2.11	2.0	1.918	24.7	2.10	33.9	
7	90	6	.400	16	6.4	1.418	57.7	2.39	2.0	3.266	25.7	2.14	57.8	
8	90	6	.400	16	6.4	1.401	70.7	2.25	2.0	2.666	54.7	2.15	47.2	
1	60	7	.400	16		1.409	15.7	7.54	2.0	1.584	14.7	28.85	28.0	
2	60	7	.400	16		1.406	21.7	7.54	2.0	1.466	4.7	25.46	25.9	
3	70	7	.400	16		1.407	51.7	2.88	2.0	2.200	18.7	3.43	38.9	
4	70	7	.400	16		1.402	51.7	2.13	2.0	2.084	18.2	2.47	36.9	
5	80	7	.400	16		1.405	71.7	2.31	2.0	2.400	31.7	2.44	42.5	
6	80	7	.400	16		1.386	45.7	2.13	2.0	2.100	28.7	2.41	37.2	
7	90	7	.400	16		1.422	14.7	3.00	2.0	3.552	14.7	3.07	62.9	
8	90	7	.400	16		1.392	60.7	2.28	2.0	2.484	44.7	2.45	44.0	
1	60	8	.400	16		1.409	15.7	7.61	2.0	1.584	14.7	32.73	28.0	

TABLE II

Performance Data for Heat-Sterilizable, Sealed Rectangular, Ni-Cd Control Cells (Unsterilized) With Polypropylene FT2140 Separator on Continuous Cycling at 22°C

			Charge Data						Discharge Data; 10V Cut Off					
Cell No	% Pore Fill	Cycle No.	Amp.	Hrs.	AH Input	ECV Volts	ECP PSIA	ECR m Ω	Amp	AH Output	EDP PSIA	EDR m Ω	Eff. %	Comments
2	60	8	.400	16	6.4	1.405	15.7	7.49	2.0	1.418	4.7	25.92	25.1	X (cells 2–8, cycle 8)
3	70	8	.400	16	6.4	1.407	54.7	2.87	2.0	2.134	20.7	3.01	37.8	
4	70	8	.400	16	6.4	1.403	52.7	2.09	2.0	2.034	21.2	2.29	36.0	
5	80	8	.400	16	6.4	1.405	74.7	2.29	2.0	2.366	35.2	2.22	41.9	
6	80	8	.400	16	6.4	1.386	26.7	2.15	2.0	1.918	22.2	2.22	33.9	
7	90	8	.400	16	6.4	1.423	14.7	2.90	2.0	3.566	11.7	3.02	63.1	
8	90	8	.400	16	6.4	1.397	60.7	2.30	2.0	2.434	46.2	2.26	43.1	
1	60	9	.400	16	6.4	1.414	15.7	7.89	2.0	1.718	14.7	39.23	30.4	
2	60	9	.400	16	6.4	1.412	22.7	7.85	2.0	1.600	4.7	31.09	28.3	
3	70	9	.400	16	6.4	1.413	55.7	3.12	2.0	2.384	19.7	3.53	42.2	
4	70	9	.400	16	6.4	1.408	52.7	2.24	2.0	2.266	18.2	2.55	40.1	
5	80	9	.400	16	6.4	1.414	68.7	2.45	2.0	2.700	30.7	2.43	47.8	
6	80	9	.400	16	6.4	1.415	74.7	2.29	2.0	2.918	39.7	2.38	51.6	
7	90	9	.400	16	6.4	1.426	38.7	2.96	2.0	3.700	17.7	3.54	65.5	
8	90	9	.400	16	6.4	1.408	64.7	2.35	2.0	2.818	47.7	2.33	49.9	
1	60	10	.400	16	6.4	1.412	14.7	7.59	2.0	1.700	15.7	38.08	30.1	X (cells 1–5, cycle 10)
2	60	10	.400	16	6.4	1.406	23.7	7.31	2.0	1.434	4.7	29.39	25.4	
3	70	10	.400	16	6.4	1.411	57.7	2.64	2.0	2.334	19.7	3.32	41.3	
4	70	10	.400	16	6.4	1.407	54.7	1.90	2.0	2.218	18.2	2.34	39.3	
5	80	10	.400	16	6.4	1.409	104.7	2.07	2.0	2.600	33.2	2.33	46.0	

TABLE II

Performance Data for Heat-Sterilizable, Sealed Rectangular, Ni-Cd Control Cells (Unsterilized) With Polypropylene FT2140 Separator on Continuous Cycling at 22°C

Cell No	% Pore Fill	Cycle No.	Charge Data						Discharge Data; 10V Cut Off					Comments
			Amp.	Hrs.	AH Input	ECV Volts	ECP PSIA	ECR mΩ	Amp	AH Output	EDP PSIA	EDR mΩ	Eff. %	
6	80	10	.400	16	6.4	1.416	72.7	1.97	2.0	2.984	38.2	2.33	52.8	
7	90	10	.400	16	6.4	14.22	46.2	2.50	2.0	3.584	19.7	3.44	63.4	–
8	90	10	.400	16	6.4	1.396	51.7	2.01	2.0	2.784	34.7	2.40	49.3	
1	60	11	.400	16	6.4	1.412	14.7	8.32	2.0	1.552	14.7	47.60	27.5	
2	60	11	.400	16	6.4	1.407	24.7	8.07	2.0	1.300	4.7	33.92	23.0	
3	70	11	.400	16	6.4	1.411	59.7	2.93	2.0	2.184	20.2	3.53	38.7	
4	70	11	.400	16	6.4	1.407	55.7	2.10	2.0	2.084	18.7	2.55	36.9	
5	80	11	.400	16	6.4	1.410	74.7	2.37	2.0	2.466	35.2	2.66	43.6	
6	80	11	.400	16	6.4	1.418	72.7	2.22	2.0	2.918	35.2	2.45	51.6	
7	90	11	.400	16	6.4	1.421	49.7	2.89	2.0	3.418	19.7	3.62	60.5	
8	90	11	.400	16	6.4	1.426	14.7	3.12	2.0	3.484	14.7	3.03	16.7	
1	60	12	.400	16	6.4	1.413	14.7	8.73	2.0	1.600	14.7	50.10	28.3	
2	60	12	.400	16	6.4	1.411	24.7	8.37	2.0	1.384	13.7	32.63	24.5	
3	70	12	.400	16	6.4	1.411	60.7	2.95	2.0	2.152	20.2	3.26	38.1	
4	70	12	.400	16	6.4	1.407	55.7	2.16	2.0	2.084	18.7	2.36	36.9	
5	80	12	.400	16	6.4	1.410	77.7	2.40	2.0	2.518	35.2	2.49	44.6	
6	80	12	.400	16	6.4	1.419	73.7	2.26	2.0	2.952	35.2	2.20	52.2	
7	90	12	.400	16	6.4	1.421	51.7	2.82	2.0	3.366	19.7	3.39	59.6	
8	90	12	.400	16	6.4	1.430	14.7	2.89	2.0	3.418	14.7	3.01	60.5	

TABLE II

Performance Data for Heat-Sterilizable, Sealed Rectangular, Ni-Cd Control Cells (Unsterilized) With Polypropylene FT2140 Separator on Continuous Cycling at 22°C

Cell No	% Pore Fill	Cycle No.	Charge Data: Amp.	Charge Data: Hrs.	Charge Data: AH Input	Charge Data: ECV Volts	Charge Data: ECP PSIA	Charge Data: ECR m Ω	Discharge Data; 10V Cut Off: Amp	Discharge Data; 10V Cut Off: AH Output	Discharge Data; 10V Cut Off: EDP PSIA	Discharge Data; 10V Cut Off: EDR m Ω	Discharge Data; 10V Cut Off: Eff. %	Comments
1	60	13	1.0	5	5.0	1.453	15.7	0	2.0	2.784	14.7	48.70	49.3	
2	60	13	1.0	5	5.0	1.440	40.7	0	2.0	2.134	4.7	31.20	37.8	
3	70	13	1.0	5	5.0	1.445	68.7	0	2.0	3.352	18.7	3.44	59.3	
4	70	13	1.0	5	5.0	1.477	65.7	0	2.0	3.284	14.7	2.28	58.1	
5	80	13	1.0	5	5.0	1.446	60.7	0	2.0	3.652	27.2	2.30	64.6	
6	80	13	1.0	5	5.0	1.448	66.7	0	2.0	3.852	27.2	2.21	68.2	
7	90	13	1.0	5	5.0	1.460	46.7	0	2.0	3.966	18.7	3.44	70.2	
8	90	13	1.0	5	5.0	1.477	54.7	0	2.0	3.666	14.7	3.08	64.9	
1	60	15	1.0	5	5.0	1.453	14.7	5.71	2.0	3.084	15.7	71.15	54.6	
2	60	15	1.0	5	5.0	1.435	5.7	5.40	2.0	2.084	46.7	45.61	36.9	
3	70	15	1.0	5	5.0	1.450	15.7	2.75	2.0	3.600	61.7	4.37	63.7	
4	70	15	1.0	5	5.0	1.443	14.7	2.02	2.0	3.634	57.7	2.86	64.3	
5	80	15	1.0	5	5.0	1.451	24.7	2.43	2.0	4.000	63.7	2.60	70.8	
6	80	15	1.0	5	5.0	1.454	22.7	2.12	2.0	4.016	63.2	2.48	71.1	
7	90	15	1.0	5	5.0	1.465	17.7	2.70	2.0	4.084	49.7	4.57	72.3	
8	90	15	1.0	5	5.0	1.488	17.7	2.48	2.0	3.752	62.7	3.55	66.4	

2.2 Effect of Various Additives on the Plate Behavior

2.2.1 Cobalt Additive to the positive plate.

Positive plates containing various amounts of cobalt hydroxide were prepared for use in the initial screening experiments. Several solutions containing various concentrations of cobalt nitrate and nickel nitrate were prepared. Porous sintered nickel plaques were vacuum impregnated in these solutions and then cathodically converted in 30% KOH to the corresponding insoluble hydroxides in the porous plaque structure. All the nitrate was completely reduced to ammonia, and the resulting plates were washed and dried. The total capacity of the plates with 7.5% and 15% cobalt was 0.203 and 0.232 AH respectively. The electrode size was 4 in^2. These plates were tested for electrochemical performance before and after heat sterilization in flooded, plastic cells using Hg/HgO reference electrodes. Negative plates with approximately 10 times the capacity of the test electrodes were used as counter electrodes to avoid complication by any possible H_2 evolution at the counter electrodes during charge. Oxygen evolved during the charge cycle was collected and measured at known ambient temperature and pressure to obtain the equivalent coulombs used in the gas evolution. "Oxygen-free capacity" was obtained by noting the total coulombs passed before any bubbles of oxygen appeared as indicated by an electronic instrument specially designed to indicate the initiation of gas evolution. Delivered capacity was determined from time potential plot with Hg/HgO reference electrode. Electrochemical data for positive plates with and without cobalt addition, both prior to and after heat sterilization are given in Table III. The ratio "O_2-free cap./delivered cap." is a measure of charge acceptance efficiency. Coulombic efficiency reported in the last column of Table III is an empirical quantity and is obtained by adding delivered capacity and AH equivalent to total gas evolved and dividing this quantity by the total charge input.

It will be noted that addition of cobalt improves the charge acceptance of the positive plate as indicated by relatively high ratio of oxygen free capacity to delivered capacity before and after sterilization. However, after sterilization the absolute values of oxygen free capacity and delivered capacity are drastically reduced for the plates containing cobalt. This reduction of oxygen-free capacity is accompanied by an increased polarization of the sterilized positive plate. Physico-chemical characterization of the unsterilized and sterilized positive plates with and without cobalt additives is now underway to determine the causes for the reduction in the oxygen-free capacity due to sterilization. These include BET surface area measurement, pore size distribution, x-ray diffraction, differential thermal analysis (DTA) and thermogravimetric analysis (TGA). Some DTA plots are given in Figures 1 to 3. Figure 1 shows the changes in the DTA curve of the positive plate after sterilization. Figure 2 shows DTA curve for the control sample not undergoing sterilization. The view that the changes in the positive plate during sterilization are probably

TABLE III

EFFECT OF COBALT ADDITION ON THE PERFORMANCE OF POSITIVE PLATE IN FLOODED PLASTIC CELL
Hg/HgO Reference; Positive Limited Cell, 75°F

PRE-STERILIZATION PERFORMANCE DATA

Plate No.	Cycle No.	Plate Add. %	Charge Rate ma	Charge Time Hrs	AH Input	O_2-Free Capacity AH	O_2 Evolved cc	O_2 Evolved AH	Discharge Rate ma	Delivered Capacity AH	O_2-Free AH / Del. AH %	Coulombic Balance %
S	2	Control	260	7.5	1.95	0.317	122.4	0.546	650	1.30	24.3	94.8
S	3	Control	260	7.5	1.95	0.273	127.9	0.568	650	1.20	22.8	90.8
1	3	7.5	200	2.0	0.400	0.201	15.8	0.147	116	0.234	85.8	95.3
	4	7.5	50	6.0	0.300	0.195	1.8	0.017	116	0.261	74.7	92.7
2	3	15.0	200	2.0	0.400	0.201	18.4	0.081	116	0.237	84.8	79.5
	4	15.0	50	6.0	0.300	0.195	4.4	0.019	116	0.267	73.0	95.5

POST-STERILIZATION PERFORMANCE DATA

Plate No.	Cycle No.	Plate Add. %	Charge Rate ma	Charge Time Hrs	AH Input	O_2-Free Capacity AH	O_2 Evolved cc	O_2 Evolved AH	Discharge Rate ma	Delivered Capacity AH	O_2-Free AH / Del. AH %	Coulombic Balance %
S	1	Control	260	7.1	1.85	0.104	107.4	0.476	650	0.725	14.3	65.0
	2	Control	260	7.0	1.82	0.182	157.8	0.697	650	0.865	21.0	86.0
1	1	7.5	46	7.0	0.322	0.046	19.0	0.084	116	0.077	59.7	50.0
	2	7.5	46	6.8	0.313	0.067	29.5	0.131	116	0.083	80.7	68.4
	3	7.5	46	6.8	0.313	0.071	25.6	0.111	116	0.106	67.0	70.0
	4	7.5	46	6.0	0.276	0.100	16.8	0.074	116	0.095	105.0	61.2
	5	7.5	46	5.8	0.267	0.104	-	-	116	0.112	92.9	-
2	1	15.0	46	7.0	0.322	0.020	28.8	0.128	116	0.054	37.0	56.5
	2	15.0	46	6.8	0.313	0.051	34.0	0.150	116	0.058	87.9	67.2
	3	15.0	46	6.8	0.313	0.058	31.4	0.136	116	0.079	73.4	69.0
	4	15.0	46	6.0	0.276	0.079	24.4	0.108	116	0.066	120.0	63.0
	5	15.0	46	6.8	0.267	0.063	30.2	0.125	116	0.083	75.9	78.3

not permanent is supported by (a) the shape of the DTA curve for the sterilized and cycled plate (Fig. 3) which is similar to the shape of the cycled unsterilized plate (Fig. 2), and (b) the partial recovery of the oxygen-free capacity observed during post-sterilization cycling.

Cobalt is a standard additive to the positive plate of sealed and vented Ni-Cd cells manufactured by several companies. However, its exact function or role is not well understood. Further work is required to clarify its beneficial effect, if any, on the performance of sterilized nickel-cadmium cells.

2.2.2 Indium Hydroxide and Thallium Hydroxide Additives to the Negative Plates.

Standard cadmium nitrate solutions containing definite amounts of indium nitrate or thallium nitrate were used for this purpose. Vacuum impregnation of the porous sintered plaque by these salts followed by cathodic conversion in KOH to form the respective hydroxides was carried out using the standard procedure used for control negative plates. The following solutions were used for these plates:

	Solution	AH Capacity
1.	4.45 molar $Cd(NO_3)_2$ plus 0.05 molar $TlNO_3$	0.56
2.	4.45 molar $Cd(NO_3)_2$ plus 0.005 molar $TlNO_3$	0.57
3.	4.45 molar $Cd(NO_3)_2$ plus 0.05 molar $In(NO_3)_2$	0.192
4.	4.45 molar $Cd(NO_3)_2$ plus 0.005 molar $In(NO_3)_2$	0.18

The capacity values are based upon analytical determination of Cd plus $Cd(OH)_2$ and does not include any contribution by indium or thallium salts. The effect of these salts will be to increase the actual capacities above the values listed on the previous page. The electrochemical behavior was determined in flooded plastic cells similar to those used for positive plates containing cobalt. The results are given in Tables IV and V.

The addition of indium to the negative plate does not appear to have any beneficial effect on the performance of the plate before or after sterilization as shown by the data in Table IV. Addition of indium generally reduces the hydrogen free capacity. The delivered capacity following heat sterilization is also significantly reduced. On the basis of these facts no further work with indium additive is planned or contemplated.

Addition of thallium hydroxide apparently increases the hydrogen free capacity prior to sterilization, although the control plate, without any thallium also gave relatively high hydrogen free capacity on first cycle. This hydrogen free capacity for control cells dropped on second and third cycle while the plates containing thallium hydrogen free capacity increased on second cycle. More definitive work with thallium addition is needed before final conclusions can be made.

TABLE IV

EFFECT OF INDIUM ADDITION ON THE PERFORMANCE OF NEGATIVE PLATE IN FLOODED PLASTIC CELL

Hg/HgO Reference; Negative Limited Cell, 75°F

PRE-STERILIZATION PERFORMANCE DATA

Plate No.	Cycle No.	Plate Add. $In(NO_3)_3$	Charge Rate ma	Charge Time Hrs	AH Input	H_2-Free Capacity AH	H_2 Evolved cc	H_2 Evolved AH	Discharge Rate ma	Delivered Capacity AH	$\frac{H_2\text{-Free, AH}}{\text{Del AH}}$ %	Coulombic Balance %
2	1	Control	260	7.2	1.86	1.11	329.6	0.615	650	0.856	130	83.5
2	2	Control	260	7.5	1.95	0.840	456.8	0.852	650	0.870	96.6	88.3
2	3	Control	260	7.5	1.95	0.780	486.2	0.907	650	0.791	98.6	59.0
1	1	0.005M	36	7.5	0.270	0.117	35.0	0.077	90	0.158	74.1	87.0
1	2	0.005M	36	7.5	0.225	0.126	23.6	0.052	90	0.158	79.7	93.3
2	1	0.05M	36	7.5	0.270	0.054	60.6	0.133	90	0.083	65.1	80.0
2	2	0.05M	36	7.5	0.225	0.027	53.6	0.119	90	0.060	50.0	79.5

POST-STERILIZATION PERFORMANCE DATA

2	1	Control	260	7.1	1.846	0.910	326.4	0.685	650	0.890	102.2	85.7
2	2	Control	260	7.0	1.846	0.905	377.6	0.834	650	0.865	104.6	93.3
1	1	0.005M	36	7.0	0.252	0.081	46.5	0.102	90	0.083	97.6	73.4
1	2	0.005M	36	7.0	0.252	0.089	29.8	0.071	90	0.091	97.8	63.8
2	1	0.05M	36	7.0	0.252	0.019	71.0	0.155	90	0.030	63.3	73.4
2	2	0.05M	36	7.0	0.252	0.025	66.6	0.160	90	0.045	55.6	81.3

TABLE V

EFFECT OF THALLIUM ADDITION ON THE PERFORMANCE OF NEGATIVE PLATE IN FLOODED PLASTIC CELL

Hg/HgO Reference; Negative Limited Cell, 75°F

PRE-STERILIZATION PERFORMANCE DATA

Plate No.	Cycle No.	Plate Additive $TlNO_3$	Charge Rate ma	Charge Time Hrs	AH Input	H_2-Free Capacity AH	H_2 Evolved cc	H_2 Evolved AH	Discharge Rate ma	Delivered Capacity AH	H_2-Free, AH / Del., AH %	Coulombic Balance %
2	1	Control	260	7.2	1.86	1.11	329.6	0.695	650	0.856	130.0	83.5
2	2	Control	260	7.5	1.95	0.84	456.8	0.960	650	0.870	97.9	94.0
2	3	Control	260	7.5	1.95	0.78	486.2	1.020	650	0.791	99.0	93.8
1	1	0.005M	115	7.5	0.862	0.604	82.5	0.184	288	0.586	103.0	89.4
1	2	0.005M	115	7.0	0.805	0.605	89.2	0.198	288	0.480	126.0	84.2
2	1	0.05M	115	7.5	0.862	0.575	99.5	0.222	288	0.523	110.0	86.3
2	2	0.05M	115	7.0	0.805	0.611	88.6	0.196	288	0.485	127.0	84.5

POST-STERILIZATION PERFORMANCE DATA

2	1	Control	260	7.1	1.85	0.910	326.4	0.685	650	0.890	102.0	85.7
2	2	Control	260	7.0	1.85	0.905	377.6	0.834	650	0.865	105.0	93.3
1	1	0.005M	115	6.8	0.782	0.316	146.0	0.324	288	0.269	118.0	76.5
1	2	0.005M	115	6.5	0.748	0.251	104.6	0.232	288	0.259	97	65.5
2	1	0.05M	115	6.8	0.782	0.358	134.0	0.298	288	0.374	96	86.8
2	2	0.05M	115	6.5	0.748	0.340	131.6	0.292	288	0.369	92	88.2

3.0 Effect of heat sterilization on KOH absorption by the separators as well as by the positive and negative plates was determined during the report period. The data for KOH absorption by separators 14019 and FT 2140 before and after heat-sterilization at 135°C for 64 hours in 30% KOH or 30% KOH saturated with CdO and with or without positive and negative plates are given in Table VI. Without exception, all samples of the separator material absorbed more KOH after sterilization. The presence of CdO in the electrolyte or the positive and the negative plates in contact with the separator did not have any measurable effect on the amount of KOH absorbed by the separator. These results indicate that the effective porosity of the separator or the capacity of the separator to retain KOH increases due to heat sterilization. This will result in effective lowering of the per cent pore fill by KOH after cell sterilization and may account for increased resistance found in many cells following heat-sterilization treatment. For these reasons, 60 and 70% pore fill levels appear inadequate.

Experimental data on the effect of heat-sterilization of the positive and negative plates on KOH absorption characteristics are given in Table VII. These results show that during the pre-sterilization cycling of the plates, the pick of 30% KOH increases. After sterilization, there appears to be little change in KOH pick. However, on cycling these plates following heat-sterilization, the pick up by the positive plates show an increase where the pick up by negative decreases. These data will be correlated with the pore size distribution data on these plates to arrive at a logical interpretation of these changes.

TABLE VI

ABSORPTION OF KOH SOLUTIONS
BY TWO BEST POLYPROPYLENE SEPARATORS
BEFORE AND AFTER STERILIZATION

TEST MEDIA →	30% KOH	30% KOH & positive plate	30% KOH & negative plate	30% KOH sat'd with CdO	30% KOH sat'd with CdO & positive plate	30% KOH sat'd with CdO & negative plate
Separator	Absorption of KOH Expressed in gmKOHsoln/gm sep.					
FT 2140						
Presterilization	2.83	2.81	2.91	2.99	3.14	2.98
Poststerilization	4.70	5.15	4.33	4.60	4.40	4.85
Increase	1.87	2.34	1.42	1.61	1.26	1.87
14019 #2						
Presterilization	6.30	6.35	6.37	6.59	6.09	6.00
Poststerilization	13.90	17.00	17.10	17.60	9.40	11.00
Increase	7.60	10.65	10.73	11.01	3.31	5.00
14019 #3						
Presterilization	4.81	5.43	5.98	5.02	4.69	5.31
Poststerilization	16.50	16.60	16.40	10.80	12.60	13.20
Increase	11.69	11.17	10.42	5.78	7.91	7.89
Separator	Absorption of KOH Expressed in cc KOH/in^2 separator					
FT 2140						
Presterilization	0.105	0.104	0.108	0.110	0.116	0.110
Poststerilization	0.174	0.190	0.160	0.166	0.163	0.179
Increase	0.069	0.086	0.052	0.060	0.047	0.069
14019 #2						
Presterilization	0.202	0.203	0.204	0.211	0.195	0.192
Poststerilization	0.445	0.544	0.547	0.563	0.301	0.352
Increase	0.243	0.341	0.343	0.352	0.106	0.160
14019 #3						
Presterilization	0.144	0.163	0.179	0.151	0.141	0.159
Poststerilization	0.495	0.498	0.492	0.324	0.378	0.396
Increase	0.351	0.335	0.313	0.173	0.237	0.237

TABLE VII

EFFECT OF STERILIZATION ON THE ABSORPTION OF 30% KOH

BY THE POSITIVE AND THE NEGATIVE PLATES

	Initial	Pre-sterilization Pick up of KOH		Post-sterilization Pick up of KOH	
Plate	Dry wt. as Received	g KOH/g Plate as Received	g KOH/g Plate Cycled	Not Cycled	Cycled After Sterilization
Neg. 1	6.3514	.127	.137	.144	.114
2	6.4344	.119	.143	.149	.110
3	6.2234	.123	.125	.132	.113
4	6.3688	.125	.148	.143	.104
Ave.	6.3445	.123	.138	.142	.110
Pos. 1	5.6122	.118	.139	.136	.200
2	5.9281	.124	.164	.158	.196
3	5.1690	.126	.145	.139	.202
Ave.	5.5697	.123	.149	.144	.199

4.0 DTA and TGA Studies

In order to get a better understanding of the physico-chemical changes occurring particularly in the state of hydration and degree of oxidation of the active material, during thermal sterilization of the Ni-Cd cells, techniques of DTA and TGA are being used as reported earlier.

Typical DTA curves for the positive plates under the following stages are given in Figures 1 to 4.

Figure 1: Positive Plate as Received

Figure 2: Positive Plate Cycled Prior to Sterilization

Figure 3: Positive Plate After Heat-Sterilization

Figure 4: Positive Plate Cycled After Sterilization

Shape of the curve of Figure 4 has been confirmed on other plates. Similar DTA curves for the negative plates in (a) as received (b) cycled (c) after sterilization and (d) cycled after sterilization are given in Figures 5, 6, 7 and 8 respectively.

An interpretation and correlation of these data with other electrochemical and physico-chemical data is now underway and will be reported in the final progress report.

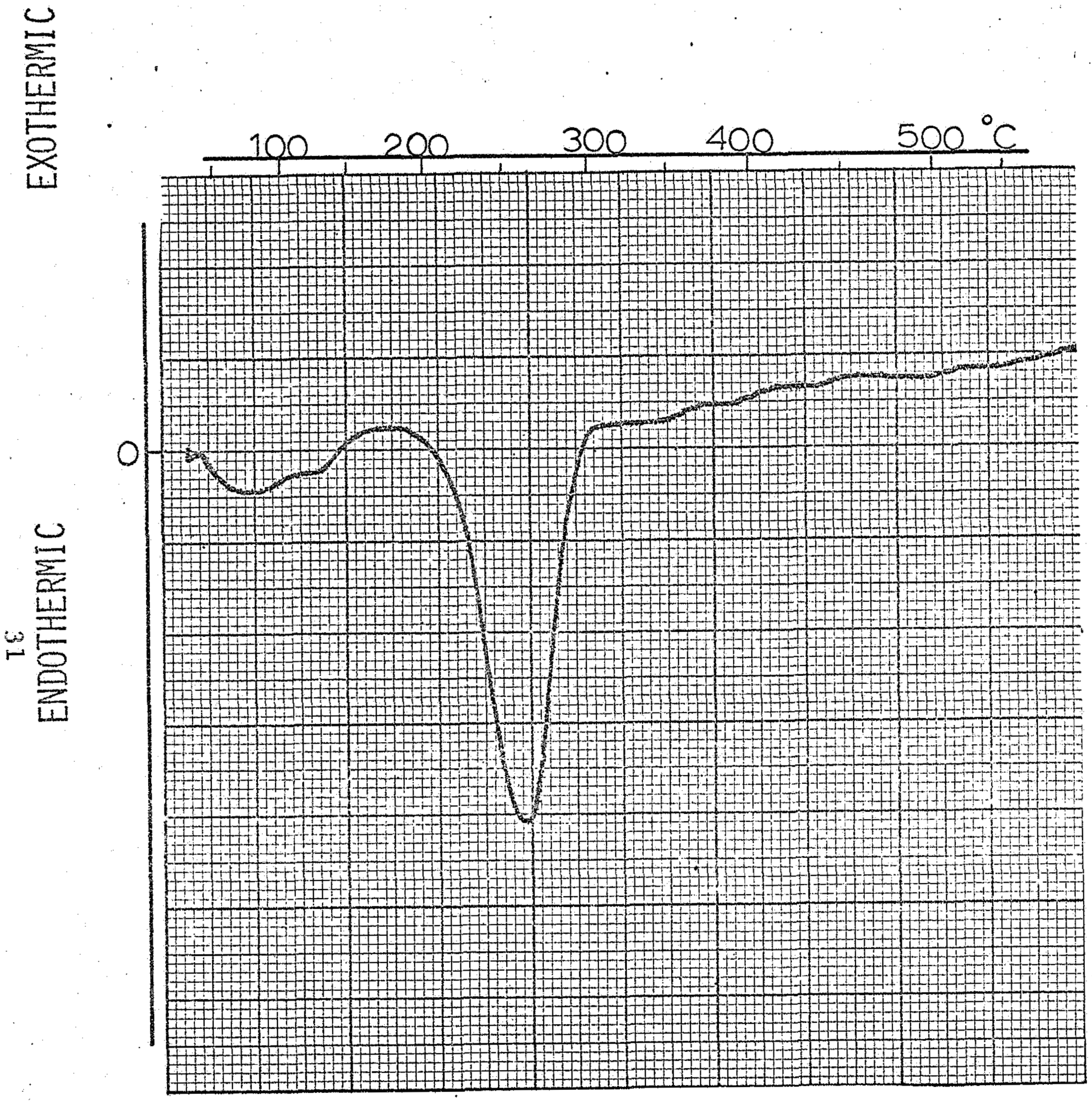


Figure 1

DTA #153

SAMPLE: POSITIVE (AS RECEIVED)

SAMPLE WT.: 10 MG

CONDITIONS: 10°C MIN^{-1}
0.10 SCFM AR

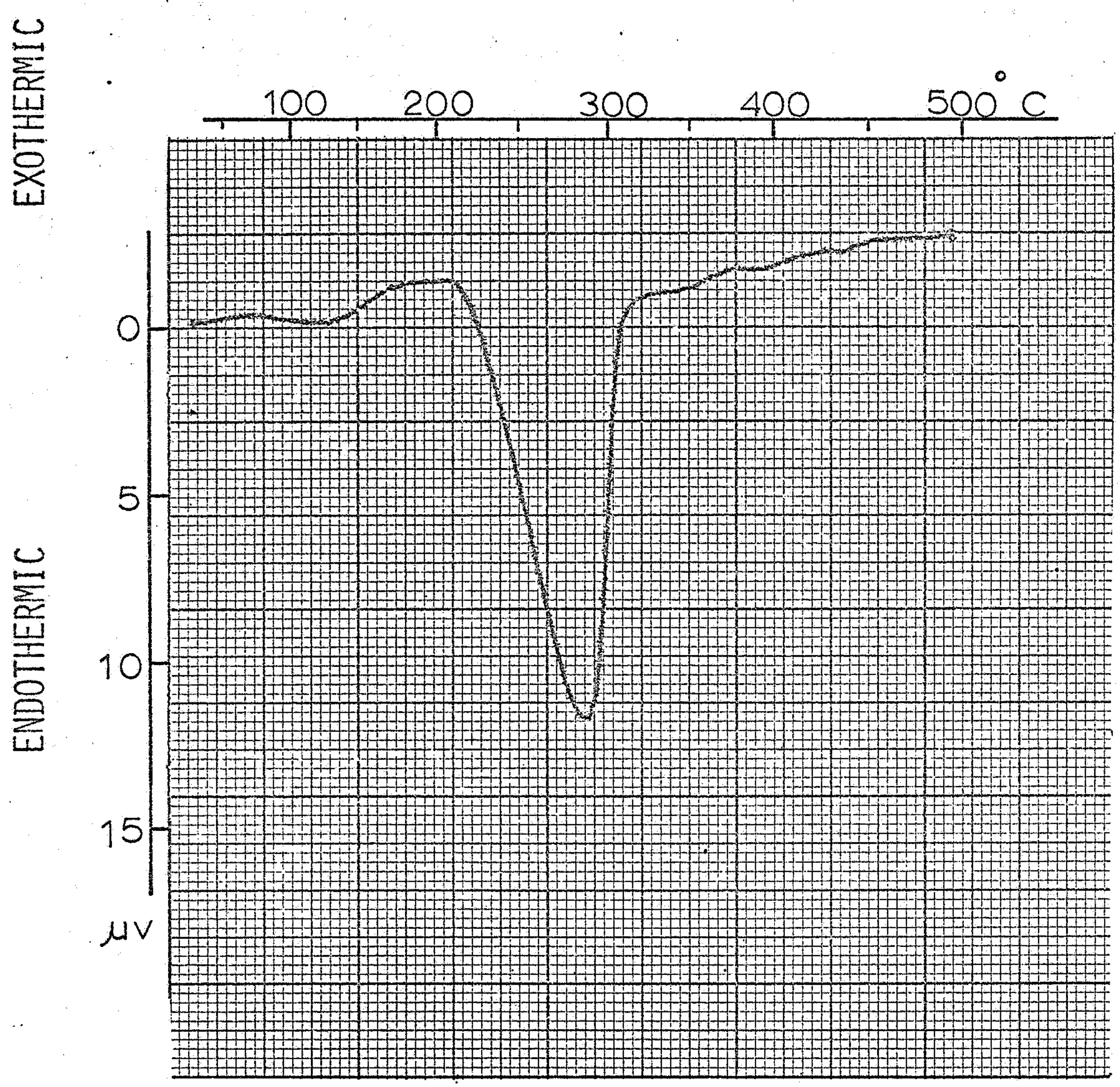
Figure 2

DTA #116

SAMPLE: #68 POSITIVE CYCLED PRIOR TO STERILIZATION

SAMPLE WT.: 8.4 MG

CONDITIONS: 10°C MIN^{-1}
0.10 SCFM AR



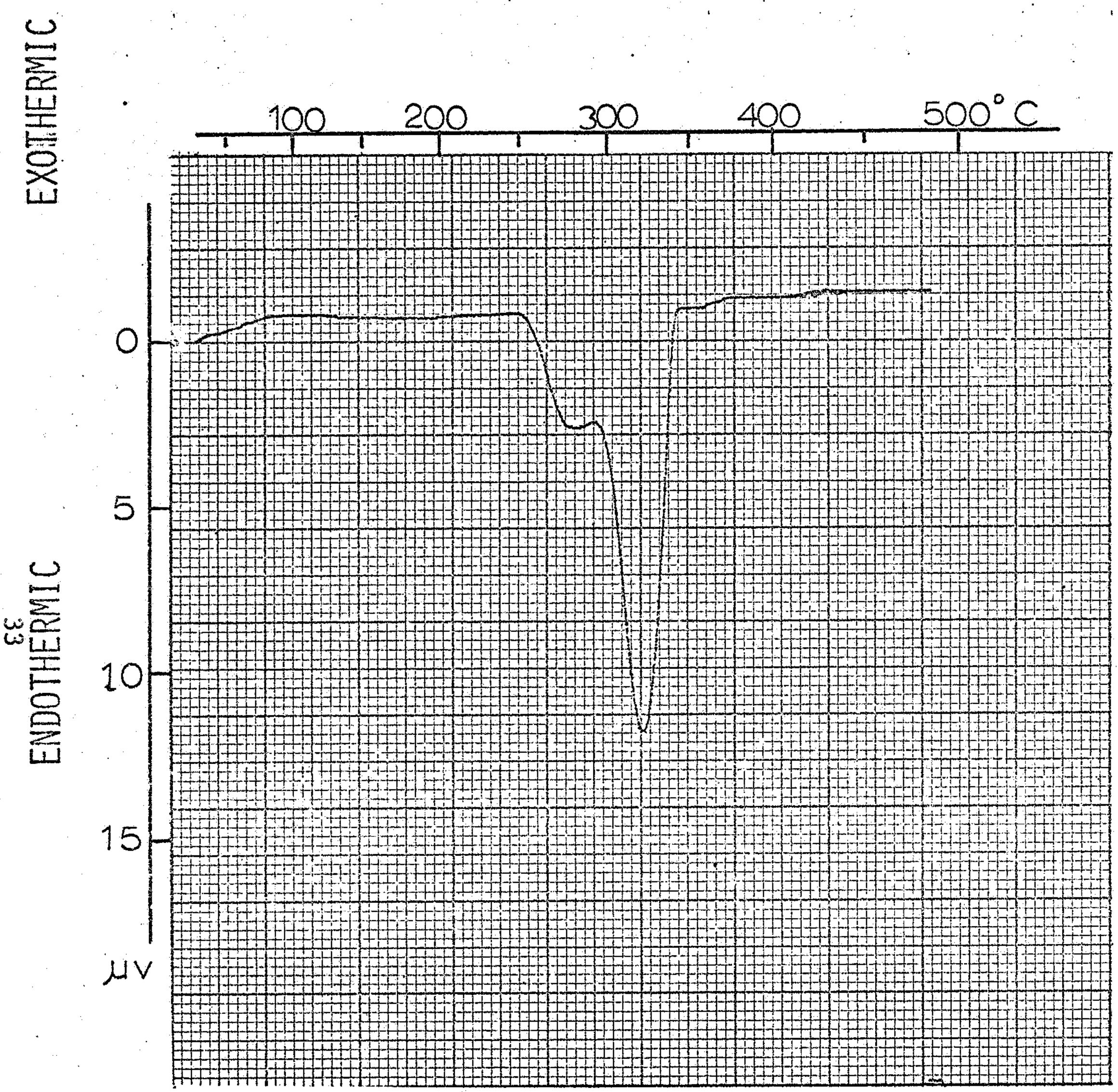


Figure 3

DTA #124

SAMPLE: #68 POSITIVE STERILIZED

SAMPLE WT.: 8.5 MG

CONDITIONS: 10°C MIN^{-1}
0.10 SCFM AR

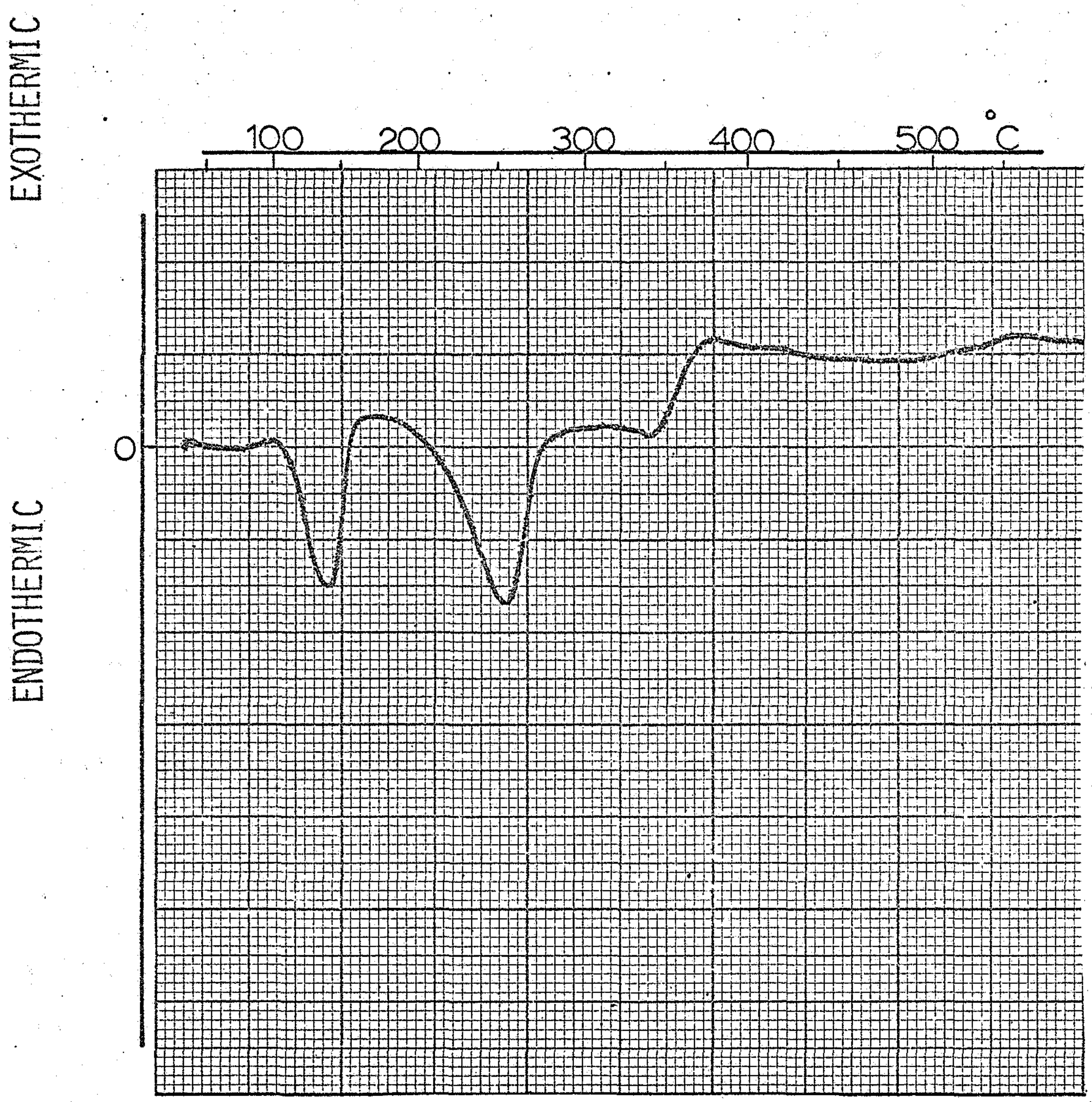


Figure 4

DTA #155

SAMPLE: POSITIVE (STERILIZED AND CYCLED)

SAMPLE WT.: 10 MG

CONDITIONS: 10°C MIN^{-1}
0.10 SCFM AR

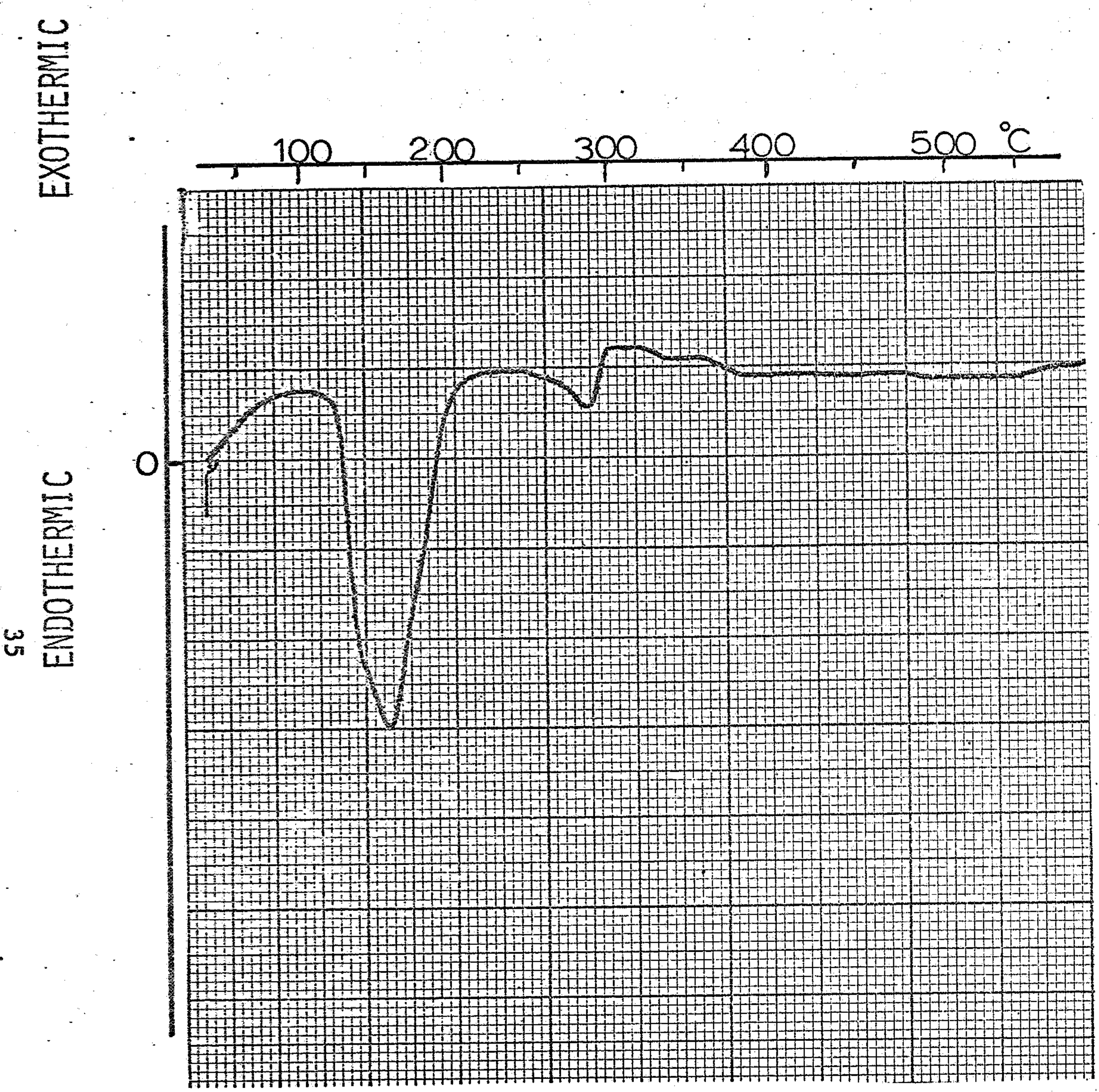


Figure 5

DTA #154

SAMPLE: NEGATIVE (AS RECEIVED) N113

SAMPLE WT.: 10 MG

CONDITIONS: 10°C MIN^{-1}
0.10 SCFM AR

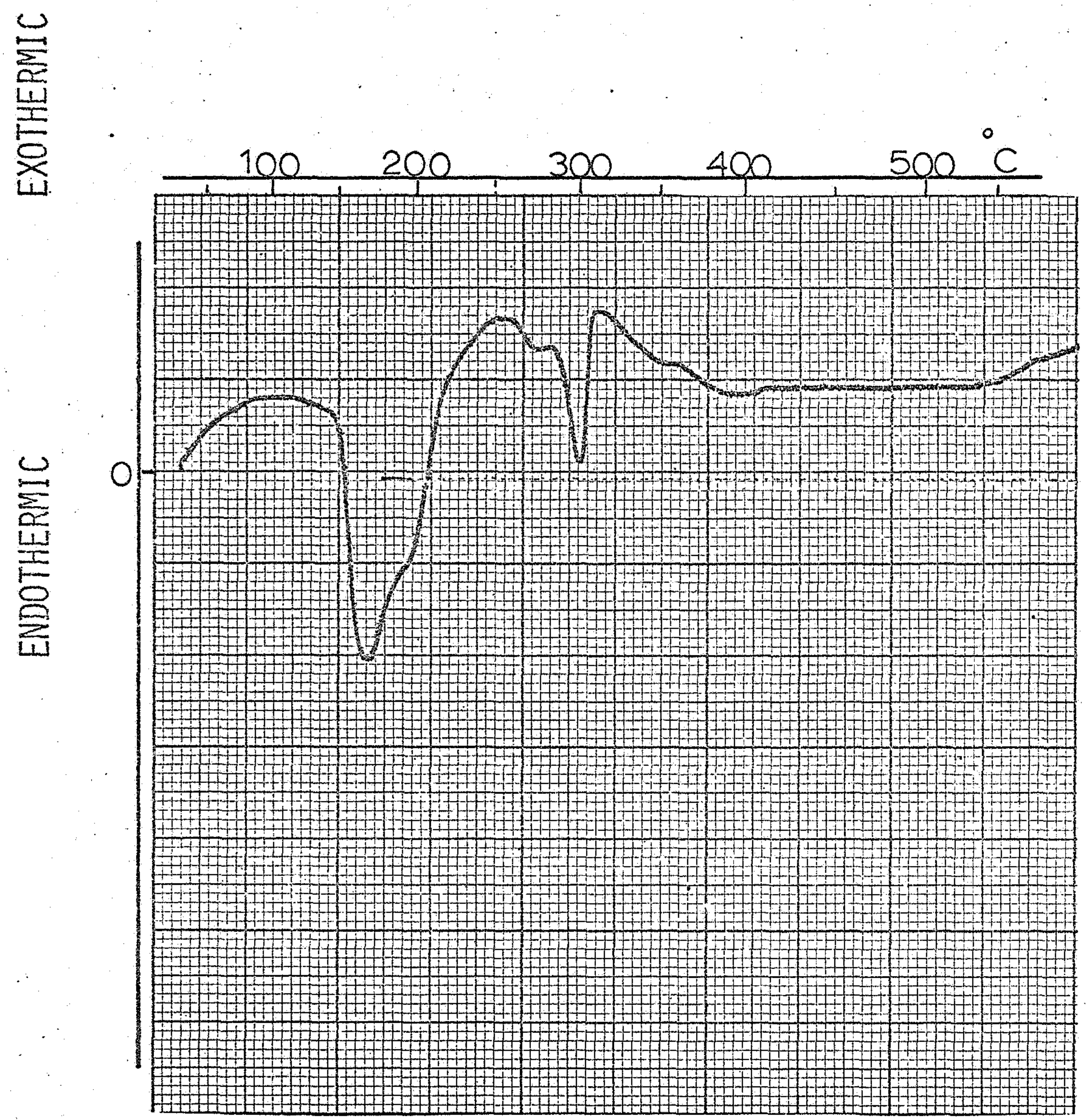


Figure 6

DTA #158

SAMPLE: NEGATIVE (CYCLED)

SAMPLE WT.: 10 MG

CONDITIONS: 10°C MIN^{-1}
0.10 SCFM AR

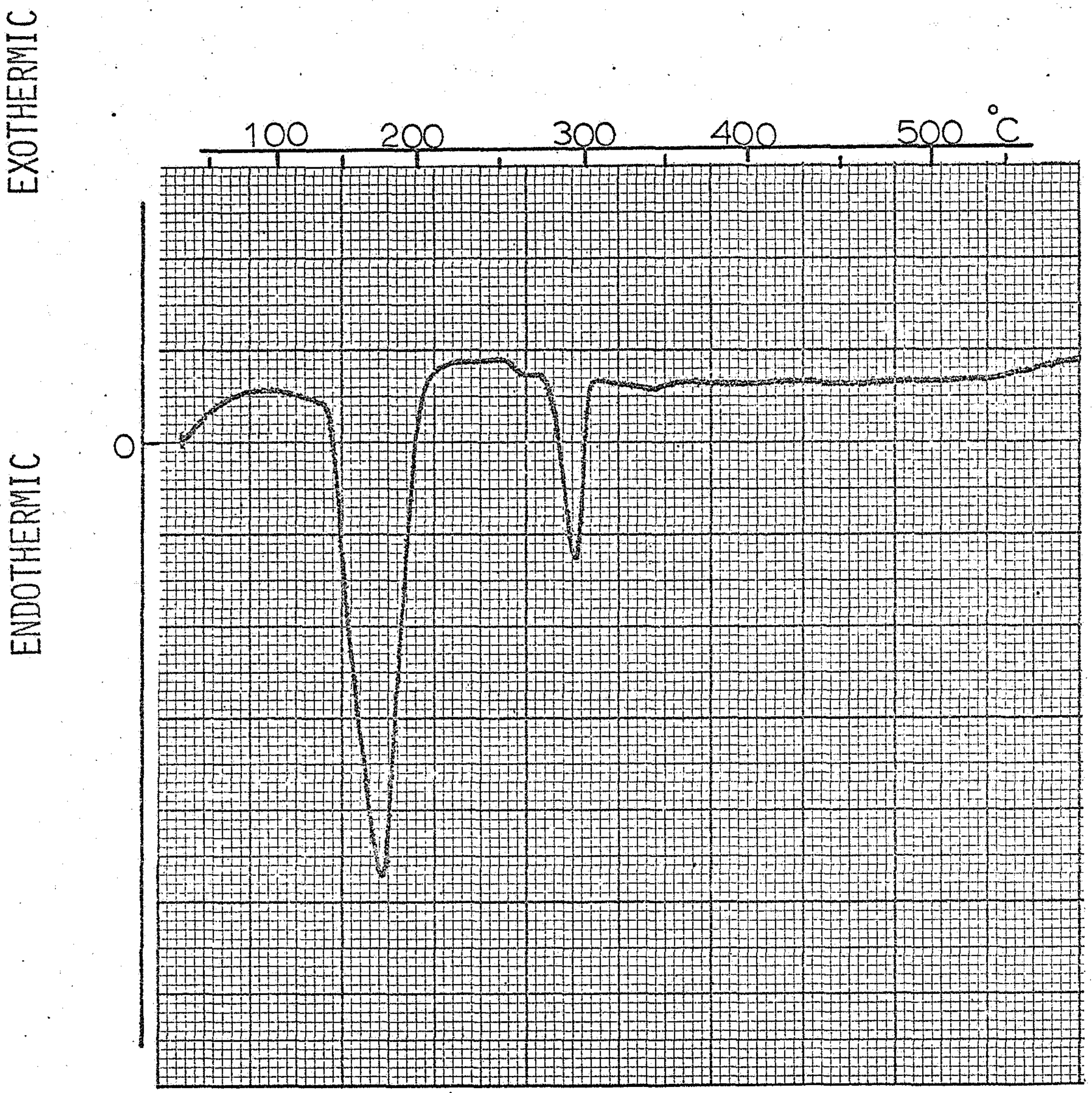
Figure 7

DTA #160

SAMPLE: NEGATIVE (STERILIZED)

SAMPLE WT.: 10 MG

CONDITIONS: 10°C MIN^{-1}
0.10 SCFM AR



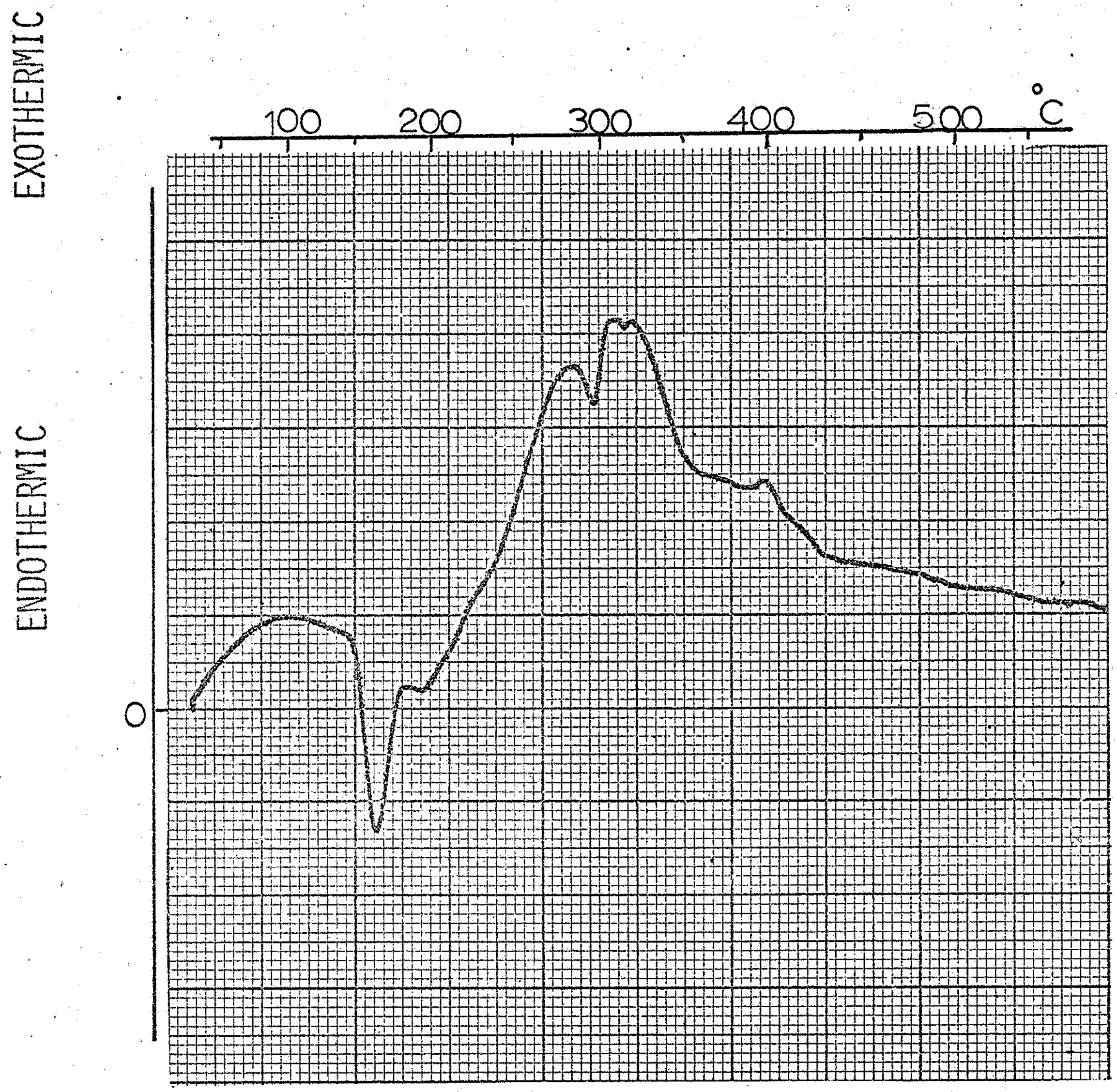


Figure 8

DTA #156

SAMPLE: NEGATIVE (STERILIZED AND CYCLED)

SAMPLE WT.: 10 MG

CONDITIONS: $10°C\ MIN^{-1}$
0.10 SCFM AR