EXIDE MISSILE AND ELECTRONICS DIVISION

ESB INCORPORATED

"World's Largest Producer of Packaged Power"

2510 LOUISBURG ROAD • RALEIGH, NORTH CAROLINA 27604
AREA CODE 919 TELEPHONE 834-[illegible] TWX 919-828-[illegible]

DEVELOPMENT OF IMPROVED
SEALED AG-ZN BATTERY
FOR MARINER 1969

JPL CONTRACT 951927
PART II
NAS7-100

This work was performed for the Jet Propulsion Laboratory, California Institute of Technology, sponsored by the National Aeronautics and Space Administration under Contract NAS7-100.

DEVELOPMENT OF IMPROVED PLATELOCK

FOR

MARINER TYPE SEALED AgO-Zn CELLS

JPL CONTRACT 951927

NAS 7-100

PART II

COVERING PERIOD AUGUST 20, 1968

TO

JANUARY 10, 1969

J. O. NO. 83988

ESB REPORT NO. E-2-69

Prepared By: C. D. Farris
C. D. Farris
Project Engineer

Reviewed By: A. M. Chreitzberg
A. M. Chreitzberg
Ass't. Director of Engineering

Approved By: G. S. Hartman
G. S. Hartman
Director of Engineering

ESB INCORPORATED
EXIDE MISSILE AND ELECTRONICS DIVISION
RALEIGH, NORTH CAROLINA

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

ABSTRACT

Phase B of Jet Propulsion Laboratory Contract 951927 is to develop an improved platelock consisting of an organic compound which will not react with the negative plate in sealed Mariner cells to produce excessive pressure during charged stand at 140°F. Cells containing the previous platelock, Bondmaster 639/CH2, passed the Jet Propulsion Laboratory vibration profile but failed the 14-day charged stand at 140°F due to excessive pressure. ESB investigated eleven different epoxy systems as platelocks singly and the components and additives of each singly in sealed Mariner cells during 14-day charged stand at 140°F. In these tests, curing agents for epoxy systems were responsible for more excessive cell pressure,more ABS cell jar attack and more decay of open circuit voltage than either the mixed epoxy systems or uncured resins. Catalyst #927, manufactured by Furane Plastics Company and Catalyst #11, manufactured by Emerson & Cuming, Inc. produced less cell pressure and were less reactive in contact with ABS cell jars than the other catalysts tested. Furane Plastics epoxy system Epocast 221/927, when used as a platelock in Mariner cells, successfully completed the 14-day 140°F storage test and the Jet Propulsion Laboratory vibration profile. This epoxy system is recommended as a suitable platelock for Mariner cells. Epoxy system LN29A/CH2, modified by PPG Industries from the previous platelock upon request by ESB, appears to be a promising platelock also.

1. INTRODUCTION

The ESB Model 350 design "E" monoblocks (ESB Drawing 257-2013, Revision F) with Bondmaster 639/CH2 epoxy platelock successfully passed Mariner '69 type approval vibration requirements but developed pressures in excess of 40 psig during 14 days charged stand at 140°F and exhibited cell case failure in the unsupported condition. Without the platelock, with a tighter cell pack, and with an Omega bend lead wire configuration to provide slack in plate leads, excessive lead breakage occurred in the vertical vibration plane. JPL elected to retain the epoxy platelock but to find a substitute epoxy for Bondmaster 639/CH2 which would not gas excessively during 14 day charged stand at 140°F and which would embody as many of the other desirable features of the Bondmaster 639/CH2 platelock as possible. Phase B of JPL Contract 951927 was awarded to ESB for the purpose of developing the new platelock.

2. SCREENING TESTS AND MATERIALS EVALUATED

To match the desirable physical and chemical characteristics of the Bondmaster 639/CH2 epoxy system six screening tests were used to evaluate prospective new systems:

- A viscosity-thixotropy test
- A wicking test
- Pot life
- Adhesion to ABS
- Resistance to 40% KOH
- Density

Test procedures and screening test results on 13 systems are given in sections to follow. Material lists and sources are given in Appendix I.

2.1 Viscosity-Thixotropy Test. - A combination test was devised to give a visual measure of the viscosity and thixotropy of a system during the cure cycle in comparison to the Bondmaster 639/CH2 resin-catalyst system. A 3-5 cc portion of mixed epoxy was placed on an 11 mil diameter aluminum wire 300 mesh per in^2 household screen supported horizontally. If the epoxy flowed through the screen its low viscosity could give extensive wicking into the cell plate pack. If the epoxy remained mounted above the screen with partial wetting of the screen forming no sharp edges during cure, then the epoxy correlated the observed physical characteristics of Bondmaster 639/CH2 on the screen test and in Mariner cells.

Epoxies failing the comparative viscosity test were mixed with varying percentages of Cabot Corporation "Cab-O-Sil" to obtain increased viscosity where needed. Additions of 2.0 to 5.5% by weight of mixed resin gave the desired increase in viscosity and thixotropy.

2.2 Wicking Test. - In order to further evaluate wicking characteristics of epoxies into cell packs, miniature dummy plate packs were made from cellophane, viskon, and porous cardboard "plates" inserted into epoxy in cell jars. Plate packs were made with "plate" thickness, the number of layers of 193-PUDO cellophane, and folded viskon retainers comparable in pack tightness to Mariner type cells. Following room temperature cure for 72 hours the 5-plate cell packs were disassembled. Wicking into viskon retainers and plates was not greater than 0.12 inch a distance comparable to Bondmaster 639/CH2 in 50 AH active cells.

2.3 Pot Life Test. - In this test 50 gms of epoxy resin was mixed in a cup with the specified amount of catalyst and additive, using a minimum of 100 stirs with a tongue depressor. A 20 cc sample was transferred to a syringe. Cure time was measured to the viscosity rise point where the mix could not be injected through a 50 mil diameter opening of a 20 cc syringe. Table I lists the resin, catalyst, additive mix ratios and their measured pot lives. A pot life of 30 minutes on this test was considered the minimum allowable time.

2.4 Adhesion to ABS Cell Case Material. - The tensile butt strength in Cycolac T-2502 ABS to Cycolac T-2502 ABS cement joints was measured for each epoxy system. Test specimens were 4.0" x 0.125" x 0.38" or 0.50" cemented and cured 72 hours at room temperature, followed by 2 hours at 150-160°F, a cure condition not adequate for maximum bond strength but similar to cures of Bondmaster 639/CH2 in Mariner '69 monoblocks and not considered detrimental to the dry cell packs. Table II summarizes pull test data, determined on a Dillon Model LW Tensile Tester, and gives the calculated mean butt tensile strengths. Lowest values were observed for Bondmaster LN29A/CH2 and Stycast 1090/CA11 both of which received inadequate cure. Bondmaster 639/CH2 gave the best tensile butt strength - 1360 psi - after room temperature cure and Bondmaster LN29A/CH2 gave the best tensile butt strength - 2350 psi - after 150-160°F cure.

Test bars were molded simultaneously with the 3-cell monoblock cases - one bar per case. A random sample of 17 test bars, molded simultaneously with lot 1252 cell cases, was machined down to 0.375" (3.0" radius) to form tensile test paddles, and pulled to break point on the "Instron" Tensile

Tester at North Carolina State University. Results are reported in Table III. Marbon Chemical gives in their Bulletin 3A a value of 5900 psi for injection molded Cycolac T specimens tested per D-638-64T. Using a normal variation of ±15% limits of 5000 minimum to 6800 maximum apply to this material. The measured tensile strength of Lot 1252 cases was $\bar{X} \pm 3S$ (5,947 ±299) with a range of 5,775 to 6,122 psi and a calculated coefficient of variation ($S/\bar{X} \times 100$) of 1.7%. Thus, the test bars were of normal strength and the cell cases molded simultaneously would be expected to have normal strength also.

2.5 KOH Resistance and Density. - Desirable epoxies were given a KOH resistance test by exposing 2.5 cc pellets of epoxy in 40% KOH solution for 6 hours at 160-170°F followed by a 16-hour exposure at 212°F. Slight changes in weight were detected, Table IV. A second exposure for 2 hours in boiling 40% KOH revealed slight changes in weight but no detectable changes in dimensions.

Density of epoxies was determined in duplicate by measurement of the volume of water displaced by 2.5 cc pellets of epoxy of known weight, Table V.

3. EXPERIMENTAL CELL DESIGN AND TEST METHOD

Mariner '69 3-cell monoblocks were modified for testing each platelock in live 50 AH sealed AgO/KOH/Zn cells. The outer cell compartments were equipped with production type cell packs, a test platelock system, and a pressure vacuum gage calibrated to read 30 inch Hg to zero vacuum and zero to 60 psig pressure. The vacant center cell compartment was filled with Plaster of Paris to provide support for the inner cell walls.

Production of each cell followed Mariner '69 procedures exactly except for -

- - substitution of the test platelock system or a system component for Bondmaster 639/CH2
- - sealing of pressure gage assembly into activation port
- - substitution of Isochem 811B/A epoxy for the production type cell case sealant.

Table VI summarizes the eleven epoxy platelock systems, the eight catalysts and resins, and two additives tested in duplicate in the 50 AH cells. Weight per cell for each of the components are based on 6.0 cc platelock per cell, the densities of Table V and weight ratios of Table I. Each component was insoluble in 45% KOH except "Cab-O-Sil" which was found to be soluble up to 10% additions by weight. Each component was added to the bottom of the empty cell jar - liquids and pastes from a 12 cc syringe and solids by simple transfer - just before insertion of the cell pack. A period of 15 days of production was required from time of addition of the test component to final cell seal after charge plus four days for scheduling of a production oven. During this period the additives were in contact with the cell case and the dry plate pack 12 days, the wet uncharged plate pack 1 day, and the wet-charged plate pack 6 days before the 14 day exposure at 140°F. During this period the only change in cell performance observed was a 117 mv increase in end of formation charge voltage for cells contaminated with pure amine catalyst Hysol H2-3404.

For the charged stand test at 140°F the 30 monoblocks with pressure gages in each of the 60 cells were randomly placed on the shelves of a "Hotpack Constant Temperature Room," a walk-in oven manufactured by the Electric Hotpack Company, Inc. Table VII shows the arrangement of the monoblocks and the temperature variation monitored with thermocouples at varying locations.

Thermo-couples were hung in air and were wedged between the bottom of the monoblocks and the aluminum shelves in the oven. On the eighth day of exposure wooden insulating blocks were inserted between the monoblocks and the metal shelves. Temperature variation at the 140°F setting was 4°F between shelf and air and 8°F from shelf-to-shelf and side-to-side within the oven.

4. CELL EXPOSURE 14 DAYS AT 140°F - EFFECT OF EPOXY COMPONENTS

Oven temperature was adjusted to 140°F ±5°F using a calibrated dial thermometer laying on the left hand lower shelf among the monoblocks and held within this range for 14 days. Cell voltages and pressures were monitored daily with visual observations of carbonation at leakage sites to confirm abrupt pressure decreases. Pressure rise curves are shown for the three classes of materials tested:

- Complete plate-lock system of epoxy resin, curing agent, and additive - Figure 1, 1a, Table VIII.
- Curing agent - Figure 2, Table IX
- Epoxy resin or additive - Figure 3, 3a, Table X

The tabulated and plotted data is the mean of two cell pressures in each case. Cells with the eleven epoxy platelock systems rose to maximum pressures of 23-33 psig in 8 days at 140°F, with the control cells in the center of the distribution. The curing agents (primary aliphatic amines and aromatic amines) as a class exhibited accelerated pressure rise curves, interacted with ABS cell cases, and caused case failure with leakage in 16 of 16 cells by the seventh day at 140°F. The resins as a class reached maximum pressures of 23-34 psig by the ninth day at 140°F with one jar failure with Stycast 1090 after 8 days exposure. Two additives - Cab-O-Sil and RD-4078 are shown in the same figure with the resins. The reactive diluent RD-4078 gave low pressure but interacted with the ABS case material cracking one of two cases on the 8th day. Cab-O-Sil, a viscosity modifier, gave the greatest pressure - 41 psig - on the 9th day.

During the 14 day exposure cell open-circuit voltages were monitored daily. Table XI summarizes the voltage losses observed for the cells contaminated with catalysts where greatest changes were observed. After the 14 day test each cell was repaired for leakage, charged at 1.85 amps to 2.1 volts sealed and then discharged at 10 amps to 1.40 volts. Top-off charge and discharge capacities are shown in Table XI. Nominal 50 AH performance was obtained in 16 of 16 cells with a range of 52.8 - 56.7 AH out after a formation charge input of 56.4 AH before test (not fully charged) plus 12.3 - 16.9 AH top-off after the test. Cells contaminated with Isochem Catalyst 811A gave the lowest open-circuit voltage during the stand test - 1.78 volts - and the highest top-off charge inputs.

5. VIBRATION TESTS AT JPL

Ten of eleven monoblocks with the new platelock systems were sealed after removing the pressure gage assemblies and shipped to the Jet Propulsion

Laboratory for vibration tests to Mariner '69 specifications. The cells were subjected to sinusoidal vibration in the frequency range 600 to 2000 to 600 Hz at one octave per minute frequency change rate and at a level of 40 g rms and in the critical Z axis. Table XII gives open-circuit voltages, load voltages at 20 amps before and after vibration. All cells passed this test. Loaded voltages increased 5 to 17 mv as a result of the vibration frictional heat but no lead breakage always shown by voltage decreases was apparent. These cells are now on cycling routines at JPL.

6. EFFECT OF SPECIAL EPOXY AND CASE CEMENTS ON PRESSURE

Results from the 60 cell exposure test suggested that Cab-O-Sil should be replaced by other viscosity modifiers and that the new ABS cell case cement, common to all cells, might be a cause of pressures being higher by 10 psi than expected from Mariner 64 and 67 cell data. Pittsburgh Plate Glass had also submitted a new Bondmaster LN29A formulation thought to be superior to Bondmaster 639. Since a reserve of 8 plate packs was available, the experiment below was undertaken to provide an answer to the cement question, a first look at the new resin, and the possibility of substituting an insoluble silica (Eccospheres) for a soluble silica (Cab-O-Sil) as a modifier.

Epoxy System Or Cell Contaminant	Sample Number	Subcover Sealing Cement
None - Control	1	Catalyzed C-11 Polystyrene
	2	Catalyzed ABS
PPG LN29A/CH2 100:8	1	C-11
	2	ABS
Isochem 811B/811A Eccospheres 100:25 : 9.8	1	C-11
	2	ABS
PPG LN29 Resin	1	C-11
PPG 639 Resin	1	ABS

Production techniques and contaminating techniques were identical to first 60 cells except for change in cement for subcovers (first and primary seal). The 14-day stand at 140°F was accomplished in a much smaller Thelco forced draft oven having an API controller (±2°F). Table XIII and Figure 4 gives pressure rise data. The rate of rise in pressure for all 8 cells is greater than the previous 60 cells tested because the charged wet stand between end of formation charge and 140°F exposure was 1 day vs 5 days for the previous 60 test cells and the formation charge was 1.0 AH greater.

A comparison of the three cells with C-11 catalyzed cement vs the three cells with ABS catalyzed cement gave a mean 17 psig higher pressure for the C-11 cement. Correcting for this effect the mean pressure rise during the first two days of test for cells with the new PPG LN29A/CH2 system is the same as the control and 4 psig lower than the Isochem 811B/A system having Eccospheres. No ABS jars crazed, but 4 of 8 test cells leaked above 35 psig around the negative plate lead wires, a leakage mechanism also noted in the first exposure test. In the 60-cell test 34 of 40 cells leaked KOH around the negative plate lead wires.

7. EFFECT OF LEAD INSULATION ON CELL PRESSURES

Control cells with no platelock exhibited higher pressures than anticipated from experience with Mariner '64 and '67 cells. Recent data[1] from tests on wet heat sterilizable sealed cells has shown that the heat shrinkable polyolefin tubing used on the test cells can contribute to cell pressure when wet cells are subjected to heat treatment in the temperature range 140 to 275°F. This tubing has a flame retardant and other modifiers which only when heated vaporize and accelerate the rate of hydrogen evolution from negative plates. Replacement of this tubing by heat shrinkable tubing without the gas inducing additives has resolved this additional cause of gassing.

8. CONCLUSIONS AND RECOMMENDATIONS

- \- The cause of the high pressure problem has been identified to be amine in the epoxy platelock systems studied.
- \- Room temperature cure amine curing agents chemically craze ABS cell jars giving jar failure at low pressures.
- \- The most inert epoxy resin platelock system found, providing vibration support at 40 g rms in 50 AH Mariner type plates and giving no cell case damage during 14 days at 140°F, was Furane Epocast 221/927.
- \- It is recommended that this system be substituted for the PPG Bondmaster 639/CH2 system for further testing in Mariner cells.
- \- To further reduce pressures during 140°F charged stand it is recommended that a resin rich formulation of 105 parts

(1) Fourth Quarterly Report for 1968, JPL Contract 951296, Task 9, March 24, 1969.

Epocast 221 resin to 8 parts Catalyst 927 be used. This formulation will give adequate bond strength to ABS cases without inducing stress cracking due to differences in thermal coefficients of expansion.

- - The amine curing agents ranked in order of their increasing attack on ABS jars and acceleration of gassing are -

Most Inert	ABS Jar Rupture	Acceleration H_2 Evolved from Negative Plates
↓	927	927
	11	11
	811A	9
	CH2	9816
	H2-3404	H2-3404
	9816	811A
	9	CH2
	TETA	TETA

- - Heat shrinkable polyolefin tubing containing thermal stabilizers and flame retardants and used as a plate lead wire insulation was found to accelerate hydrogen gassing rates of negative plates at 140°F test temperature.

APPENDIX I

List of Epoxies Tested, Manufacturer and Major Uses of the Epoxies

Bondmaster 639 with CH2 or TETA	PPG Industries, Inc. 225 Belleville Ave. Bloomfield, New Jersey	Epoxy adhesive for bonding plastic and for laminating.
Epon 815, 820 & 828 with TETA	Shell Chemical Co. 20575 Center Ridge Rd. Cleveland, Ohio	Epoxy Resins for casting, laminating, filament winding and adhesive applications.
Stycast 1090 with CA9 or 11	Emerson & Cuming, Inc. Canton, Mass.	A low density epoxide casting resin for electronic embedments.
Epocast 221 with 927 or 9816	Furane Plastics, Inc. 16 Spielman Rd. Fairfield, New Jersey	A filled epoxy for encapsulation of electronic components.
Isochembond 811B with 811A	Isochem Resins Cook St. Lincoln, Rhode Island	A filled epoxy for bonding, potting, or encapsulation
Hysol A-4309/RD4078 with CAH2-3404	Hysol Division The Dexter Corp. Franklin St. Olean, New York	Epoxy resin adhesive for bonding common materials including most plastics.

TABLE I

POT LIFE OF EPOXIES AT ROOM TEMPERATURE

Epoxy System	Mix Weight Ratio	Pot Life* Minutes
Bondmaster 639/CH2	100:8	40
Bondmaster 639/TETA	100:10	60
LN29A/CH2	100:8	90
Epon 828/TETA & 2% Cab-O-Sil	100:12:2.2	50
Epon 820/TETA & 2% Cab-O-Sil	100:12:2.2	95
Epon 815/TETA & 5.5% Cab-O-Sil	100:12:5.6	75
Stycast 1090/CA9	100:8.8	50
Stycast 1090/CA11	100:12	135
Epocast 221/927	100:8	135
Epocast 221/927	105:8	135
Epocast 221/927	110:8	135
Epocast 221/9816 &1% Cab-O-Sil	100:8:1.1	35
Isochembond 811B/811A & 2.5% Cab-O-Sil	100:25:3.1	133
Hysol A4-309/H2-3404/RD4078	100:9:10	45

NOTES: Time epoxy can be ejected from a 50 mil diameter hole in a filled 20 cc syringe using easy thumb pressure.

TABLE II

ABS TO EPOXY TO ABS BUTT TENSILE STRENGTH OF PLATELOCK SYSTEMS[2]

Epoxy System	Force To Break, lbs.[1]	Dimensions At Break, in.	Area of Break, in^2	Tensile Strength, PSI	
				Observed	Mean
Bondmaster 639/CH2	55	.120 x .378	.045	1,210	
100:8	40	.119 x .375	.0446	900	
	55	.119 x .380	.0452	1,220	1,360
	95	.119 x .378	.045	2,110	
Bondmaster 639/TETA	65	.119 x .375	.0447	1,460	
100:10	25	.119 x .375	.0447	560	1,170
	65	.119 x .377	.0449	1,440	
	55	.120 x .377	.0452	1,220	
Epon 828/TETA/Cab-O-Sil	15	.121 x .380	.0460	326	
	20	.120 x .379	.0455	440	440
100:12:2.2	25	.119 x .379	.0451	555	
Epon 820/TETA/Cab-O-Sil	15	.120 x .383	.0460	326	
	15	.119 x .379	.0451	332	500
100:12:2.2	10	.120 x .374	.0448	222	
	50	.119 x .379	.0451	1,110	
Epon 815/TETA/Cab-O-Sil	30	.120 x .378	.0454	660	
	50	.120 x .373	.0447	1,110	1,160
100:12:5.6	85	.120 x .375	.0450	1,890	
	45	.119 x .383	.0455	986	
Stycast 1090/CA9	25	.119 x .373	.0444	560	
100:8.8	20	.120 x .375	.0450	445	420
	15	.119 x .374	.0445	336	
	15	.119 x .375	.0446	336	
Epocast 221/9816	15	.121 x .374	.0453	332	
100:8	25	.119 x .391	.0465	536	430
Isochem 811B/811A/	45	.119 x .377	.0449	1,000	
Cab-O-Sil	30	.119 x .377	.0449	667	740
100:25:3.1	25	.119 x .379	.0451	555	
Epocast 221/927[3]	100	.120 x .500	.060	1,670	
(100;8)	115			1,920	1,560
	70			1,170	
	90			1,500	
Bondmaster LN29A/CH2[3]	35	.120 x .500	.060	580	
100:8	180			3,000	2,350
	150			2,500	
	200			3,340	

(1) Dillon Tester, 1/4 inch per minute speed.
(2) Cured for 72 hours at room temperature.
(3) Cured 24 hours at room temperature plus 2 hours at 150-160°F.

TABLE II

ABS TO EPOXY TO ABS BUTT TENSILE STRENGTH OF PLATELOCK SYSTEMS (CONTINUED)

Epoxy System	Force To Break, lbs. (1)	Dimensions At Break, in.	Area of Break, in^2	Tensile Strength, PSI	
				Observed	Mean
Hysol A4309/H2-3404 (2) Reactive Diluent 4078 100:9:10	50	.119 x .374	.0445	1,120	
	45	.120 x .374	.0450	1,000	1,120
	35	.120 x .375	.0450	775	
	70	.119 x .375	.0447	1,570	
Epocast 221/927 (3) Ratio 110:8	80	.120 x .500	.060	1,330	
	40			670	
	30			500	810
	45			750	
Epocast 221/927 (3) Ratio 105:8	35	.120 x .500	.060	580	
	135			2,250	
	65			1,080	1,390
	100			1,670	
Isochem 811B/811A (3) Ratio 100:20	45	.120 x .500	.060	750	
	25			420	
	105			1,750	1,020
	70			1,170	

(1) Dillon Tester, 1/4 inch per minute speed.

(2) Cure: 72 hours at room temperature.

(3) Cure: 24 hours room temperature plus 2 hours 150-160°F.

TABLE III

TENSILE TESTS ON LOT 1252 ABS CELL CASE TEST BARS

Test No.	Jar No.	Force To Break (1) (lbs.)	Thickness At Break (in.)	Break Area (2) (in^2)	Tensile Strength (psi)
1	512	268.5	.121	.0454	5,914
2	533	268.5	.120	.0450	5,967
3	540	271.5	.122	.0458	5,928
4	542	267.5	.120	.0450	5,944
5	544	267.0	.121	.0454	5,881
6	546	272.0	.119	.0446	6,099
7	551	269.0	.121	.0454	5,925
8	562	272.0	.122	.0458	5,939
9	577	268.5	.120	.0450	5,967
10	590	266.0	.122	.0458	5,808
11	606	270.5	.122	.0458	5,906
12	612	275.5	.120	.0450	6,122 max.
13	618	275.5	.120	.0450	6,122
14	623	264.5	.122	.0458	5,775 min.
15	633	271.0	.121	.0454	5,969
16	635	265.0	.121	.0454	5,837
17	640	270.0	.120	.0450	6,000
$\bar{X}$		269.3	.1208	.0453	5,947
S					99.7
± 3S					299
Min. ($\bar{X}$ - 3S)					5,648
Max. ($\bar{X}$ + 3S)					6,246

(1) "Instron" Tensile Tester, North Carolina State University, Raleigh North Carolina.

Test speed 0.2 inch per minute.

Test date 10-22-68.

(2) Specimen width at beak 0.375 inch.

TABLE IV

40% KOH RESISTANCE TESTS

Epoxy System	Step 1 (1) Weight, GM. Before	After	Mg	Percent Change	Step 2 (2) Weight, GM. After	Mg.	Percent Change
Bondmaster 639/CH2	2.9784	2.9721	-6.3	-.21	2.9573	-14.8	-5.0
Bondmaster 639/TETA	3.2946	3.2996	5.0	.15	3.2957	- 3.9	- .18
Epon 828/TETA 2% Cab-O-Sil	2.8772	2.8774	0	0	2.8760	- 1.4	- .05
Epon 820/TETA 2% Cab-O-Sil	2.6730	2.6748	1.8	.07	2.6739	- .9	- .03
Epon 815/TETA 5.5% Cab-O-Sil	2.4872	2.4857	-1.5	-.06	2.4811	- 4.6	- .18
Stycast 1090/CA9	1.9450	1.9424	-2.6	-.13	1.9446	2.2	.11
Stycast 1090/CA11 (3)	1.8853	1.8831	-2.2	-.12	1.8856	2.5	.13
Epocast 221/927 (3) 4% Cab-O-Sil	3.6428	3.6389	-3.9	-.11	3.6401	1.2	.03
Epocast 221/9816 % Cab-O-Sil	3.5093	3.5079	-1.4	-.04	3.5050	-2.9	-.08
Isochem 811B/811A 2.5% Cab-O-Sil	3.3359	3.3202	-15.7	-.47	3.2963	-23.9	-.72
Hysol A4309/H23404	2.6992	2.7040	4.8	.18	2.7043	.3	.11

Cure: 72 hours at room temperature unless otherwise indicated.

(1) Step 1: Exposed in 40% KOH solution for 6 hours at 160-170°F followed by 16 hours at 212°F.

(2) Step 2: Exposed in boiling 40% KOH solution for 2 hours.

(3) Post cured for 2 hours at 150-160°F following room temperature cure for 24 hours.

TABLE V

DENSITY OF CURED EPOXY SYSTEM

Epoxy System	Sample Weight, Grams	Volume cc	Density - gm/cc Observed	Mean
Bondmaster 639/CH2	3.44 3.12	2.50 2.40	1.38 1.30	1.34
Bondmaster 639/TETA	3.19 3.24	2.50 2.60	1.28 1.24	1.26
Epon 828/TETA 2% Cab-O-Sil	2.50 2.88	2.50 2.50	1.00 1.15	1.08
Epon 820/TETA 2% Cab-O-Sil	2.88 2.93	2.00 2.50	1.44 1.17	1.3
Epon 815/TETA 5.5% Cab-O-Sil	2.63 2.65	2.00 2.10	1.31 1.27	1.29
Stycast 1090 Catalyst 9	1.96 1.94	2.50 2.50	.78 .78	.78
Stycast 1090 Catalyst 11	1.93 1.92	2.50 2.50	.77 .77	.77
Epocast 221/927 4% Cab-O-Sil	3.72 4.11	2.50 2.50	1.49 1.64	1.56
Epocast 221/9816 1% Cab-O-Sil	3.59 3.83	2.50 2.50	1.44 1.53	1.48
Isochem 811B/811A 2.5% Cab-O-Sil	3.46 3.47	2.50 2.50	1.38 1.39	1.38
Hysol A4309/H23404/RD4078	2.55 2.65	2.50 2.50	1.02 1.06	1.04

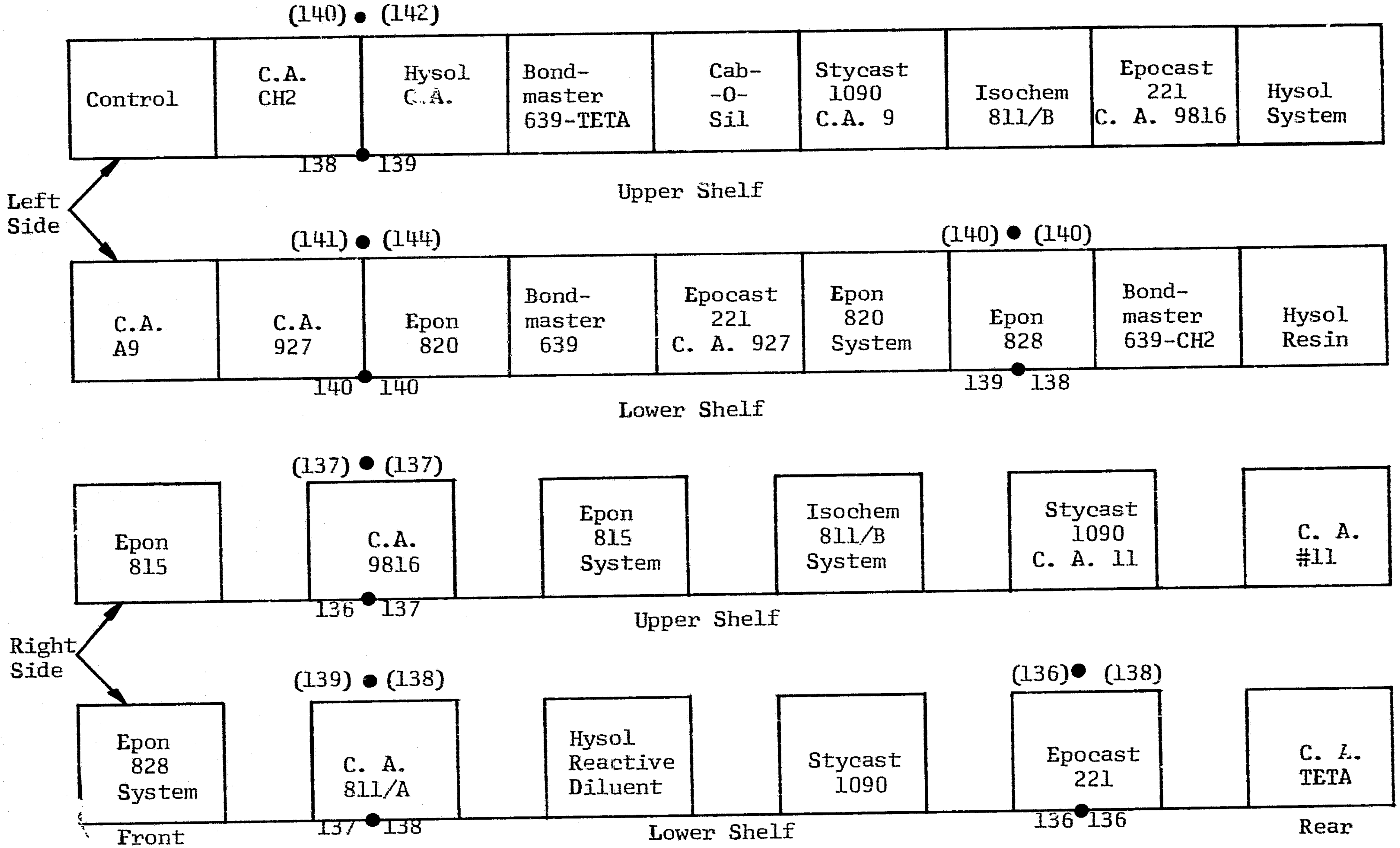
TABLE VI

EPOXY PLATELOCK SYSTEMS AND COMPONENTS IN 50 AH TEST CELLS

System or Component	Number Cells Tested	Weight of Component in Cell-gms Resin	Catalyst	Additive
Resin/Catalyst/Additive/System:				
Bondmaster 639/CH2	2	7.4	.59	--
Bondmaster 639/TETA	2	7.4	.74	--
Epon 828/TETA/Cab-O-Sil	2	5.7	.68	.13
Epon 820/TETA/Cab-O-Sil	2	6.7	.80	.15
Epon 815/TETA/Cab-O-Sil	2	6.8	.82	.42
Stycast 1090/CA9	2	4.3	.39	--
Stycast 1090/CA11	2	4.3	.52	--
Epocast 221/927	2	8.4	.67	--
Epocast 221/9816	2	8.4	.67	--
Isochem 811B/811A/Cab-O-Sil	2	6.5	1.62	.20
Hysol A4309/H23404/RD4078	2	5.2	.47	.57
Control (none)	2			
Resins Alone:				
Bondmaster 639	2	7.4		
Epon 828	2	5.7		
Epon 820	2	6.7		
Epon 815	2	6.8		
Stycast 1090	2	4.3		
Epocast 221	2	8.4		
Isochem 811B	2	6.5		
Hysol A4309	2	5.2		
Catalysts Alone:				
CH_2	2		.60	
TETA	2		.70	
#9	2		.39	
#11	2		.49	
927	2		.67	
9816	2		.64	
811A	2		1.62	
H23404	2		.46	
Additives Alone:				
Cab-O-Sil	2			.42
Reactive Diluent 4078	2			.47
Total Cells: -------------	60			

TABLE VII

ARRANGEMENT OF MONOBLOCKS IN OVEN* AND MONITORED TEMPERATURE VARIATION IN °F



(*) "Hotpack Constant Temperature Room," Model 1270, manufactured by The Electric Hotpack Company. Outside dimensions: 6.6' long x 4.5' wide x 7.5' high.

( ) Temperature taken in air. Other temperatures taken in contact with shelf.

TABLE VIII

CELL PRESSURES DURING 140 ±5°F STORAGE OF MODEL 257 CELLS WITH EPOXY PLATELOCK SYSTEMS

Elapsed Days	Average Cell Pressure, PSIG (1)											
	1	2	3	4	5	6	7	8	9	10	11	12
1	4.0	4.0	4.0	5.8	5.9	4.2	4.6	5.0	4.1	5.0	4.6	2.8
2	7.8	7.0	5.8	8.5	7.5	8.0	6.8	8.8	7.0	7.8	7.5	7.0
3	12.1	13	10	13	11.2	13	10.6	16.5	11.5	12.2	13.6	12.8
4	17.7	20	16.2	18.5	17.7	20.5	17	20.8	16.5	17.5	22.5	19.0
5	20.5	25.5	21.2	22.2	22.5	24.2	21.5	24.0	20.7	20.2	27.2	23.0
6	23.5	31.0	27.5	25.0	29.2	28.2	26.5	28.5	23.5	23.8	33.2	27.5
7	27.0	33.3	29.6	25.9	32.4	29.5	27.5	30.0	25.4	24.5	35.0	28.8
8	24.5	34.0	30.7	26.5	33.2	31.0	27.5	30.5	25.5	25.0	35.0	28.8
9	24.5	34.2	32.0	27.5	34.3	29.5	27.8	31.0	25.7	24.7	35.0	29.0
10	25.2	34.6	31.0	28.0	34.5	29.6	27.6	31.2	26.4	24.3	34.9	29.5
11	24.5	34.0	31.0	28.0	34.0	29.0	27.2	31.2	26.0	24.0	35.0	29.0
13	24.6	33.5	30.4	27.5	34.2	29.1	27.0	31.0	26.0	23.1	34.0	27.8
14	24.6	33.2	29.5	27.5	33.8	28.5	26.2	30.5	24.9	23.4	33.4	27.5

1. Bondmaster 639/CH2
2. Bondmaster 639/TETA
3. Epon 828/TETA with Cab-O-Sil
4. Epon 820/TETA with Cab-O-Sil
5. Epon 815/TETA with Cab-O-Sil
6. Stycast 1090/CA9
7. Stycast 1090/CA11
8. Epocast 221/927
9. Epocast 221/9816
10. Isochembond 811B/811A with Cab-O-Sil
11. Hysol A-4309/H2-3404 with Reactive Diluent 4078
12. None (control)

(1) Sample size of two cells.

TABLE IX

PRESSURES DURING 140 ± 5°F STORAGE MODEL 257 CELLS CONTAMINATED WITH EPOXY CURING AGENTS

Elapsed Days	Average Cell Pressure, PSIG (1)							
	1	2	3	4	5	6	7	8
0	7.2	9.8	3.5	2.5	2.0	4.5	7.5	6.5
1	12.5	12.0 (2)	4.6	6.0	5.0	9.0	14.0	11.1
2	18.5	(3)	10.2 (2)	9.9	8.2	12.2	18.0	15.7
3	26.7		(3)	17.5	14.5	18.3 (2)	23.5	22.0 (2)
4	33.5			27.7	24.6	(3)	30.0	30.0 (2)
5	(3)			34.5	32.0		33.8	36.0 (2)
6				39.0 (2)	39.0 (2)		(3)	(3)
7				(3)	41.5 (2)			
8					(3)			

Code	Curing Agent
1	Catalyst CH_2
2	Catalyst TETA
3	Catalyst 9
4	Catalyst 11
5	Catalyst 927
6	Catalyst 9816
7	Catalyst 811A
8	Catalyst H2-3404

(1) Sample size of two cells.
(2) Sample size of one cell. Other cell leaked.
(3) Both cells leaked.

TABLE X

PRESSURES DURING 140 ±5°F STORAGE MODEL 257
CELLS CONTAMINATED WITH EPOXY RESINS OR ADDITIVES

Elapsed Days	Average Cell Pressure, PSIG (1)									
	1	2	3	4	5	6	7	8	9	10
0	1.5	1.0	1.5	1.5	2.0	2.0	1.5	1.0	2.8	0.5
1	5.0	4.0	4.0	3.5	5.4	5.0	4.1	4.1	5.9	3.1
2	10.8	7.0	6.0	4.5	9.0	8.5	7.1	6.8	9.6	7.0
3	18.1	12.2	9.8	7.0	14.1	13.0	12.6	13.1	16.0	13.5
4	23.6	19.5	15.5	11.0	20.5	18.6	19.0	19.7	25.7	19.1
5	25.8	23.7	19.0	13.4	24.7	22.0	22.8	23.5	32.2	22.0
6	27.0	28.0	23.5	17.2	30.5	25.9	26.5	26.7	37.7	24.5
7	27.2	29.6	25.1	20.0 (2)	32.8	26.8	27.5	27.2	40.0	25.2
8	26.9	29.7	25.4	22.0 (2)	33.8	27.0	27.8	27.2	40.5	24.6
9	26.2	29.7	25.6	23.4 (2)	34.2 (2)	27.0	27.7	27.0	41.0	24.5
10	25.7	29.7	26.0	24.3 (2)	34.3 (2)	27.2	27.6	26.8	40.2	23.8
11	25.0	28.5	26.0	25.5 (2)	34 (2)	26.7	27.5	26.0	39.0	23.5
13	23.6	28.2	26.0	26.7 (2)		26.0	26.5	25.8	37.3	(2)
14	22.9	28.0	25.2	27.0 (2)		26.0	26.2	24.8	35.6	

Code	Resin Type
1	Bondmaster 639
2	Epon 828
3	Epon 820
4	Epon 815
5	Stycast 1090
6	Epocast 221
7	Isochembond 811B
8	Hysol A-4039

Code	Additive
9	Cab-O-Sil
10	RD-4078 Hysol reactive diluent

(1) Sample size of two cells.
(2) One cell developed leak.
(3) Sample size of one cell - other cell leaked.

TABLE XI

VOLTAGES AND CAPACITIES OF CELLS CONTAMINATED WITH CATALYSTS

Test Parameter	Unit	Days at 140°F	Catalyst Type and Cell Serial Number															
			CH2		TETA		9		11		927		9816		811A		H23404	
			1575	1576	1577	1578	1599	1600	1601	1602	1609	1610	1611	1612	1619	1620	1625	1626
Open-Circuit Voltage During Exposure at 140°F (1)	Volts	1	1.86	1.86	1.86	1.86	1.86	1.86	1.86	1.86	1.86	1.86	1.86	1.86	1.86	1.86	1.86	1.86
	Volts	2			Leak	Leak	1.85	Leak					1.86					1.84
	Volts	3					Leak						Leak	1.82				Leak
	Volts	4	1.82	1.82										Leak				
	Volts	5	Leak	Leak					1.82		1.82				1.78	1.78	1.82	
	Volts	6							Leak	1.84	Leak				Leak	Leak	Leak	
	Volts	7								Leak		1.82 Leak						
After Test (2)	Volts		1.819	1.821	1.794	1.786	1.839	1.833	1.839	1.814	1.834	1.834	1.830	1.833	1.772	1.760	1.818	1.780
Top-Off Charge (3) Capacity	AH	-	14.2	15.4	15.0	14.8	13.5	12.3	15.7	15.7	15.7	14.9	13.1	13.7	15.7	16.9	13.9	16.6
Full Discharge (4) Capacity	AH	-	55.5	54.3	54.3	52.8	55.5	54.2	55.5	56.7	56.7	56.7	55.5	55.5	55.5	54.2	54.2	52.8
Leak Site on Cell (5)	-	-	High	Low	Low	Low	High	Low	High	High	High	(6)	High	High	High	(6)	High	(6)

NOTES:
(1) Leak established by a cell pressure drop of 4.5 pounds or more per day.
(2) Taken following seal of leak sites and readjustment of electrolyte.
(3) At 1.85 amps to 2.1 volt/cell unsealed.
(4) At 10.0 amps to 1.40 volts/cell sealed. Formation charge input was 56.4 AH on all cells.
(5) High leak site refers to middle to top of cell; low leak site refers to side of cell near bottom or directly on bottom adjacent liquid catalyst.
(6) No leak found in jar by pressurizing cell at 8 inch Hg pressure submerged in water.

TABLE XII

EFFECTS OF VIBRATION ON TWO CELL MONOBLOCKS(1)

Platelock	Open-Circuit Voltage Before Vib.	Open-Circuit Voltage After Vib.	Open-Circuit Voltage Change MV	Load Voltage @ 20A Before Vib.	Load Voltage @ 20A After Vib.	Load Voltage @ 20A Change MV	Cell S/N
Bondmaster 639/CH2	1.858	1.858	0	1.435	1.446	+11	1579
	1.858	1.857	-1	1.447	1.453	+ 6	1580
Bondmaster 639/TETA	1.858	1.858	0	1.432	1.438	+ 6	1581
	1.858	1.858	0	1.432	1.437	+ 5	1582
828/TETA/Cab-O-Sil	1.858	1.856	-2	1.431	1.443	+12	1585
	1.858	1.857	-1	1.429	1.443	+14	1586
820/TETA/Cab-O-Sil	1.858	1.857	-1	1.434	1.446	+12	1589
	1.858	1.857	-1	1.437	1.445	+ 8	1590
Stycast 1090/CA9	1.858	1.857	-1	1.441	1.452	+11	1603
	1.858	1.857	-1	1.441	1.452	+11	1604
Stycast 1090/CA11	1.858	1.857	-1	1.428	1.439	+11	1605
	1.858	1.857	-1	1.426	1.437	+11	1606
Epocast 221/927	1.858	1.857	-1	1.434	1.443	+ 9	1613
	1.858	1.857	-1	1.443	1.452	+ 9	1614
Epocast 221/9816	1.858	1.857	-1	1.434	1.441	+ 7	1615
	1.858	1.857	-1	1.442	1.454	+12	1616
Isochem 811B/811A/Cab-O-Sil	1.858	1.857	-1	1.436	1.448	+12	1621
	1.858	1.858	0	1.437	1.450	+13	1622
Hysol System	1.858	1.858	0	1.447	1.452	+ 5	1629
	1.858	1.857	-1	1.439	1.456	+17	1630

(1) Dummy middle cell compartment filled with Plaster of Paris.
(2) Vibration profile: 40 "grms" 600 to 2000 to 600 HZ sinusoidal at one octave per minute on the Z axis.

Test Date: 12/3/68.

TABLE XIII

PRESSURES GENERATED DURING 140 ±5°F STORAGE OF MODEL 257 CELLS CONTAMINATED WITH EPOXY PLATELOCKS, RESINS AND CEMENTS

Modification	Cured Platelock		Cured Platelock		None		Resin Only	
Platelock or Resin	LN-29A/CH2 100:8		811B/811A Eccospheres 100:25:9.8		Control		LN29A	Bondmaster 639
Subcover Cement	C-11	ABS	C-11	ABS	C-11	ABS	C-11	ABS
Exposure Time, Days								
0	5.0	7.0	6.0	5.0	4.0	5.0	4.0	6.0
1	19.0	18.0	25.0	14.0	20.0	16.0	21.0	17.0
2	31.0	24.0	40.0	22.0	36.0	23.0	43.0 (1)	29.0 (1)
3	33.0	26.0	45.0	28.0	40.0	27.0	--	--
4	34.0	30.0	49.5	33.0	37.0	31.0	--	--
5	34.0	31.0	3.0	5.0	Leak	33.0	--	--
6	34.0	33.0	Leak	Leak	--	35.0	--	--
7	34.0	33.0	--	--	--	36.0	--	--
8	33.0	33.0	--	--	--	36.0	47.0 (2)	30.0 (2)
9	32.5	33.5	--	--	--	37.0	Leak	33.0
Sum Pressures to 2 days, psi	55	49	71	41	60	44	68	52
Sum by Contaminant, psi	104		112		104		X	X
Pressure Increase C-11 vs ABS cement psi	+6		+30		+16		X	X
Mean Effect of Case Cement	C-11, 17 psi greater than ABS						X	X
Corrected mean Sum 2 Day Pressures (3) psi	44		48		44		51	52

(1) Two-cell monoblock removed from test at 47 psig on cell 1.

(2) Two-cell monoblock replaced into oven on the 7th day.

(3) Projected pressures for all cells cemented with ABS Cement.

FIGURE 1

PRESSURE GENERATED DURING 140 ±5°F STORAGE
OF MODEL 257 MONOBLOCKS WITH DIFFERENT EPOXY PLATELOCKS

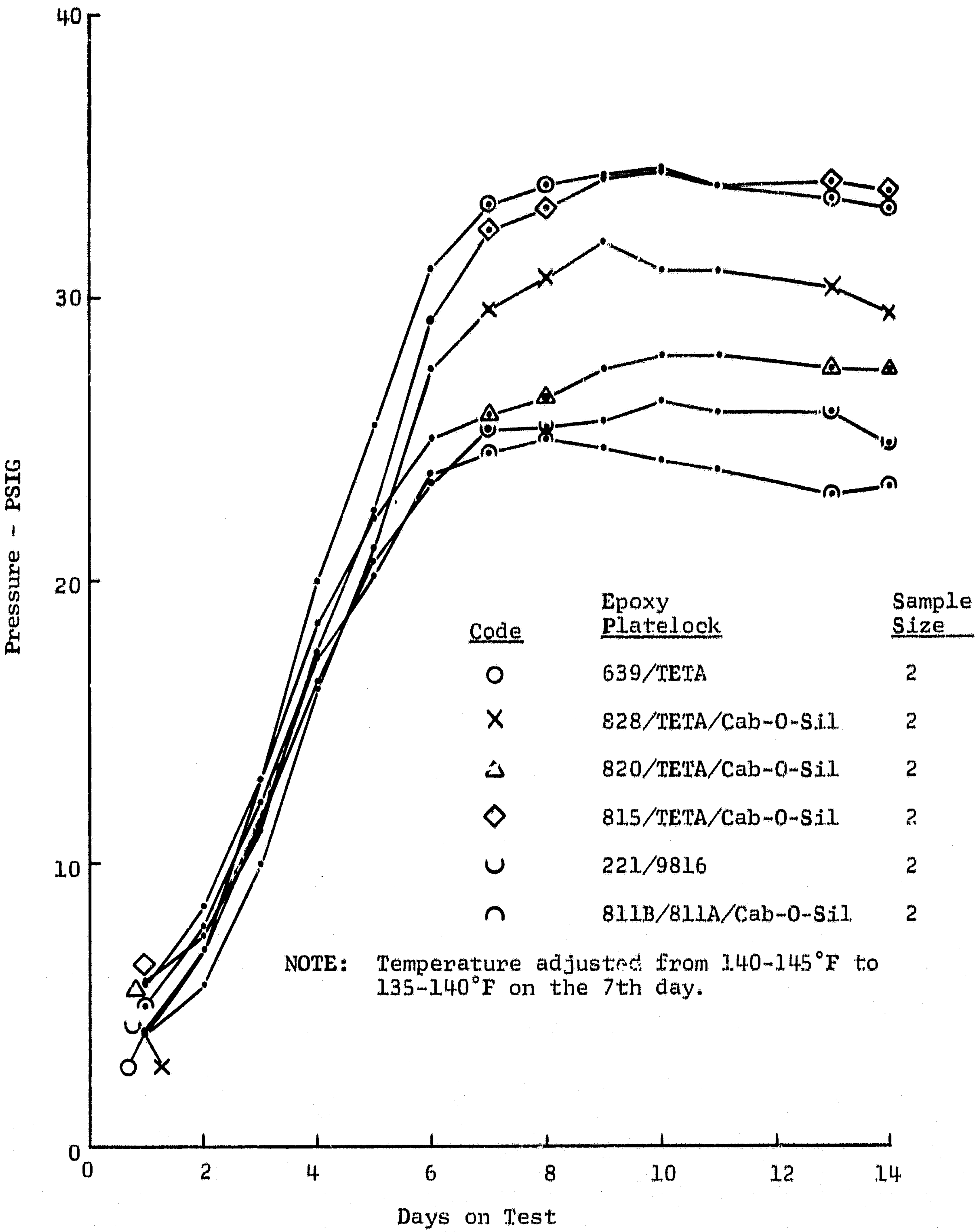


FIGURE 1
(Continued)
PRESSURE GENERATED DURING 140 ±5°F STORAGE
OF MODEL 257 MONOBLOCKS WITH DIFFERENT EPOXY PLATELOCKS

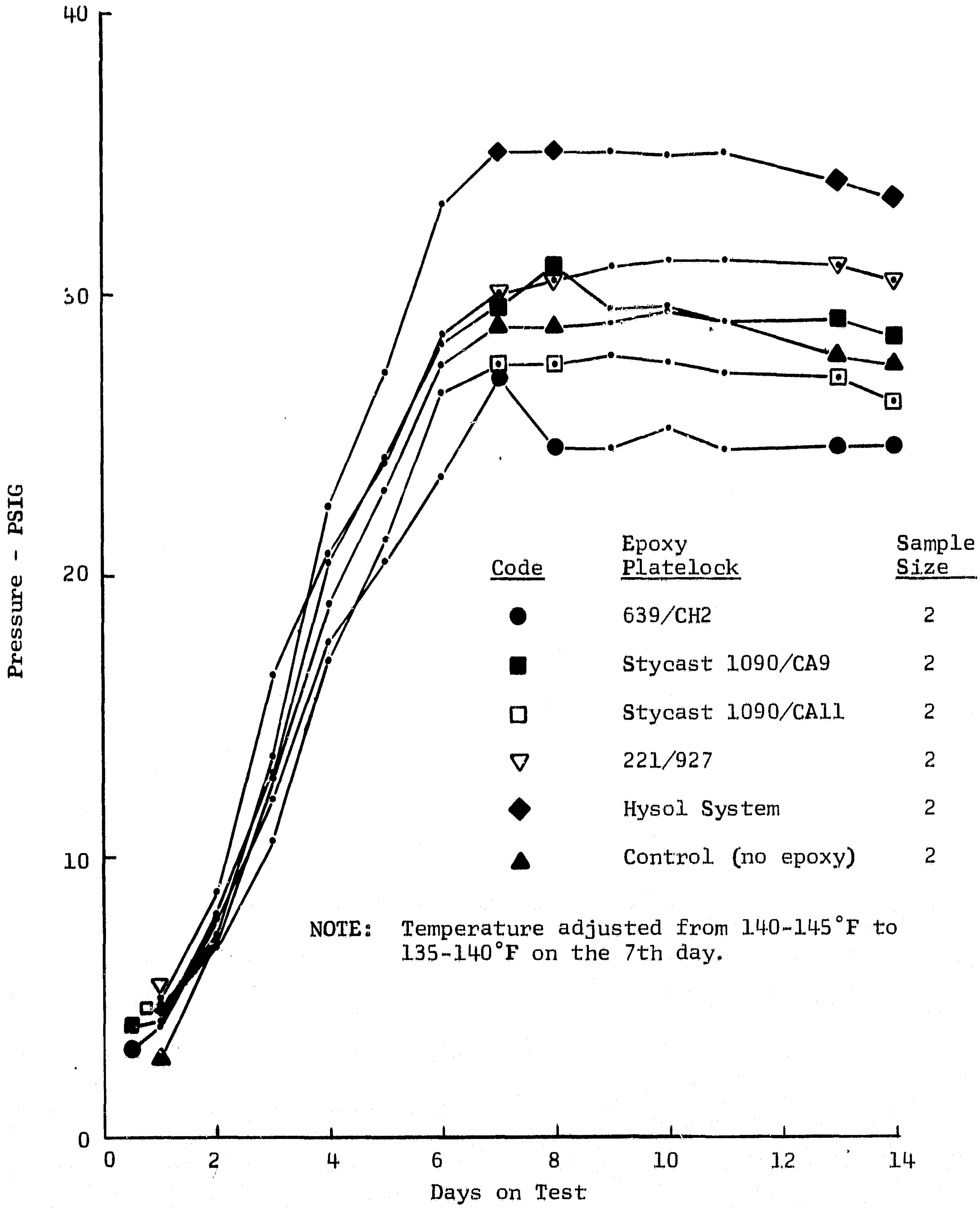
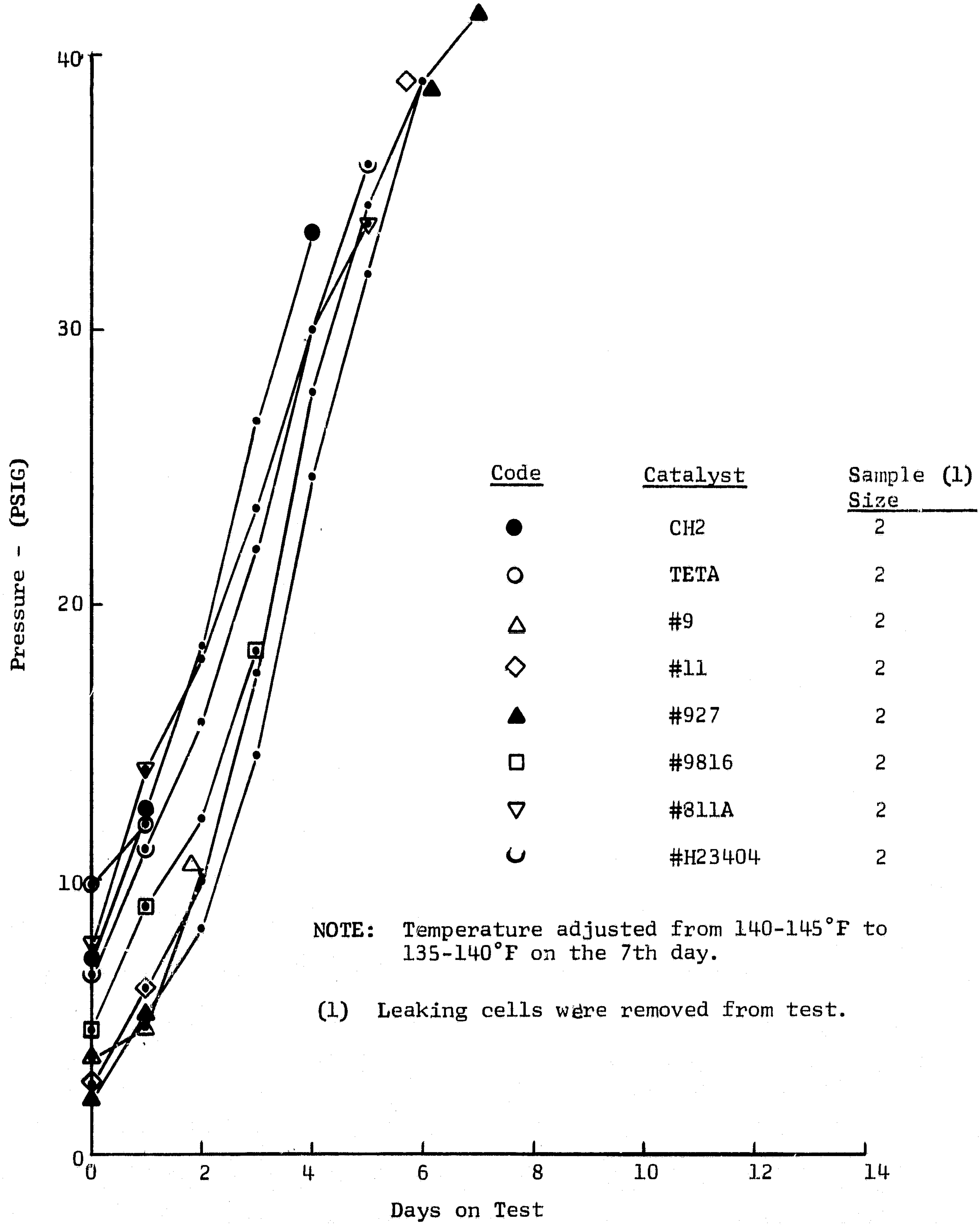


FIGURE 2
PRESSURE GENERATED DURING 140 ±5°F STORAGE
OF MODEL 257 MONOBLOCKS CONTAMINATED WITH EPOXY CATALYSTS



NOTE: Temperature adjusted from 140-145°F to 135-140°F on the 7th day.

(1) Leaking cells were removed from test.

FIGURE 3
PRESSURE GENERATED DURING 140 ±5°F STORAGE
OF MODEL 257 MONOBLOCKS CONTAMINATED WITH EPOXY RESINS
OR ADDITIVES

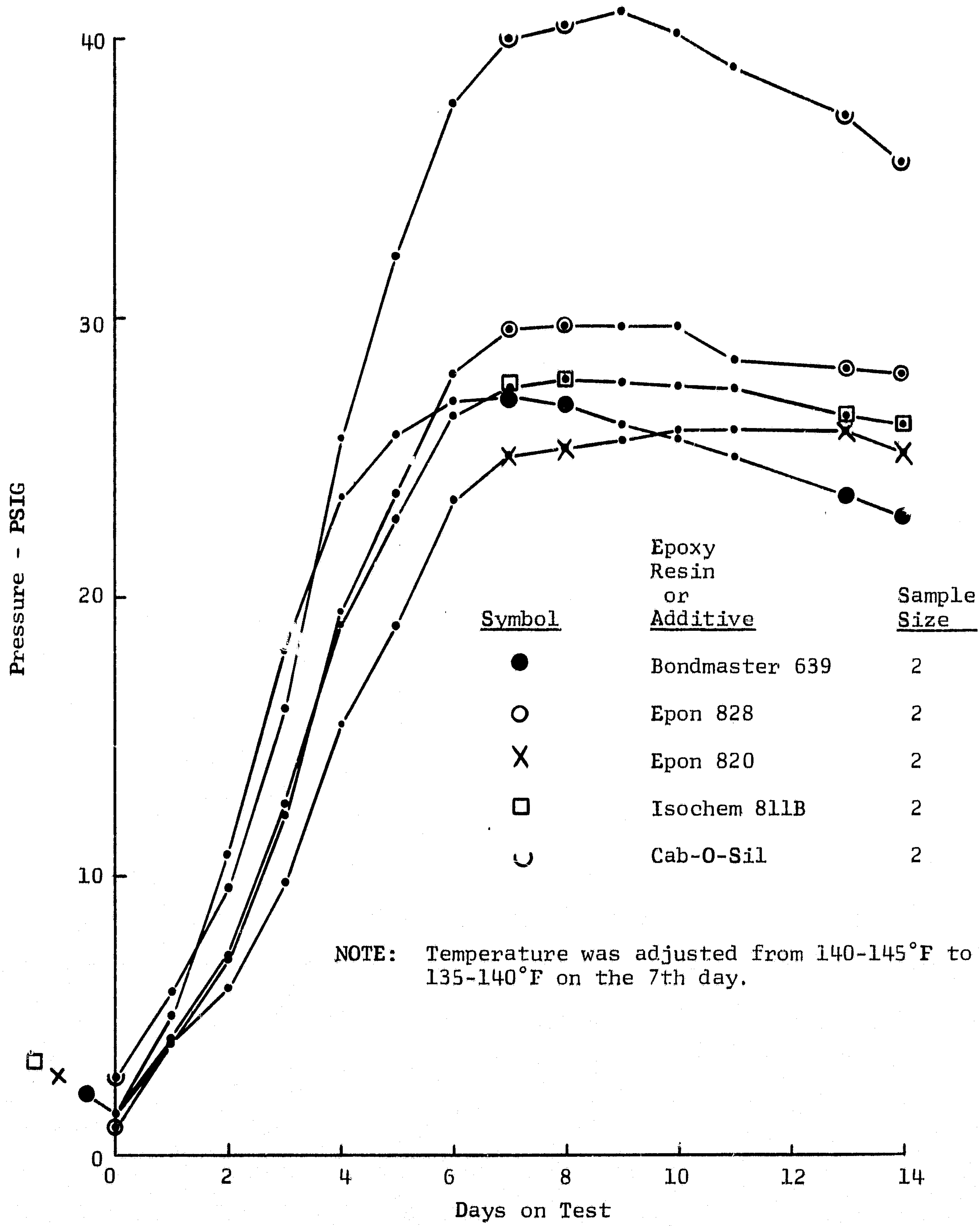


FIGURE 3
(Continued)
PRESSURE GENERATED DURING 140 ±5°F STORAGE
OF MODEL 257 MONOBLOCKS CONTAMINATED WITH EPOXY RESINS OR ADDITIVES

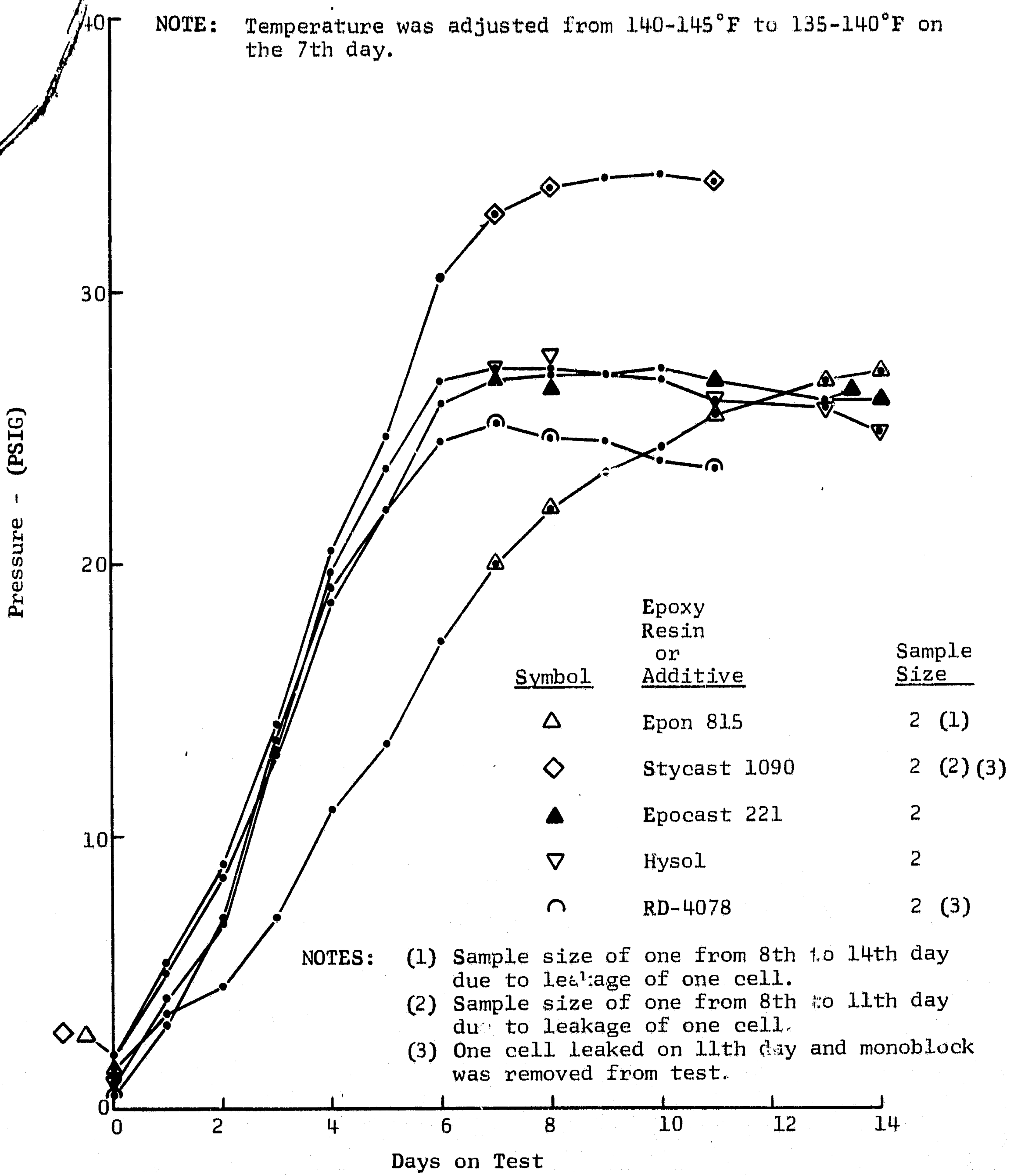
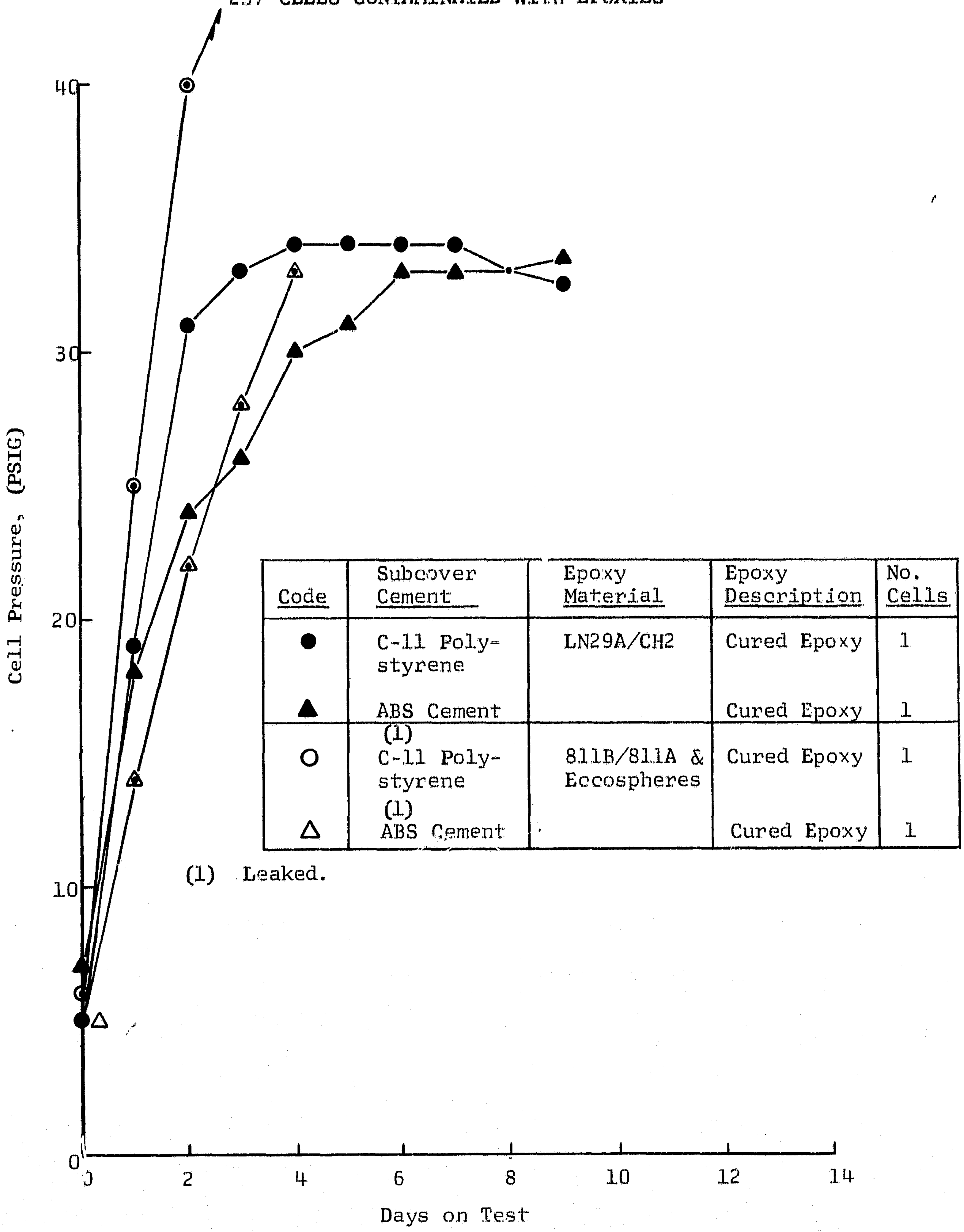


FIGURE 4
PRESSURE GENERATED DURING 140 ±5°F STORAGE OF MODEL 257 CELLS CONTAMINATED WITH EPOXIES

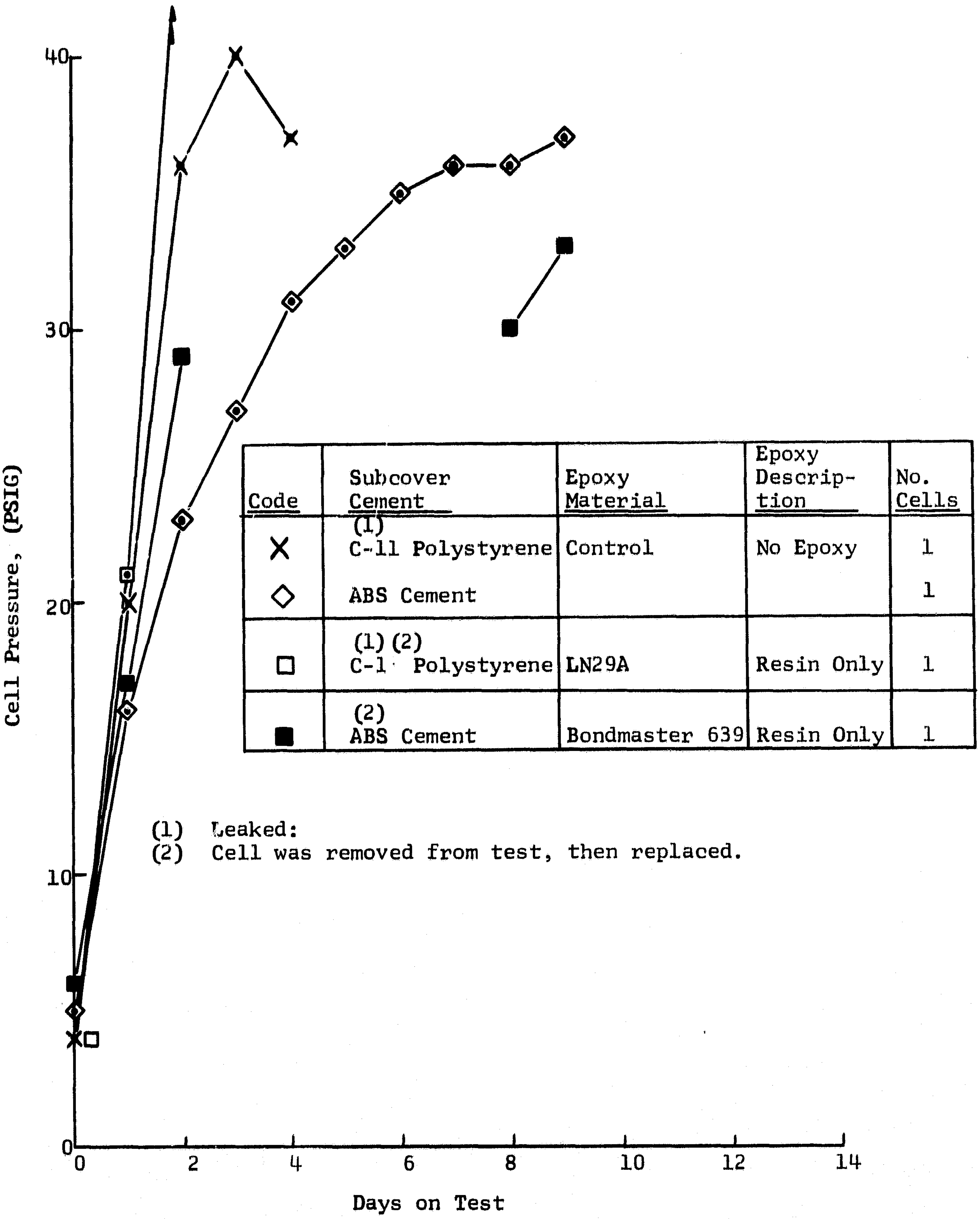


Code	Subcover Cement	Epoxy Material	Epoxy Description	No. Cells
●	C-11 Poly-styrene	LN29A/CH2	Cured Epoxy	1
▲	ABS Cement		Cured Epoxy	1
○	(1) C-11 Poly-styrene	811B/811A & Eccospheres	Cured Epoxy	1
△	(1) ABS Cement		Cured Epoxy	1

(1) Leaked.

FIGURE 4

PRESSURE GENERATED DURING 140 ±5°F STORAGE OF MODEL 257 CELLS CONTAMINATED WITH EPOXIES



Code	Subcover Cement	Epoxy Material	Epoxy Description	No. Cells
X	(1) C-11 Polystyrene	Control	No Epoxy	1
◇	ABS Cement			1
□	(1) (2) C-1 Polystyrene	LN29A	Resin Only	1
■	(2) ABS Cement	Bondmaster 639	Resin Only	1

(1) Leaked:
(2) Cell was removed from test, then replaced.