

**TIME- TO- EXPLOSION
THERMAL INITATION TEST FOR EXPLOSIVES (U)**

L.C. Myers

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Mason & Hanger- Silas Mason Co., Inc.

Pantex Plant

April, 1964

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ABSTRACT

A controlled series of experiments have been performed in an attempt to evaluate the Henkin time-to-explosion test as a reactivity test and a quality assurance tool. This was done by comparing the Henkin results with those obtained with the presently used gas chromatograph chemical reactivity test. Two explosives, PBX 9404 and LX-04-1, were tested with four "inerts": sodium chloride, lead powder, Silastic Q-300030 and epoxy 633111. The experimental data from the two tests are given; a method for mathematically reducing the Henkin data is considered.

The data from the two tests are not in complete agreement. Both predict that the epoxy is reactive with and that NaCl is unreactive with both explosives. However, the Henkin data indicated reactivity of lead powder and Silastic with LX-04-1; the gas chromatograph did not. For PBX 9404, the Henkin test indicated that the lead was borderline reactive and the Silastic unreactive, while the gas chromatograph predicted the opposite. DTA thermograms are included to help interpret the comparison test results.

A section in the appendix is devoted to the experimental problems encountered in getting the Henkin test operational. All raw data generated during that time are included in the appendix. In addition, data obtained subsequently with a fairly wide variety of explosives (and propellants) are given; these indicate the present quality of data.

In the determination of the thermal sensitivity of explosives, the time-to-explosion investigation has proven to be of value. Henkin and McGill¹ investigated the time-temperature relationship at which decomposition becomes explosive in character within short time intervals. It was apparent to them that the time-temperature relation was not constant, but depended on several variables. This test had been used for many years, but was abandoned when it failed to give the correct activation energy of pure explosives. The theoretical work of Zinn², which introduced a chemical depletion term, explained the shortcomings in the previous treatments. Mader's³ experimental work with a scaled-up test verified the theoretical work. R. N. Rogers³ has recently modified the experimental arrangement; this has resulted in a lower energy input rate, operation at lower temperatures and expansion of the time range. In general, this reduces considerably the perturbation of thermal lags, simplifies the measurement of the time-to-explosion, allows one to work in a region where a lesser accuracy in time measurement is acceptable, and makes the test a better mock-up of some important practical situations (safety and chemical reactivity).

The test has been proposed as a means of detecting incompatibility; the purpose of the present work was to evaluate the test to that end and to determine the possibility of its use as a quality assurance tool. In short, the approach was to compare the results from the Henkin and gas chromatographic chemical reactivity tests in a controlled, statistical manner.

¹Henkin, H., McGill, R., I. & E.C. 44, 1391 (1952)

²Zinn, J., Mader, C.L., Journal of Applied Physics 31, 323 (1960)

³Rogers, R.N., I. & E.C. Production Research & Development, Vol. I, No. 3, (1962)

Additional modifications, generally in the methods of loading and timing, were adopted in the course of the work at Pantex. The various problems encountered along the way (appendix) and the test results are presented and discussed in this report.

Apparatus and Experimental Procedure Used in Comparison Test

In this section, the procedure and technique used in performing the Henkin and gas chromatographic comparison experiment will be described. The latter test was used as a "yard stick" because it is presently accepted and used as a means of measuring chemical reactivity. The problems encountered in the development of the procedure (and subsequent development) will constitute a separate section (the appendix of this report). It is believed that this separation will permit a minimum of confusion for the various readers.

The basic apparatus used for the Henkin portion of the test was similar to that described by Rogers. The reaction chamber was identical; the metal bath temperature was controlled via a Fenwal Electronics, Inc. probe thermistor (in series with a $42\ \Omega$ resistor) by a Fisher Temperature Controller Model 44. The reaction temperature was measured by a chromel-alumel thermocouple placed in the bath liquid and monitored with a Leeds and Northrup K-3 Potentiometer. Major modifications to Rogers' apparatus resulted from changes in the loading and sealing of the blasting cap sample containers. The technique settled upon after numerous other systems had failed was as follows. The blasting cap (#8 brass made by du Pont) was loaded with the desired sample and followed by two 6.5 mm diameter gas checks (obtained from Hornady) which were also brass and were resized to slide easily but snugly into the 0.260 inch I.D. blasting caps. The first gas check had the concave side turned toward the sample and the second had the concave side turned away from the sample. This was followed by a lead plug, 9/16 inch long and 1/4 inch in diameter. A punch and die was used to press the assembly at 350 pounds

(7000 psi). The cap was then crimped around the lead plug (about 1/4 inch from top of plug) with a Magneformer. Caps loaded in this way often blew the bottom out of the cap rather than forcing the gas check-plug assembly out the top. The timing system originally used had to be changed. A carbon-type earphone used in conjunction with a digital counter (Computer Measurements Co., Model 727B Universal Counter-Timer) proved to be satisfactory.

Four "inerts" with varying degrees of reactivity with the explosives PBX 9404 (Blend 1 of Lot 524-61) and LX-04-1 (Blend 2 of Lot 673-62) were chosen, ground, and sent to Pantex by LRL. These were sodium chloride (Baker & Adams Co.), lead powder (Fisher Scientific Co.), Silastic Q-30030, and epoxy 633111. The H.E. samples were ground with a Wiley Intermediate Model Mill; total (i.e., no sieving or other segregation) H.E. samples were used. The explosives, epoxy and Silastic were then dried in a vacuum oven at 60°C. for two hours. All of the samples were kept in a desiccator cabinet (calcium sulfate was the desiccant) with the exception of the sodium chloride which was kept in a sealed container. The pure H.E. sample size was 50 mg; the H.E.-inert mixtures were made by placing 50 mg of inert and 50 mg of explosives into the blasting cap and then rolling and shaking to achieve some degree of homogeneity.

The first step in the procedure was to experimentally obtain the time-temperature curves for the pure explosives. When these curves were determined, two temperatures, T_1 and T_2 , were chosen for each H.E. such that the time-to-explosion would be on the order of 50 and 200 seconds. Fifteen replicate runs (in subgroups of five) were then made for each combination of temperature, "inert" and explosive; these

subgroups of five were "randomly" sequenced within a given temperature and explosive. Pure explosive control runs were made each time a group of mixtures was tested. A typical day's run consisted of five pure H.E., five H.E. + NaCl, five H.E. + Pb, five H.E. + Silastic, and five H.E. + epoxy, all at T_1 .

The gas chromatograph reactivity experiment was performed in a two-column chromatograph designed by LRL and manufactured by the Royal Research Corporation, Hayward, California. The experimental conditions were those of USAEC Specification RM-251808 with the exception that modified sample tubes and Pyrex glass sample crucibles were used. The inert materials and H.E. samples were the same as those used in the Henkin portion of the test. The H.E. samples were given a different treatment however; they were ground and the fractions passing U.S. Sieve #100 (149μ) and retained on U.S. Sieve #270 (53μ) were used. All samples were dried for two hours in vacuo at 60°C . The sample weight taken was 250 mg or 250 mg of each material in the case of mixtures. After placing the sample crucibles in the sample holder, the holders were evacuated, purged, filled with helium (15 psig), and immersed in a $120 \pm 0.5^\circ\text{C}$. oil bath for 22 hours (experimentally, this varied from 22 to ~ 23 hours). The evolved fixed gases (N_2 , O_2 , NO, CO, N_2O , CO_2) were then analyzed in the chromatograph; the organics and other products were not analyzed. Instrument parameters were:

1. Column 1 Polypropylene glycol on firebrick, furnace 50°C ., Filament current 350 ma., 15 psig He.
2. Column 2 Silica gel, furnace 45°C ., Filament current 350 ma., 13 psig He.
3. Thermal Conductivity Cell Furnace 50°C .
4. He flow rate 100 cc/minute

Results

The Henkin raw data plotted as the time-to-explosion versus the reciprocal of the absolute temperature for the pure explosives are given in Figures 1 and 2. The solid lines were drawn for clarification and to indicate our choices of T_1 and T_2 . It should be noted that the lines are drawn along the locus of concentrated short time points in compliance with the principle that the worst conditions should be given the most weight in safety or compatibility considerations. During the course of checking out the loading technique, the PBX 9404 + lead powder Henkin curve was also generated; it is included here as Figure 3 because it introduces an interesting point to be discussed later.

The Henkin data for the comparison tests are given in Table I; the gas chromatograph reactivity data are reported in Table II.

DTA thermograms of the pure explosives and the mixtures are shown in Figures 4 through 13. These are included for comparison with chromatography and Henkin data.

The harmonic mean is offered as a possibility for the analysis of the Henkin data. The harmonic mean, M_h , is defined as

$$M_h = \frac{N}{\sum_{i=1}^N \frac{1}{X_i}}$$

where N is the number of trials in a subgroup; i.e., five for this experiment and X_i is the explosion times of the i th trial. For a reactivity coefficient

RC, the following relationship is used:

$$RC = \frac{\sum_{j=1}^S \left[\frac{M_h (H.E.)}{M_h (mix)} \right]_j}{S}$$

where $M_h (H.E.)$ is the harmonic mean for the pure explosive, $M_h (mix)$ is the harmonic mean for a mixture (of the explosive and the inert for a particular subgroup) and S is the number of subgroups; i.e., three for this test.

The numbers that result from this calculation are shown in Table III. It should be noted that those trials which did not produce an explosion (these were mixtures that were stopped after a reasonable length of time) were not used in the calculations; since the times would be large, their contribution to the mean reactivity coefficient would be very small.

Discussion

The Henkin curves of LX-04-1 and PBX 9404 in Figures 1 and 2 show that at a given temperature, the PBX 9404 explosion times are shorter than those for LX-04-1; this agrees with data from other thermal tests. The curves are of the general shape expected for explosives. They qualitatively agree with the Henkin data generated by Rogers of LASL; quantitatively, they would not be expected to agree since the cap loading techniques (ours being contained and sealed more tightly) are different. We feel the change in cap loading makes the data in this report considerably more representative and reproducible than was previously possible.

The data taken on September 16, 1963 (Table I) show that no explosions occurred with the PBX 9404 + NaCl; indeed, this happened a few other times. Since NaCl is hygroscopic, it is possible that the NaCl picked up some moisture and caused this difference in times. In the comparison test, certain mixtures consistently blew the plug and seal out the top; more recent data, using more refined cap loading techniques, have been generated in which these same mixtures blew the bottom out of the caps. There was no appreciable change in the explosion times so the comparison test data are considered as giving a true picture. It is felt that the most desirable loading system is one that ruptures the cap; experience has shown that this is more reproducible from day to day.

In analyzing the Henkin data the shortest explosion time at a given temperature represents the worst condition that exists. However, experience has shown that pressure blows can result from systems improperly sealed; these can give times that are anomalously short. Therefore, a method is needed that will

mathematically weigh the shorter times more heavily than the longer times but not overdo it. The harmonic mean does this rather well. It weighs the shorter times heavier than either the arithmetic or geometric mean. Also, the harmonic mean seems to be more promising for it can better handle data with a greater spread of values. Regardless of the choice, one must still make a reasonable selection of the critical value(s) of the reactivity coefficient, RC. On the basis of this one comparison test, the following ratio is considered a possibility. Samples having an RC value less than 1.5 are unreactive or indeterminant while those with RC greater than 1.5 but less than 3 are borderline reactive. Those with RC greater than 3 are reactive. The low critical ratios are dictated by safety criteria. The proper value would, of course, depend on the use to which the data are to be put.

No attempt was made here to subject the chromatograph data to a rigorous mathematical treatment. For our purposes, the reactivity coefficient, RC, taken as the ratio of the total volume of gas evolved by the mixture to the sum of the volumes from the individual components, was considered to be adequate (although very naive). Somewhat arbitrarily, critical values were assigned; namely, the system is considered unreactive or indeterminant for $RC < 2$, borderline reactive for $2 < RC < 3$ and reactive for $RC > 3$.

Using these ground rules, the comparison of the two tests is as follows:

1. Henkin test LX-04-1 + "inerts"

- a. LX-04-1 + epoxy 633111: Reactive; $RC \approx 23$
- b. LX-04-1 + lead powder: Reactive; $RC \approx 6.2$
- c. LX-04-1 + Silastic Q-30030: Reactive; $RC \approx 3.6$
- d. LX-04-1 + NaCl: Unreactive; $RC \approx 1.1$

2. Reactivity test LX-04-1 + "inerts"

- a. LX-04-1 + epoxy 633111: Reactive; $RR \approx 4.2$
- b. LX-04-1 + lead powder: Unreactive; $RR \approx 0.8$
- c. LX-04-1 + Silastic Q-30030: Unreactive; $RR \approx 0.5$
- d. LX-04-1 + NaCl: Unreactive; $RR \approx 0.4$

3. Henkin test PBX 9404 + "inerts"

- a. PBX 9404 + epoxy 633111: Reactive; $RC \approx 6.4$
- b. PBX 9404 + lead powder: Borderline reactive; $RC \approx 2.4$
- c. PBX 9404 + Silastic Q-30030: Unreactive; $RC \approx 0.6$
- d. PBX 9404 + NaCl: Unreactive; $RC \approx 0.7$

4. Reactivity test PBX 9404 + "inerts"

- a. PBX 9404 + epoxy 633111: Reactive; $RR \approx 3.9$
- b. PBX 9404 + lead powder: Unreactive; $RR \approx 1.0$
- c. PBX 9404 + Silastic Q-30030: Borderline reactive; $RR \approx 1.7$
- d. PBX 9404 + NaCl: Unreactive; $RR \approx 0.8$

The data for the two tests are not in complete agreement. They do agree that epoxy reacts strongly and NaCl is very unreactive with both explosives. However, the Henkin test reflects reactivity of lead and Silastic with LX-04-1, while the gas chromatograph does not. Also, the Henkin test indicates some reactivity of lead, but no reactivity of Silastic, with PBX 9404; the gas chromatograph predicts the opposite situation. The discrepancies are not as clear cut in the PBX 9404 cases as for the LX-04-1's. The reasons for the discrepancies have not generally been investigated (except to some extent for lead + PBX 9404) and, therefore, only a priori explanations can be offered. These probably involve one or more of confinement, reaction temperature, geometry, area of contact and thermal conductivity. For the record, we will say here that the DTA results align themselves more closely with those from the Henkin than the gas chromatograph test. The one case for which

some additional data are available is PBX 9404 + lead powder.⁴ It will be noted from Figures 2 and 3 that at low temperatures, the curves of PBX 9404 and PBX 9404 + lead cross. The explosion times for the latter are considerably shorter for all temperatures above about 215°C. At 210°C. ($\frac{1000}{T^{\circ}K} = 2.07$); however, PBX 9404 times were on the order of 700 seconds while the mixture did not explode after 2000 seconds. The latter caps were opened; the black residue was run in the DTA. The differential thermogram was essentially that of lead; i.e., the material never would have exploded. A possible explanation is that the lead powder can conduct away (both locally and on the whole) the heat generated by the slowly decomposing explosive, thus preventing it from reaching the explosion threshold temperature.

More important than any explanation of this activity is the lesson which can be learned and the warning sounded by these particular data. Great care must be exercised in choosing reaction temperatures in future Henkin work in order to minimize misinterpretation of hazardous or reactive mixtures. Obviously, choices of reaction temperatures corresponding to $\frac{1000}{T^{\circ}K}$ values of about 2.05 and 1.89 would present a much different picture than the temperatures chosen in this work. It would appear that considerable knowledge about a system is needed; i.e., fairly complete time-temperature curves, before one could "rely" on Henkin data; in critical situations, use of the two-temperature method alone would be

⁴The lead sample used for the generation of the PBX9404 + lead Henkin curve was supplied by Rogers, LASL, and was known to be reactive with PBX 9404. A different sample was used for the comparison test; the comparison test sample was bluish in color while the other was brown, indicating that some oxidation had probably taken place in the latter. The two samples gave essentially the same explosion times when mixed with PBX 9404.

unacceptable. It sort of boils down to the fact (and we are speaking from past experience) that new explosive systems will come equipped with their own eccentricities which may require variations in loading techniques, data interpretation, etc. For proposed routine testing programs or specification requirements, these problems could, of course, be worked out (and indeed must be for each specific system) in advance.

Building of workable Henkin equipment and mastering of loading techniques at other contractor installations could now surely be done with relative ease if the personnel are acquainted with basic thermal techniques and if the explosive systems are not too greatly different from those used in this work.

We feel the greatest problem with the Henkin test (and the gas chromatograph reactivity test for that matter) is in determining and quantitating what it is trying to tell. It would appear to be able to diagnose hazardous or unsafe situations, but not be directly or immediately relatable to long term surveillance problems. Therefore, it is thought that the test is not ready for use as a routine quality assurance tool. However, the Henkin test shows possibilities and should be allowed to take a place along side and be coupled with the DTA, pyrolysis, gas chromatograph, hot stage microscopy, etc., in diagnosing potentially hazardous materials and in investigating the thermal decomposition mechanisms, etc. of explosives and explosive systems.

TABLE I

HENKIN REACTIVITY TEST COMPARISON

Sample	9-12-63			9-24-63			9-11-63			9-13-63			9-18-63			9-20-63		
	Time			Time			Time			Time			Time			Time		
	Run (sec)@			Run (sec)@			Run (sec)@			Run (sec)@			Run (sec)@			Run (sec)		
	No.	243°C		No.	242°C		No.	242.5°C		No.	264°C		No.	264°C		No.	@264°C	
LX-04-1	1	594.4		5	1014.7		1	767.9		8	243.4		5	75.0		1	56.9	
	10	539.8		10	1130.7		6	733.6		11	121.8		10	270.4		6	218.7	
	15	481.3		15	1093.8		11	704.1		13	69.7		15	71.4		11	85.2	
	20	709.5		23	702.2		20	724.8		18	53.4		20	117.2		16	78.5	
	28	735.9		25	616.7		25	737.0		20	88.2		25	62.5		25	62.5	
LX-04-1 + Pb	2	81.1†		4	92.7†		2	127.4†		2	22.3†		4	21.8†		2	9.4	
	9	55.2†		9	87.4†		7	106.3†		4	15.8†		9	18.3†		7	17.4†	
	11	81.2†		14	84.3†		12	149.9†		15	19.7†		14	21.0†		12	17.2†	
	21	100.6†		18	142.8†		17	149.4†		17	12.7†		19	25.8†		17	17.4†	
	25	59.1†		21	111.1†		22	126.8†		22	22.4†		24	14.8†		22	17.1†	
LX-04-1 + NaCl	3	536.7		3	849.6		3	497.0		5	112.9		3	58.1		3	37.2	
	8	576.4†		8	1008.5		8	477.1		10	196.3		8	42.5		8	103.3	
	16	538.1		13	848.8		13	483.3		12	374.3		13	144.1		13	125.8	
	24	495.4		22	447.7		19	586.2		19	288.2		18	204.4		18	273.8	
	26	402.3		24	767.1		24	609.2		23	98.6		23	42.8†		23	36.7	
LX-04-1+ Silastic	4	175.6†		2	195.6†		4	149.8†		1	44.1†		2	37.4†		5	32.6†	
	7	218.5†		7	196.2†		9	191.4†		6	17.3†		7	40.2†		9	21.2†	
	13	98.1†		12	228.3†		14	252.1†		14	22.2†		12	55.1†		14	25.6†	
	18	312.8†		17	159.9†		18	119.7†		16	38.8†		17	50.8†		19	24.8†	
	23	160.7†		20	110.7		23	151.0†		25	37.7†		22	36.8†		24	22.6†	
LX-04-1 + Epoxy	5	17.5†		1	19.6†		5	18.7†		3	10.0†		1	10.5†		4	8.6†	
	6	17.1†		6	17.9†		10	21.7†		7	10.9†		6	11.8†		10	9.0†	
	14	18.5†		11	18.9†		15	21.4†		9	8.5†		11	18.4†		15	8.4†	
	19	16.4†		16	18.4†		16	18.2†		21	9.9†		16	16.8†		20	10.2†	
	27	19.3†		19	19.4†		21	30.9†		24	11.2†		21	11.7†		25	9.0†	

† - Blew seal out of top.

TABLE I (Con't.)

Sample	9-23-63		9-25-63		9-16-63		9-17-63		9-10-63		9-19-63	
	Time		Time		Time		Time		Time		Time	
	Run (sec)@		Run (sec)@		Run (sec)@		Run (sec)@		Run (sec)@		Run (sec)@	
	No.	220°C	No.	219.5°C	No.	220°C	No.	242°C	No.	242.5°C	No.	242°C
9404	1	299.4	1	269.5	5	539.0	1	63.1	1	55.5	3	20.6†
	6	275.5	10	216.1	10	281.1	6	64.4	6	39.8	8	25.7
	11	299.2	15	143.4†	15	293.9	11	27.8	11	48.8	13	61.4
	16	700.0‡	20	184.8	20	241.8	16	62.0	16	39.2	18	51.4
	21	234.0	25	241.2	24	306.0	21	32.2†	21	52.1	23	25.2
9404 + Pb	2	586.7	2	111.8	4	169.8†	4	13.5	2	11.7	1	16.2†
	7	106.2†	9	148.4	9	209.3†	9	13.7	7	12.3	6	12.9
	12	112.2	14	270.5	14	143.4	14	13.9	12	14.6	11	14.6†
	17	114.9†	19	150.8	19	157.5	19	15.1	17	16.0	16	14.5†
	22	102.6†	24	111.6	23	143.5†	24	13.3	22	17.2	21	11.8†
9404 + NaCl	3	467.5	3	277.2†	3	1500.0‡	2	73.2	3	81.5	4	118.7
	8	226.2	8	261.0†	8	1000.0‡	7	69.0	8	85.5	9	74.0
	13	232.8	13	1000.0‡	13	1000.0‡	12	69.9	13	102.1	14	74.2
	18	421.8	18	296.2†	18	1000.0‡	17	76.0	18	60.0	19	71.4
	23	720.0‡	23	700.0‡	25	1800.0‡	22	63.5	23	68.8	24	76.5
9404 + Silastic	4	1000.0‡	4	397.8†	2	428.8†	5	103.0†	4	172.8†	2	70.5†
	9	403.7†	7	475.0†	7	418.0†	10	72.5	9	101.1†	7	81.2†
	14	338.0†	12	374.0†	12	1000.0‡	15	58.4†	14	95.2	12	82.2
	19	215.3	17	557.8†	17	664.4†	20	59.5†	19	61.2†	17	82.0†
	24	246.3†	22	272.2†	22	477.0†	25	76.4†	24	51.8†	22	94.3†
9404 + Epoxy	5	23.7	5	26.0†	1	328.9	3	16.4†	5	14.2†	5	15.1†
	10	22.8†	6	23.2†	6	22.0†	8	16.0†	10	14.6†	10	15.2†
	15	22.5†	11	25.8†	11	26.0†	13	14.8†	15	13.5†	15	14.4†
	20	21.9†	16	25.0†	16	24.8†	18	15.5†	20	17.6†	20	13.8†
	25	21.8†	21	25.1†	21	24.6†	23	16.2†	25	17.3†	25	15.8†

† Blew seal out of top.

‡ No explosion

Table II

Gas Chromatographic Reactivity Results^{1,2,3}
(Gas Evolved in μ liter @ STP)

Sample	Run No.	Date (1963)	Time (hrs.) @120°C. $\pm$ 0.5	N ₂ + O ₂	NO + CO	N ₂ O	CO ₂	TOTAL
NaCl	471	6-4	22:15	27.3	0	2.5	16.4	46.2
NaCl	472	6-4	22:00	19.5	0	0.6	16.1	36.2
Pb	473	6-4	22:00	21.1	0	1.0	17.9	40.0
Pb	474	6-4	21:45	20.1	0	0.8	5.0	25.9
Epoxy	475	6-4	22:00	16.7	0	1.5	16.1	34.4
Epoxy	476	6-4	22:00	17.3	0	2.5	17.4	37.2
Silastic	477	6-4	22:00	3.3	32.0	3.2	27.9	66.4
Silastic	478	6-4	22:15	6.3	15.2	1.8	30.5	53.9
9404	480	6-5	21:45	30.4	185.	31.0	114.	360.
9404	481	6-5	22:00	17.4	190.	25.5	114.	347.
NaCl + 9404	482	6-5	22:00	29.2	170.	18.9	99.3	317.
NaCl + 9404	483	6-5	21:45	32.7	154.	26.1	113.	326.
Epoxy + 9404	484	6-5	22:00	242.	272.	924.	327.	1760.
Epoxy + 9404	485	6-5	22:00	238.	322.	1050.	445.	2050.
Silastic + 9404	486	6-5	22:00	61.1	415.	194.	168.	839.
Silastic + 9404	487	6-5	22:15	69.4	445.	218.	204.	936.
9404	488	6-6	22:45	32.2	178.	23.4	115.	349.
9404	489	6-6	22:00	25.8	172.	25.0	115.	338.
Pb + 9404	490	6-6	22:00	36.4	172.	35.2	85.0	329.
Pb + 9404	491	6-6	22:15	39.5	174.	53.5	93.8	361.
NaCl + 9404	492	6-6	22:30	25.8	201.	28.8	124.	380.
NaCl + 9404	493	6-6	22:00	29.6	206.	29.6	138.	394.
Epoxy + 9404	494	6-6	22:15	240.	309.	1040.	391.	1980.
Epoxy + 9404	495	6-6	22:00	236.	326.	1020.	388.	1970.
Silastic + 9404	515	6-12	22:00	124.	195.	353.	204.	876.
Silastic + 9404	516	6-12	22:15	55.0	317.	125.	138.	635.
Epoxy + 9404	517	6-12	23:15	83.0	221.	228.	186.	718.
Epoxy + 9404	518	6-12	22:00	237.	289.	921.	374.	1821.
NaCl + 9404	519	6-12	22:30	26.3	206.	29.0	113.	374.
NaCl + 9404	520	6-12	22:45	34.3	226.	35.8	143.	439.
9404	521	6-12	23:15	30.2	211.	27.2	141.	409.
9404	522	6-12	23:00	32.6	206.	28.8	154.	421.
Pb + 9404	523	6-13	22:00	87.3	227.	145.	143.	602.
Pb + 9404	524	6-13	22:15	73.3	248.	93.6	133.	548.

¹Runs made from 6-4 to 6-19 were calculated using the calibration of 6-19.

²Volumes are not corrected to 22:00 hours.

³There is some uncertainty as to the reliability of the third significant figure.

Table II (Con't)

-17-

Gas Chromatographic Reactivity Results^{1,2,3}
(Gas Evolved in μ liter @ STP)

Sample	Run No.	Date (1963)	Time (hrs.) @120°C. $\pm$ 0.5	N ₂ + O ₂	NO + CO	N ₂ O	CO ₂	TOTAL
9404	525	6-13	22:15	430.	0	21.8	127.	579.
9404	526	6-13	22:15	32.8	224.	27.3	128.	412.
Pb + 9404	527	6-13	23:00	43.4	175.	44.3	88.8	352.
Pb + 9404	528	6-13	22:30	53.4	242.	54.1	99.5	449.
Silastic + 9404	529	6-13	22:30	61.6	376.	145.	132.	715.
Silastic + 9404	530	6-13	22:75	52.5	380.	115.	141.	728.
Pb	532	6-17	22:15	37.4	0	3.2	7.0	47.6
Pb	533	6-17	22:15	66.8	0	2.4	4.1	73.3
Silastic	534	6-17	22:00	6.2	28.4	20.8	13.4	68.8
Silastic	535	6-17	22:00	2.9	58.0	4.0	27.6	92.4
NaCl	536	6-17	23:00	24.7	0	5.3	4.4	34.4
NaCl	537	6-17	23:15	34.0	0	2.3	4.9	41.2
Epoxy	538	6-17	23:30	21.0	0	2.3	5.8	29.0
Epoxy	539	6-17	23:30	27.8	0	0.8	4.0	32.6
NaCl + LX-04-1	540	6-18	22:30	32.3	0	3.2	3.5	39.0
NaCl + LX-04-1	612	7-2	22:30	17.5	0	7.5	10.0	35.0
LX-04-1	544	6-18	22:00	133.	0	6.5	3.6	143.
LX-04-1	545	6-18	22:30	61.6	0	5.1	5.2	71.9
Pb + LX-04-1	546	6-18	22:45	31.2	0	20.4	3.6	55.2
Calibration Run 6-19-63								
Pb + LX-04-1	627	7-9	23:00	33.6	0	38.9	8.5	81.
Epoxy + LX-04-1	564	6-24	23:00	77.9	85.5	290.	34.7	488.
Epoxy + LX-04-1	565	6-24	22:30	62.8	58.0	256.	40.2	417.
Pb + LX-04-1	566	6-24	22:30	80.8	0	29.4	4.3	114.
Pb + LX-04-1	567	6-24	22:45	40.3	0	35.7	3.8	79.8
NaCl + LX-04-1	568	6-24	23:00	48.9	0	4.5	5.0	58.4
NaCl + LX-04-1	569	6-24	23:00	11.4	0	4.8	3.6	19.8
LX-04-1	570	6-24	23:15	37.9	0	8.7	3.3	49.9
LX-04-1	571	6-24	23:15	33.2	0	3.8	3.6	40.6
Pb + LX-04-1	572	6-25	22:15	97.0	0	25.0	4.9	127.
Pb + LX-04-1	609	7-2	23:00	30.0	0	37.4	7.4	74.8
NaCl + LX-04-1	574	6-25	22:30	41.7	0	3.4	4.4	49.5
NaCl + LX-04-1	575	6-25	22:30	13.9	0	5.5	4.2	23.6

¹Runs made from 6-4 to 6-19 were calculated using the calculation of 6-19.

²Volumes are not corrected to 22:00 hours.

³There is some uncertainty as to the reliability of the third significant figure.

Gas Chromatographic Reactivity Results^{1,2}
(Gas Evolved in μ liter @ STP)

Sample	Run No.	Date (1963)	Time (hrs.) @120°C. ± 0.5	N ₂ + O ₂	NO + CO	N ₂ O	CO ₂	TOTAL
Silastic + LX-04-1	576	6-25	23:30	37.2	0	16.6	5.9	59.7
Silastic + LX-04-1	577	6-25	23:30	38.4	0	14.2	7.4	60.0
LX-04-1	578	6-25	23:15	51.6	0	4.4	1.7	57.7
LX-04-1	579	6-25	23:15	28.6	0	3.6	2.7	34.9
Calibration Run 6-26-63								
LX-04-1	607	7-2	22:00	15.8	0	4.8	31.4	52.0
LX-04-1	608	7-2	22:00	17.7	0	20.4	17.6	55.7
Calibration Run 7-8-63								
Epoxy + LX-04-1	613	7-8	22:15	39.0	54.4	168.	97.0	358.
Epoxy + LX-04-1	614	7-8	22:30	36.0	60.4	181.	30.3	308.
LX-04-1	615	7-8	22:45	25.6	0	2.1	58.0	85.7
LX-04-1	616	7-8	22:30	19.0	0	3.0	27.6	49.6
Silastic + LX-04-1	618	7-8	22:15	29.8	0	6.5	9.7	46.0
Silastic + LX-04-1	622	7-9	22:30	35.6	0	12.4	18.9	66.9
Epoxy + LX-04-1	623	7-9	22:00	53.5	64.2	158.	162.	438.
Epoxy + LX-04-1	624	7-9	22:00	39.2	59.6	168.	60.5	327.
LX-04-1	626	7-9	22:30	17.8	0	5.0	16.9	39.7
Silastic + LX-04-1	621	7-9	22:15	35.5	0	9.8	58.5	104.
Silastic + LX-04-1	604	7-1	23:00	74.7	9.9	14.1	18.5	117.
Pb	628	7-10	22:45	22.0	0	0.9	29.2	52.1
Pb	629	7-10	23:00	21.4	0	3.2	15.6	40.2
Epoxy	630	7-10	22:45	18.0	0	2.0	12.2	32.2
Epoxy	644	7-12	22:45	31.0	0	1.8	5.1	37.9
NaCl	632	7-10	22:45	16.3	0	1.5	28.8	46.6
NaCl	643	7-12	21:45	31.1	0	0.2	2.9	34.2
Silastic	638	7-11	22:15	1.8	14.5	2.1	16.6	35.0
Silastic	637	7-11	22:15	1.7	18.4	1.4	18.5	40.0
LX-04-1	601	7-1	23:00	19.6	0	4.0	17.8	41.4
LX-04-1	602	7-1	23:00	44.4	0	2.0	8.9	55.3

¹Volumes are not corrected to 22:00 hours.

²There is some uncertainty as to the reliability of the third significant figure.

Table III

-19-

<u>Material</u>	<u>Date</u>	<u>Temp. (°C).</u>	<u>Harmonic Mean (sec.)</u>	<u>Ratio, RC</u>	<u>Average Ratio</u>
LX-04-1	9-12	243	596.6		
	9-24	242	857.7		
	9-11	243	733.0		
	9-13	264	88.1		
	9-18	264	90.0		
	9-20	264	79.8		
LX-04-1 + Pb	9-12	243	71.8	8.31	
	9-24	242	99.8	8.59	7.52
	9-11	242	129.8	5.65	
	9-13	264	17.7	4.98	
	9-18	264	19.6	4.59	4.98
	9-20	264	14.8	5.39	
LX-04-1 + NaCl	9-12	243	502.0	1.19	
	9-24	242	726.3	1.18	1.26
	9-11	242	525.0	1.40	
	9-13	264	165.4	0.53	
	9-18	264	65.8	1.37	1.03
	9-20	264	66.3	1.20	
LX-04-1 + Silastic	9-12	243	167.3	3.57	
	9-24	242	167.4	5.12	4.40
	9-11	242.5	162.1	4.52	
	9-13	264	28.1	3.14	
	9-18	264	42.9	2.10	2.82
	9-20	264	24.8	3.22	
LX-04-1 + Epoxy	9-12	243	17.7	33.7	
	9-24	242	18.8	45.6	37.86
	9-11	242	21.4	34.3	
	9-13	264	10.0	8.8	
	9-18	264	13.2	6.82	8.17
	9-20	264	9.0	8.87	

Table III (Con't)

<u>Material</u>	<u>Date</u>	<u>Temp. (°C)</u>	<u>Harmonic Mean (sec)</u>	<u>Ratio, RC</u>	<u>Average Ratio</u>
PBX 9404	9-16	220	308.3		
	9-23	220	274.3		
	9-25	219.5	201.1		
	9-10	242.5	46.2		
	9-17	242	43.7		
	9-19	242	30.7		
PBX 9404 + Pb	9-16	220	161.5	1.91	
	9-23	220	129.9	2.11	1.81
	9-25	219.5	143.0	1.41	
	9-10	242.5	14.0	3.30	
	9-17	242	13.9	3.14	2.89
	9-19	242	13.8	2.22	
PBX 9404 + NaCl	9-16	220			
	9-23	220	302.5	.907	.816
	9-25	219.5	277.4	.725	
	9-10	242.5	77.0	.600	
	9-17	242	70.1	.623	.536
	9-19	242	80.0	.384	
PBX 9404 + Silastic	9-16	220	480.4	.642	
	9-23	220	282.9	.970	.708
	9-25	219.5	391.9	.513	
	9-10	242.5	80.9	.571	
	9-17	242	70.9	.616	.522
	9-19	242	81.3	.378	
PBX 9404 + Epoxy	9-16	220	29.8	10.35	
	9-23	220	22.5	12.19	10.19
	9-25	219.5	25.0	8.04	
	9-10	242.5	15.3	3.02	
	9-17	242	15.8	2.77	2.62
	9-19	242	14.8	2.07	

LX-04-1

-21-

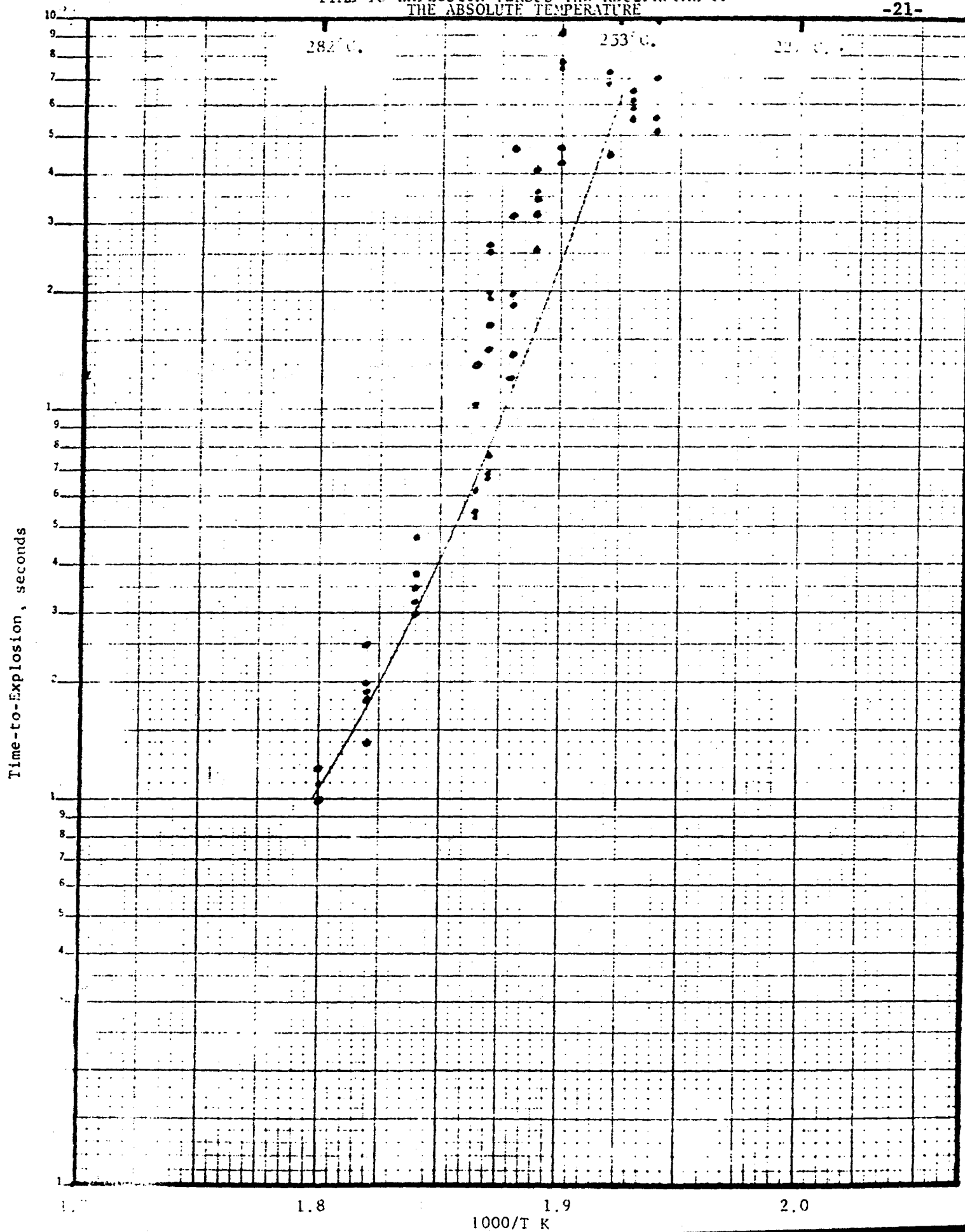


Figure 2
PBX 9404

TIME-TO-EXPLOSION VERSUS THE RECIPROCAL OF
THE ABSOLUTE TEMPERATURE

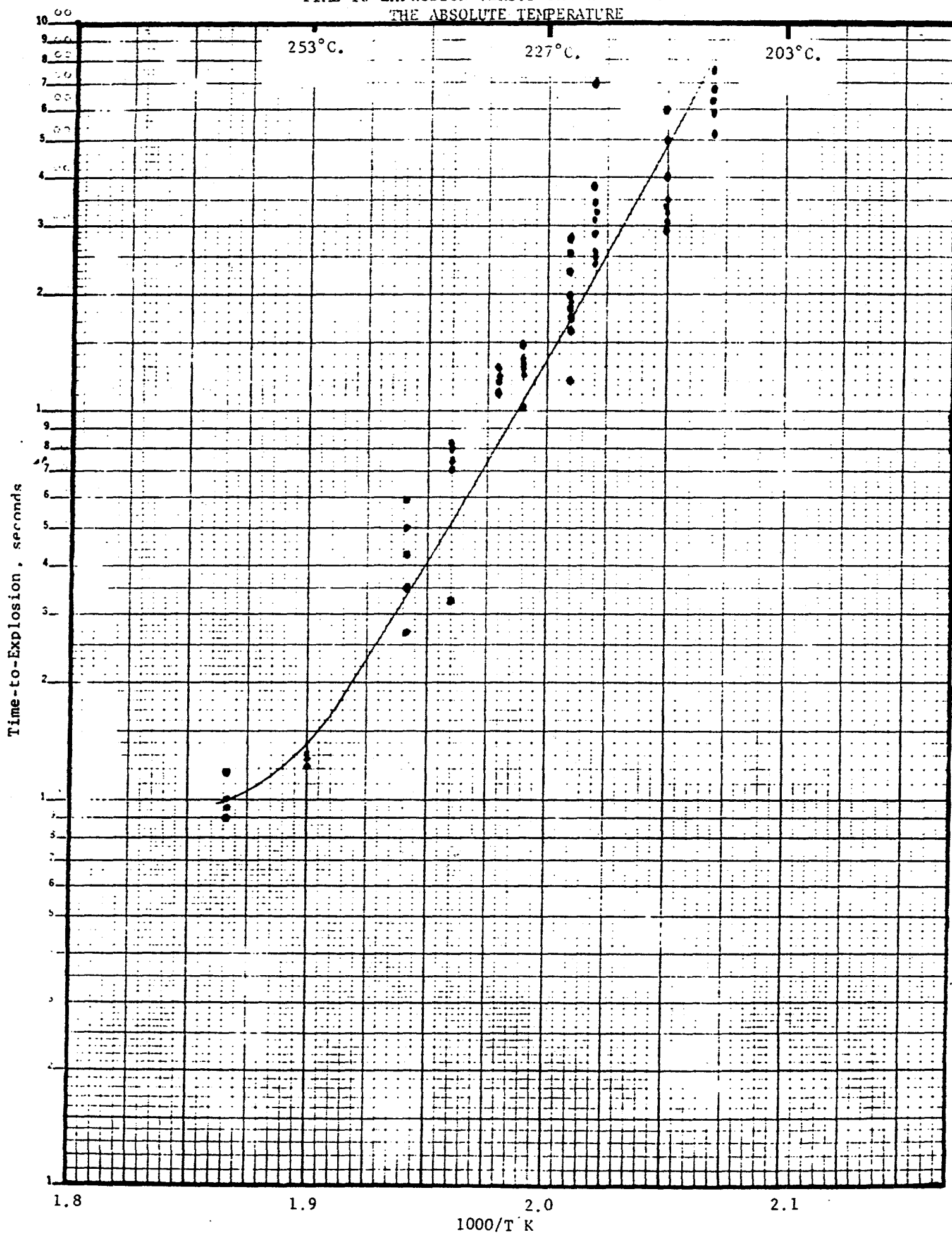


Figure 3

PBX 9404 + LEAD
TIME-TO-EXPLOSION VERSUS THE RECIPROCAL OF
THE ABSOLUTE TEMPERATURE

-23-

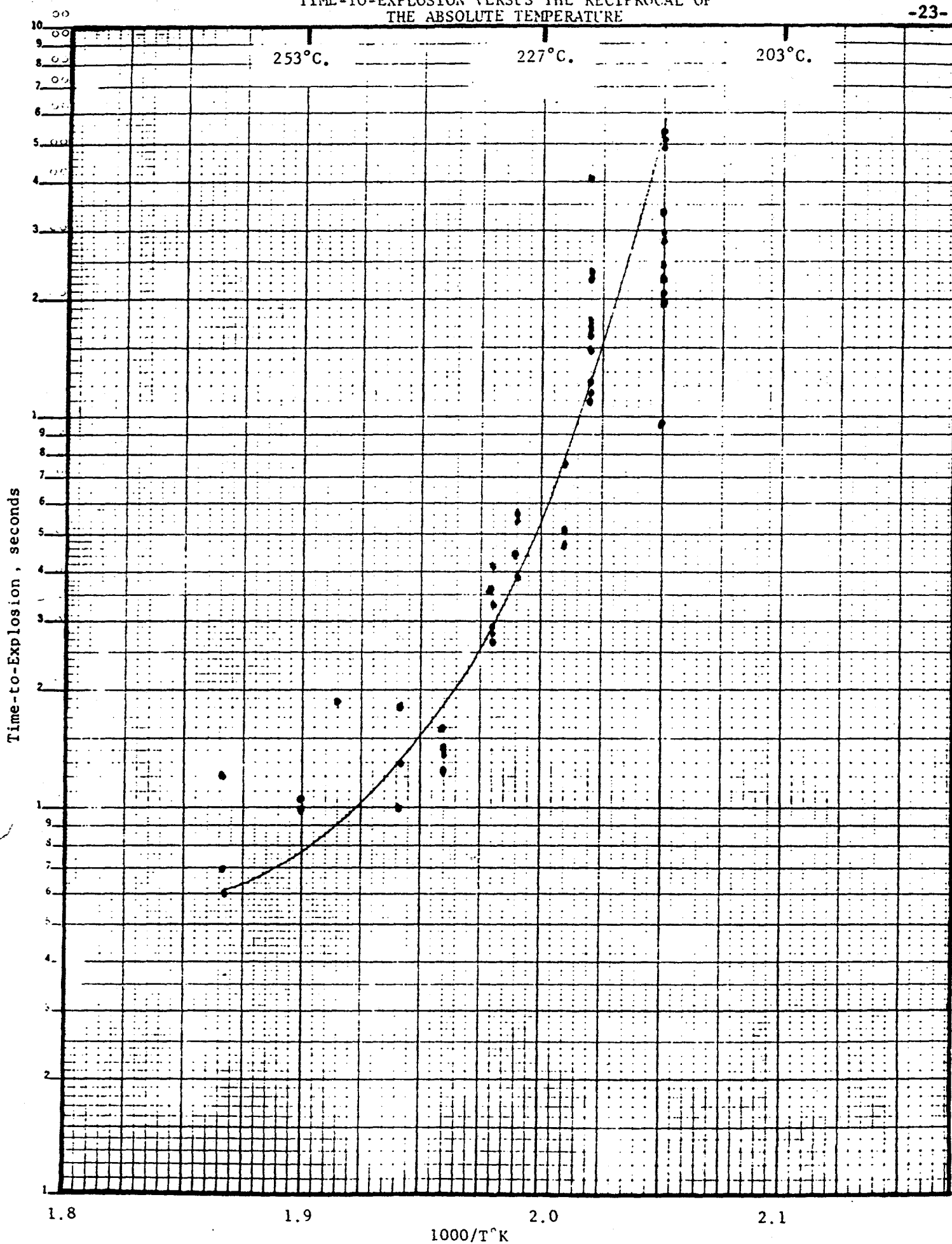


Figure 4

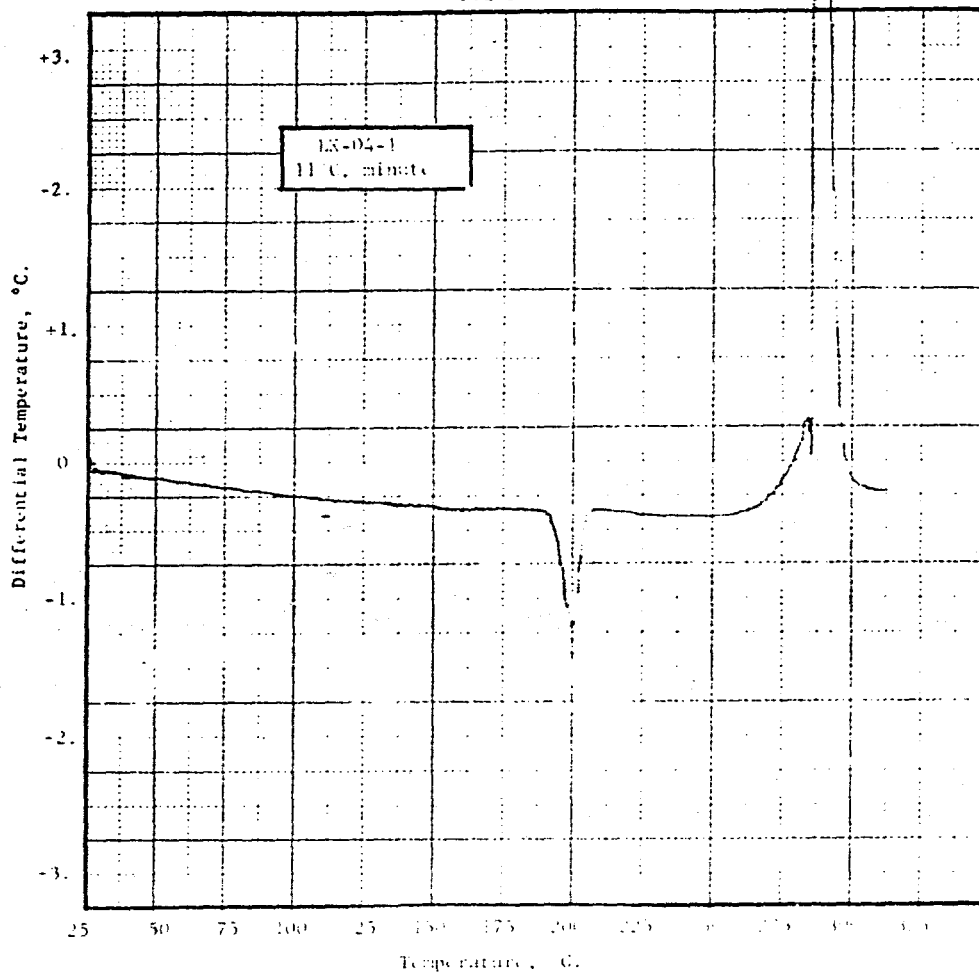
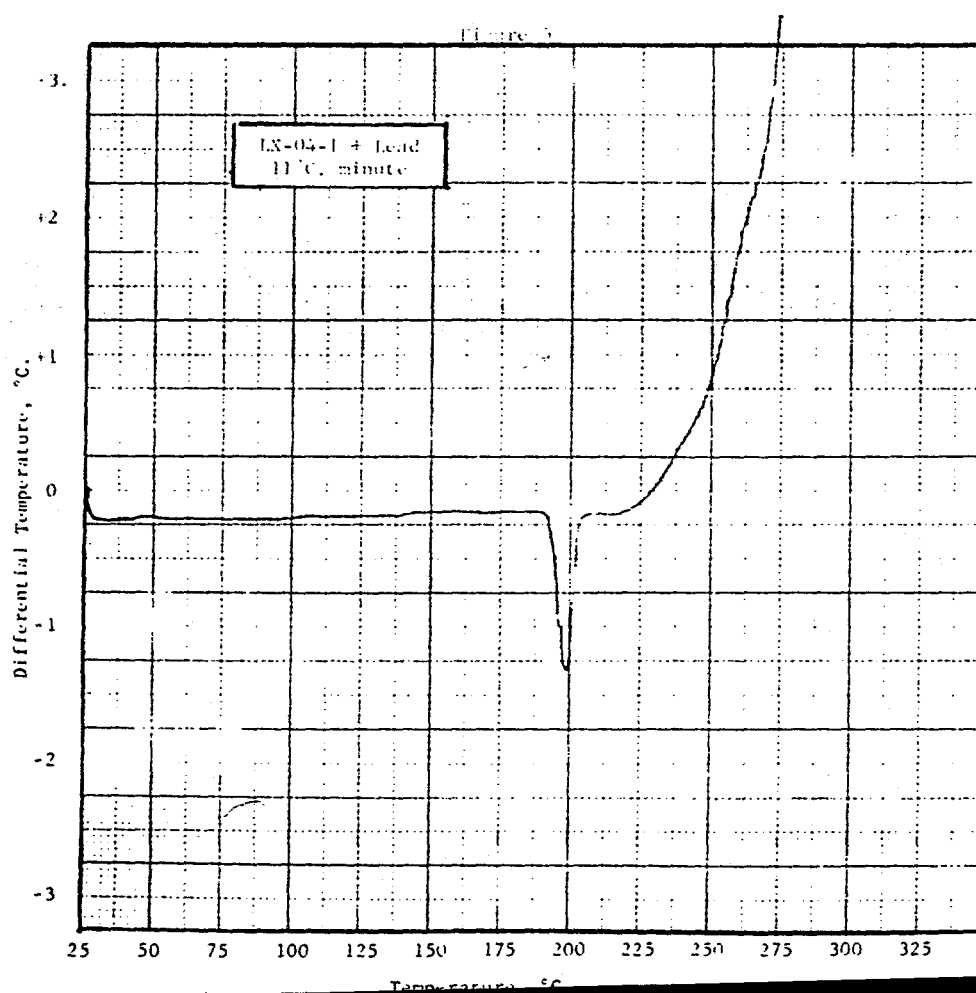


Figure 5



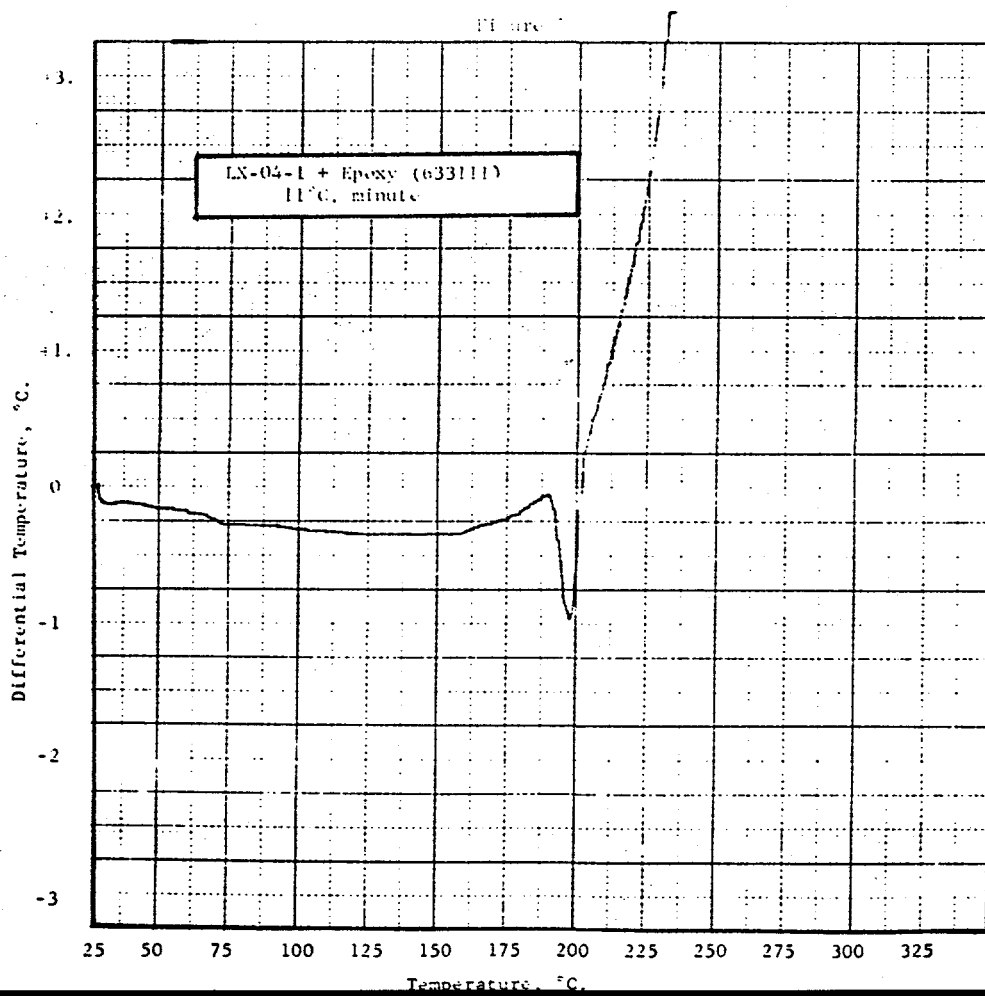
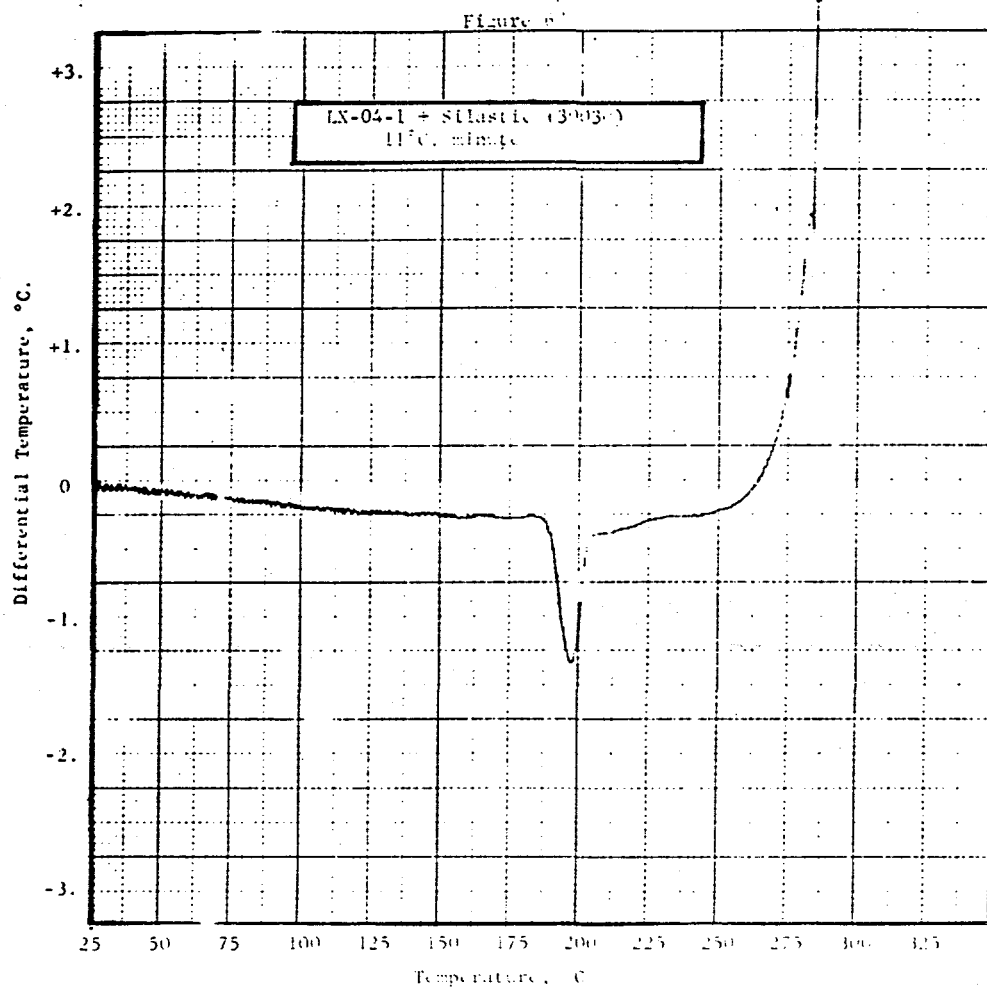


Figure 8

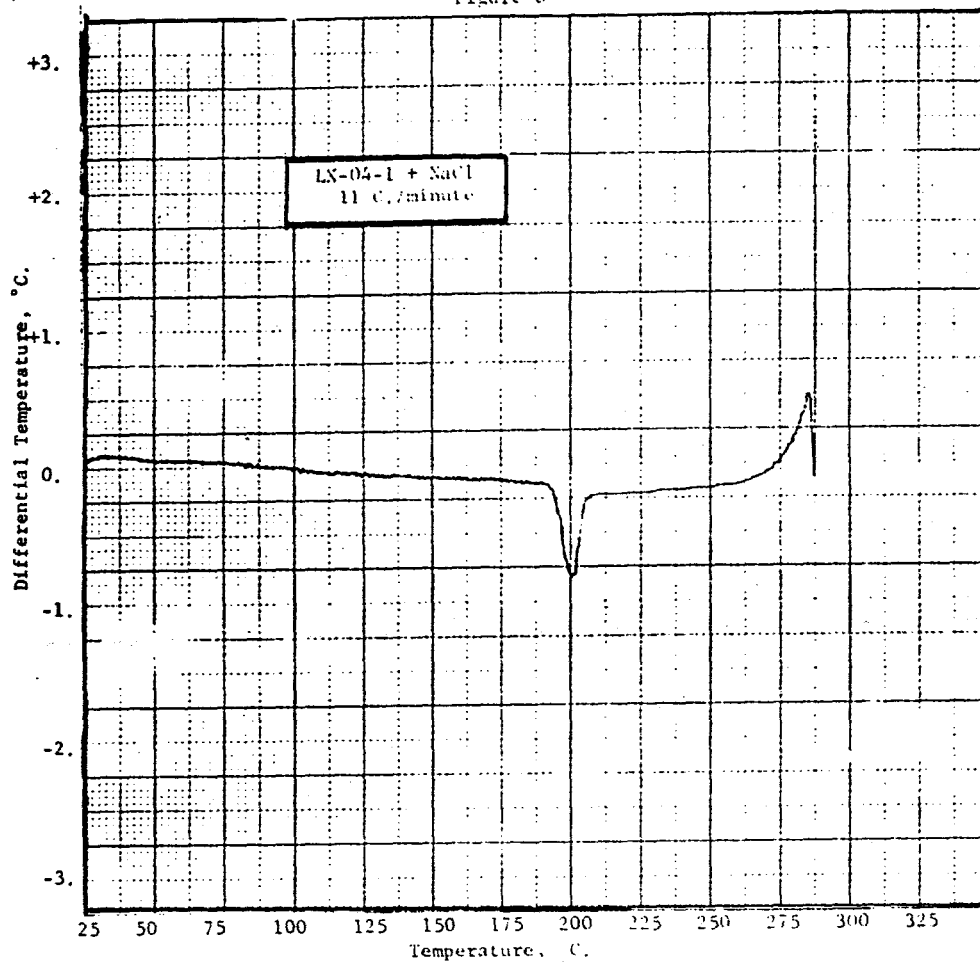


Figure 9

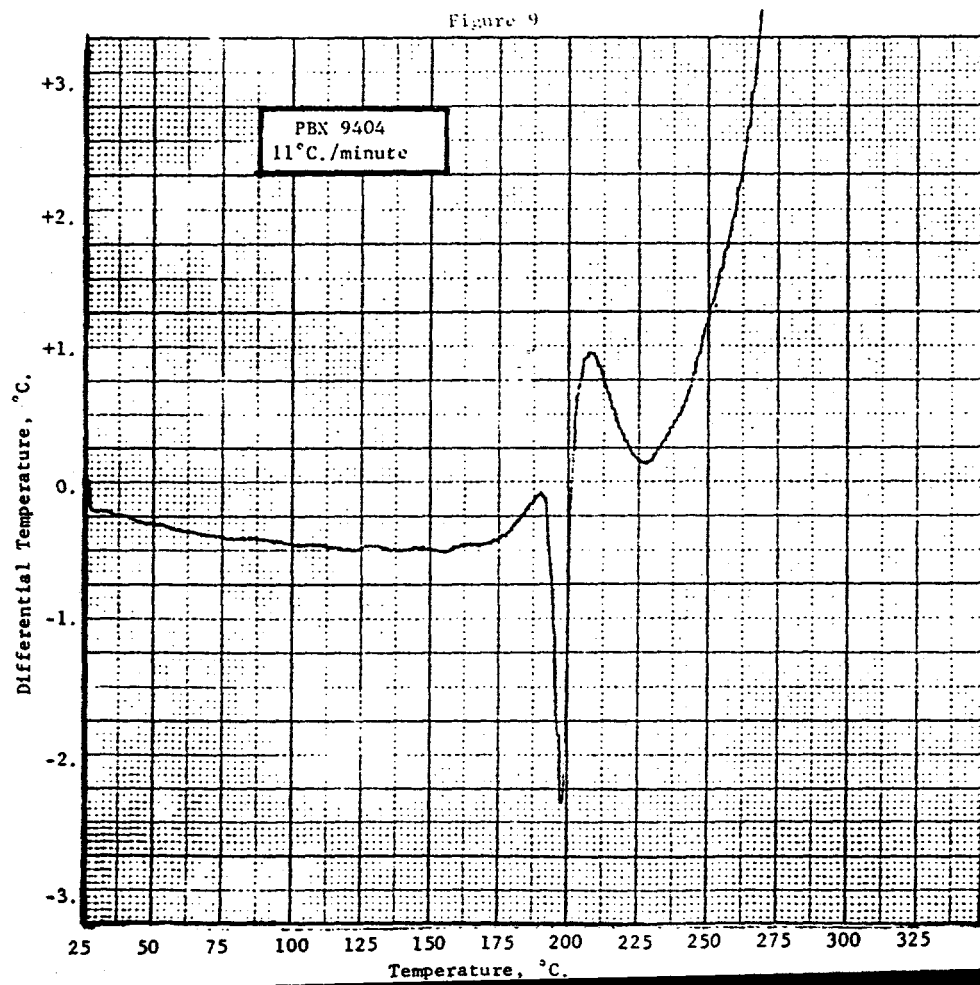


Figure 10

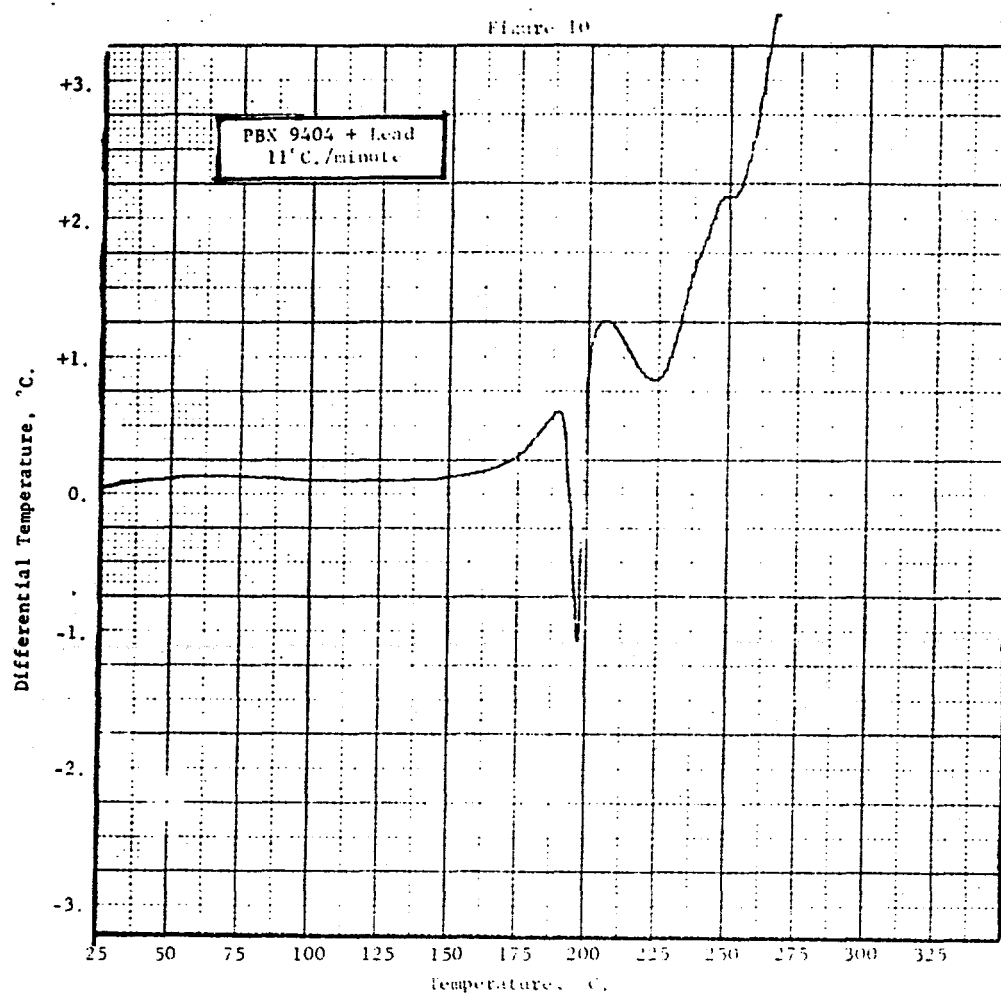


Figure 11

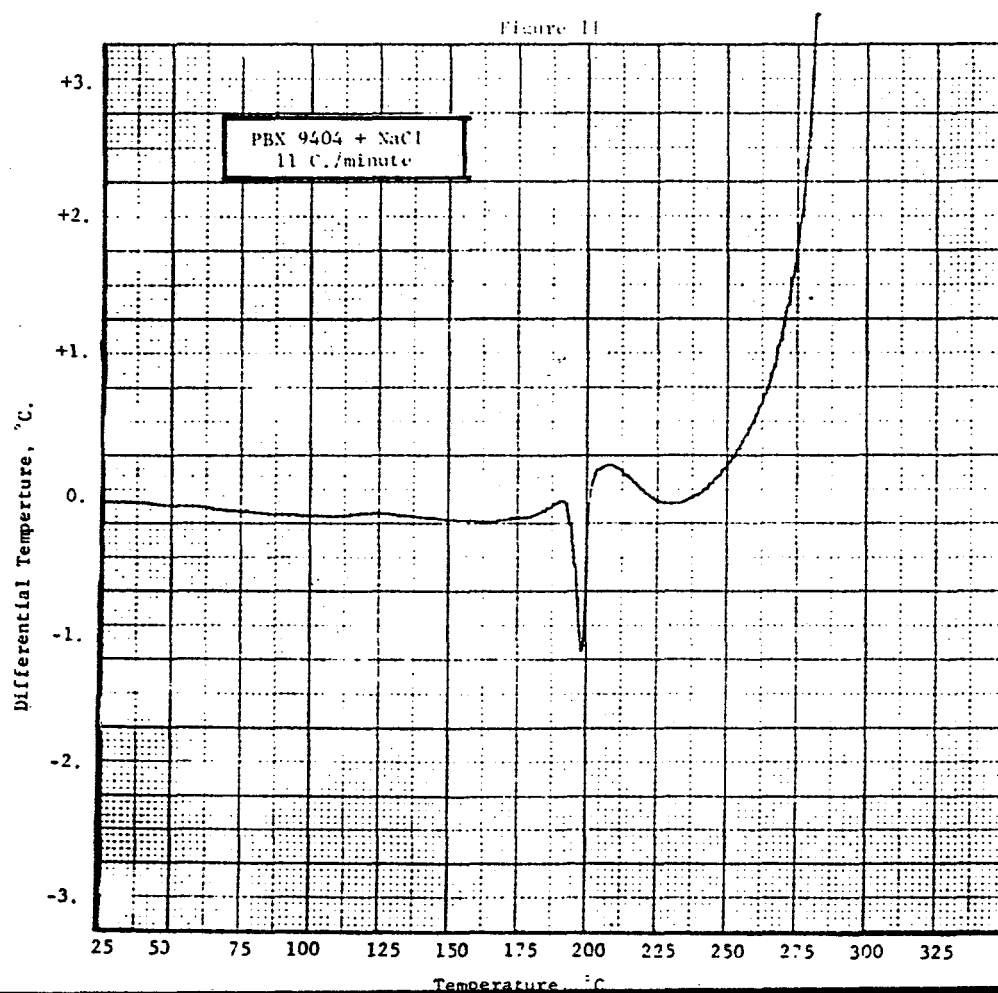


Figure 12

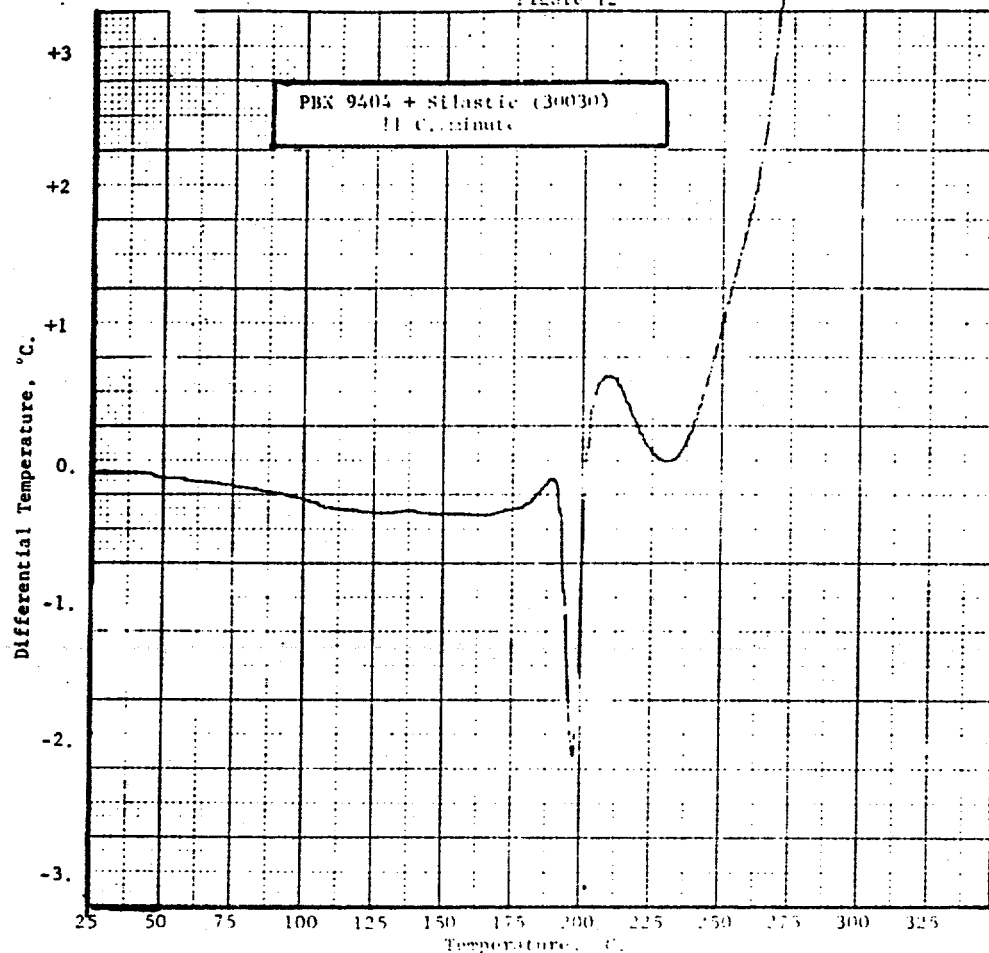
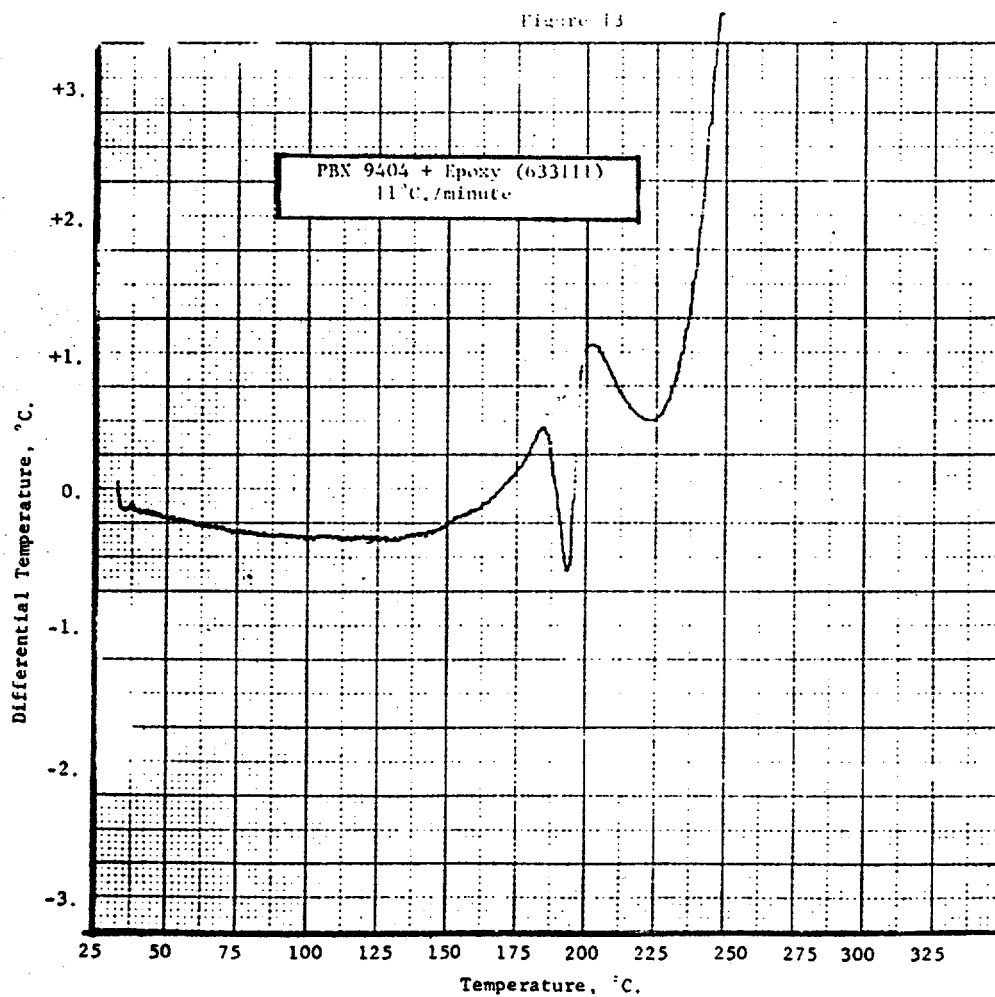


Figure 13



A P P E N D I X

EXPERIMENTAL DIFFICULTIES ENCOUNTERED WITH HENKIN TEST

Pages 1 through 7

DATA SHEETS FROM HENKIN EXPERIMENT

Appendix Data Sheets 1 - 74

Experimental Difficulties Encountered with Henkin Test

This section contains information to be considered in assessing the usability of the Henkin test as a routine quality assurance tool; it may also serve as a guide for future builders of Henkin test equipment (at least it will indicate some avenues not to pursue).

The major difficulty in getting the Henkin test in operation was the sample cap loading technique. Many different methods were tried at Pantex. The following is a chronological account of the major features of the evolution; it extends in time beyond the actual comparison experiment. The data sheets of all the Henkin work to date are reproduced in the appendix.

The initial plan was to adopt the method as reported by Rogers. His loading technique consisted of a #8 brass blasting cap (du Pont, 0.265 inch in diameter) loaded with a 40 mg sample; this was followed by a gas check (6.5 mm resized to 0.259 inch) with the convex side toward the sample, then a #00 cork and another gas check turned the same as the first. A punch and die was used to press the assembly to 300 pounds (6000 psi). The cap was then ejected from the die and crimped just above the first gas check with a dulled tubing cutter. A second cork was used at the top of the cap to hold a timing wire in place.

A variation in this loading method resulted from our ordering #8 blasting caps from Hercules; these turned out to be aluminum and were 0.22 inch in diameter. Twenty-two caliber (0.22 inch in diameter) gas checks and #000 corks were obtained for the aluminum caps. It was necessary to resize the gas checks so they could be easily pushed into the blasting cap; in systems where the gas check had to be

forced into the blasting cap, a much larger percentage of the caps developed leaks and failed to explode. This technique was used with Grade II, 98% pure HMX. The data (see appendix data sheets 1 & 2.) show that while some caps failed to explode, those that did gave reproducible times even when the sample size and loading pressure were varied. The failure to explode seemed to be associated with the corks. Just what happens to the cork when pressed was not determined; however, the cork could turn slightly causing the gas checks to tilt and not seal properly. The same technique was tried with PBX 9404; the results were horrendous (see data sheet 3).

To reduce the number of failures, another loading system was tried which used one gas check (convex side toward sample) and a 1/8 inch lead plug (data sheets 4 - 11). With HMX and LX-04-1, this system gave few failures and reproducible times-to-explosions. It would not give satisfactory results with PBX 9404. At 225°C., for example, two groups of times-to-explosion were obtained, one at approximately 25 seconds and the other at approximately 2000 seconds; intermediate times could not be obtained for PBX 9404 samples in the range 225 - 235°C. (this was thought to be where the time-temperature curve broke sharply). The brass gas checks were than aluminum plated and used with PBX 9404, but this did not change the results.

The samples of PBX 9404 that exploded in 25 seconds at 225°C. had a residue left in the cap. This residue was run in the DTA and gave a thermogram that compared to HMX suggesting that the nitrocellulose had caused the explosions.

Twenty unreacted samples were subjected to the normal nitrocellulose analysis; no abnormal amount or intersample variation of NC was observed. Other lots of PBX 9404 and different sampling techniques (total sample, sieve cuts, etc.) were tried, but they gave the same results. The short time (25 seconds at 225°C.) explosions were not as noisy as the longer times; this strengthened the pressure blow theory. To eliminate the pressure blows, different loading techniques were again tried. The first used a gas check, cork, gas check, and lead plug in that order. It was thought that the pressure buildup would compress the cork slightly, thereby increasing the volume and reducing the pressure sufficiently to maintain a seal. Some intermediate times, approximately 200 seconds at 231°C., were obtained which had not been obtained before, but the times ranged from 19 to 700 seconds (data sheets 20, 21). The next method consisted of pressing the H.E. in the cap, placing washers next to the H.E. and then sealing with a gas check and lead plug. This was to provide an air space so the pressure buildup would not be as great. This system gave short times, but no long times (appendix data sheets 23, 24). Then we decided to try more than one 1/8 inch lead plug to provide a better seal as well as some additional inertia and side wall friction. This gave no "extra long" times at 225 - 235°C.; the times were in two groups of approximately 20 and 200 seconds (appendix data sheet 26).

In an attempt to further strengthen the seals, the following was done: two gas checks were used; the first had the convex side turned away from the sample, the second had the convex side toward the sample. This was followed by two 1/8 inch lead plugs (see appendix data sheets 29 - 35). The results for the first ten samples were quite reproducible times of about 350 seconds at 230°C.

The first five were shot on Friday and the second five on the following Monday; the latter group all blew the side out of the cap. After these ten shots, the succeeding ones gave a large scattering of times at 230°C. While this turn of events was heart rending, we were able to maintain some feeling of progress.

At this time the construction of a Magneformer was completed. It permits crimping by sharply increasing a magnetic field around the caps and pressing the walls into the lead. A 1/4 inch long lead plug was substituted for the dual 1/8 inch plug arrangement; a crimp was made such that it would be on the lead plug. The various methods using lead plugs were re-evaluated by including magneforming (appendix data sheets 37 - 42). The results were generally improved, but a large deviation in time still persisted. At this point other parameters were varied in an attempt to define the problem, i.e., sample size and loading pressure, with very little improving effect noted (appendix data sheets 34, 35, 37). A few samples were tried in which an epoxy L933A was poured over the gas check-plug assembly. The adhesive was dried overnight and shot the following day. All these blew the side out of the cap; there was less deviation in the times than in previous loadings (appendix data sheets 36, 37). The approach was discontinued because of undeterminable side effects (CTE problems, heat evolution and/or heat sink problems, "thermal strength", etc.); it did indicate, however, that if the sealing assembly could be held in place, the explosions could be made to rupture the caps and give more reproducible times.

Following a discussion with R. Rogers, in which we discovered that our reproducibility goals were unrealistic (i.e., all previous work had been plagued by

considerable scatter), it was decided that the experimental approach and philosophy should be changed as follows:

1. Accept the fact that scatter and odd times are inherent in the experiment.
2. Realize that just a few trials of any system are not sufficient to evaluate that system. This is especially true since loading is an art and the operator cannot duplicate seals except after considerable practice.
3. Choose one or two loading systems that appear to have the best chance of success and work with them extensively (with a minimum of modification). One desirable feature would be that the assembly be strong enough to cause explosions to rupture the cap rather than blow the seals out the top. It turns out that a shortage of aluminum caps occurred at this time; brass caps were amply available. We decided to load as follows:
 - a. Load caps with H.E.
 - b. Follow with two gas checks; the first with convex side away from H.E., the second with convex side toward H.E.
 - c. Use a 1/2 inch long lead plug.
 - d. Press at approximately 7000 psi.
 - e. Magneform crimp the caps on the lead plug.
 - f. Use both aluminum and brass caps.
4. Choose a known incompatible mixture of H.E. and "inert", compare its times with the pure H.E. (i.e., run the time-temperature curves) and use this to check out the loading techniques. The mixture chosen was PBX 9404 + lead powder; the latter was supplied by Rogers.

In generating these time-temperature curves, the brass cap assemblies quickly proved superior to the aluminum caps (appendix data sheets 38, 39). As a matter of fact, the brass system sealed in the manner described above was deemed sufficiently satisfactory (appendix data sheets 40 - 51) to be used in the comparison test. The

comparison test data, which are quite good (in precision), are found in appendix data sheets 52 and 53. During the experiment, it was noted that mixtures containing lead, Silastic and epoxy consistently blew the seal out the top of the caps. The pure explosives and mixtures with NaCl ruptured the caps. By including a second 1/2 inch lead plug or a single 1 inch lead plug, all these systems could be made to rupture the caps (appendix data sheets 54, 55). In addition, these systems functioned as well (both in terms of reproducibility and absolute times) when the magneform crimp step was eliminated (appendix data sheets 54, 55, etc.). This latter is very desirable and helpful since magneforming is quite time consuming; all subsequent experiments have been performed without crimping (and the last described loading assembly).

A considerable number of experiments involving a wide variety of explosive systems have now been performed with satisfactory results (appendix data sheets 57 - 74). One one occasion, vendor supplied lead plugs would not seal well; this turned out to be caused by a surface lubricant (on the plugs). Cleaning in acetone eliminated the problem (appendix data sheets 58 - 60). Aluminum cap assemblies were tried again (appendix data sheets 57); these again gave poor results, probably because of their relative lack of strength. Some concern has arisen about the use of brass because of possible chemical reaction with the reactants. Experiments to date with gold plated caps and check have neither verified or rejected this concern (data sheets 68, 69).

The presently used cap assembly is as follows:

1. Load #8 brass blasting caps, 0.260 inch diameter with the desired explosive.

2. Resize 6.5 mm diameter brass gas checks to .0.259 inch diameter.
3. Slide a gas check into the cap with its convex side turned toward the sample.
4. Insert a 1 inch long by 1/4 inch diameter into the cap.
5. Press the assembly in a Carver Laboratory Press to 350 pounds; i.e., 7000 psi.

[illegible]

[illegible]

6-17-63 (6)

Uncon Program

TC

Temp inside

200 2.20 22.5
212 229 235
225 238 245

if

26.7 12.0 9.2
25.55 9.8 13.05
1144.4 15.25
1700.45 NE.

1.9 gas check
1.8 lead plug

777.8 10.2
9.45

965.15

150 d1

6-12-63

⑦

P.C.
T.C.

Inside

200	225
214	239
228	248

q₁₀

22.1	11.65
19.5	13.9
30.8	12.0
24.2	14.3
11.5	16.5
21.0	15.2

1900 check
1/8" plug

Temp ~ 230

4082.4	1251.45
1797.9	
1181.2	
1251.2	
1232.85	

L₁₀

1262.9

6-13-63

8

Pro.

T.C.

Sample Temp

150	170	185	200
162	183	196	210
171	192	205	225

227715 2850 2100 4
 2000 1800 2000

~ (4252)
 415645

1 gas check
 1" head plug

6-14-63

1

10

Handwritten: Handwritten

9404

1 one check
1 1/2 land plan

110

[illegible]

11

Pres.
TWC in bottom
branches down

234
244
255

240
251
263

1 X-cv11

670.2
655.45
636.85
633.1
812.3

481.8
128.8
234.8
143.7
89.55
296.8

1900 check
1900 plug

Σ = 679.8

396.4
339.0
297.5
150.7
223.25

174.25
84.6
94.6

[illegible]

6-14-63

13

Temp.

Avg.

Bottom

Inside

205

210

217

222

227

232.5

1 good checked

1 good plug

1/4

9404

7999.5 (NE)

(NE)

~ 4100

(Dried, Mach.

~ 4000 (NE)

13.7

Proden)

25.0

1700.0 (NE)

6-21-63

[illegible]

Data Sheet

[illegible]

6-25-63

12

[illegible]

6-26-63

19

[illegible]

1-2-63

(21)

8
3.5

9165 → 2255
9453 → 2233

23.7°

797.5

22.5

322.4

288.4

21.8

19.8

→ 2900 NE

71000

Acid rain cup

3 samples were loaded with Agardite
carb + loading. NE was spread
to room after 150 lbs
loaded at 150 lbs

3 samples were divided over night
... at 150 lbs

233

162.6

431.8

→ 2930 NE

→ 2900 NE

233

→ 2900 NE

3 samples were divided over night
loaded at 120 lbs

236

15.9

367.5

→ 2400 NE

3 samples were divided over night
loaded at 150 lbs

3 samples were divided over night
loaded at 120 lbs

7-333

22

		260				°C	
LX041	429.3					68.5	
	605.1					333.5	
	534.7					465.4	
	686.7						
	648.8						
	500.7					400.1	
	374.7					904.5	
	509.2						

LX041

7-10

25

245°

LX041

10.372

10.770 265

134.0

703.9

440.8

318.8

7-10-63
Next to wall
Apex in

NE 1081.0

49.6

NE 1007.3

17.8 } 2,000 260/300

Σ 14.9

Σ 18.5

27

Data Sheet 27

7-12-63

2

[illegible]

8-2-63

29

228.4

9404

weaker 321.6

22.9
 171.2
 16.9
 17.6

out gas
 checked,
 two plugs
 260-300

390.1

(one up -
 side down)
 405.1
 261.7
 298.2
 273.1

out gas
 checked,
 two plugs
 260-300

Data Sheet 31

8-7-63

32

235

320.4

9404 UGNE

820.1

290-2p

S tinged did
S tinted

?

250-300

44.1

21.5

536.9

15.5 } 190 new-2p

14.3 } 260-300

186.9

S 289-

290 (new)

S 217.4

2p-260-300

S 282.0

16.6

same - old imp

17.7

290 2p 260-300

41.6

290-3p

16.3

8-8-63

(all crimped)

(33)

234.5

9404

S

?

2gc-1p, 300

SN155 1gc-1p, 200

SN390 2gc-1p, 300

SN155

219

S

?

copper cap 300

SN67 1gc-1p, 300

17.4

8-9-63

34

9404

233

N25

NE 700. 19cr-1ply 300 2 crimps

231.5 Temp

SN 62. 19cr-1p-300-

S 19.4 " "

19.3 " "

21.5 9c-cable-gc-p-300

19.3 19c-2p

18.8 " "

Brave

SN 165 } 29cr-2p-269/300

238.6

S 235.8

Alum

429.4 29c-1p/19 260/arc 11,000v

207.5 29c-1p/19 260 25mg no crimp

295.2 " " " " " "

NE 400+ " " " "

NE 400+ " " " "

NE 400+ " " " "

346 " " " "

8-12-63

35-

9404

238

Wad 277.4

SN 142.

25mg

S 212.0

SN 138.

S 129.4

35mg

S 98.1

55mg

65mg

NE 325

15mg

29C-2P
200-300

Temp. cont.
R-6, C-9, 1-6.7

7-9-93

52

Temp. cont.
R-6, C-9, 1-6.7

Data Sheet 24

LX041

7-10

25

245°

LX041

10.372

10.770 265

134.0

703.9

440.8

318.8

7-10-63
Hart Co. Inc.
~~Appl. Co. Inc.~~

NE 1081.0

49.6

NE 1007.3

17.8 } 2.5 19.0 260/300

34.9

31.5

18.5

7-10-63

243 K-6, C-9, F-8, F-9

Data Sheet 26

		245°			231.5°		
9404		10.7	1st plug NE-260	NE	205.2		
		12.4	2nd plug 300		25.5		
		11.1	3rd plug 100	NE	220.3		avg matl 2nd plug
		12.9			151.2		260/300
2 plugs over: 12.1		16.5	avg plug all 4 plugs		19.5		
1 plug over: 16.05		17.9		NE	351.6		ground matl
	NE	15.9	avg matl		19.0		260/300 2 plugs
		16.9	avg plug 260		290		260/300 3 plugs
		12.4	avg matl	NE	238.7		100/200 2 plugs
		13.3	avg plug		19.6		1 plug
		12.6	avg matl				
		11.4	avg plug 260/300				
	NE	124.5					
		106.0	2 avg plugs				
	NE	208.3	avg plug - 125				
	average	20.9	avg matl				
		22.1					

27

Data Sheet 27

8-2-03

29

228°

9404

weak

321.6

eye gas
checked,
two plugs
260-306

22.9

171.2

16.9

17.6

cons up-
air flowing
two gas
checked,
two plugs
260-306

390.1

405.1

261.7

298.2

273.1

8-8-63

(all crimped)

33

234.5

9404

S

?

2gc-1p, 300

SN155

1gc-w-1p 200

SN390

2gc-1p 300

SN155

27.9

S ?

Copper cap
1gc-w-1p 300

SN67

" "

17.4

Data Sheet 34									
233									
9404	N25								
	NE 700.	19cr-1pug 300	2 crimps						
	231	19cr-1pug							
	SN 62.	19cr-1p-300-							
	S 19.4	"							
	19.3	"							
	21.5	9cr-1pug-9cr-1p-300							
	19.3	19cr-2p							
	18.8	"							
	SN 165	29cr-2p-26g/300							
	238.6								
	S 235.8								
	429.4	29cr-1pug	260/0r @ 11000v						
	207.5	29cr-1pug	260	25mg	no crimp				
	295.2	"	"	"	"				
	NE 400+	"	"	"	"				
	NE 400+	"	"	"	"				
	NE 400+	"	"	"	"				
	346	"	"	"	"				

8-12-63

35-

9404

238

WCA 277.4

SN 142.

S 212.0

SN 138.

S 129.4

S 98.1

25mg

35mg

29C-2P

260-300

55mg

65mg

NE 325

15mg

8-13-63

(36)

9404

239

S 281
70.5S 268
19S 273
70.5S 251
70.5S 288
70.5S 282
53.4S 250
53.4S 255
53.4S 237
53.4S 243
53.4S 215
53.4S 220
53.4S 15.6
53.4

28.9

NE 500

14.5

15.9

NE 100

NE 100

290-20

260-200

glue

290-10

260

glue

190-200

260

glue

290-20

260-300

260 glue

190-200

260 glue

37

235																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																				
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(38)

8-15-6

8-14-63

237

252.5

26.8

S 16.6

NE 56.0

S ~16

223.3

S 13

266.7

NE 49.5

S 22.2

S 13.5

S 12.3

S 14.4

279.7

S 11

NE 403

S 9.3

S 105

S ~11

S 214

S ~11

11.4

S 10.0

16.7

12.9

14.0

S 12.3

18.3

S ~13

S ~13

S ~10

10.5

290-1P
260290-1P
260290-1P
260290-1P
260290-1P
260290-1P
260290-1P
260290-1P
260290-1P
260290-1P
260290-1P
260290-1P
260290-1P
260290-1P
260290-1P
260290-1P
260290-1P
260290-1P
260290-1P
260

8-16-63

(39)

9404

225

229°C

652.7
approx. 100%
lost. not crimped

NE 4000 Plug had moved

(290-1p
260)

(alum)

18.2

lead

17.7

alum

16.5

19.8

290-1p
260

NE 1000 plug moved *

27.5

s

27.1

alum

28.4

23.4

s N/28

s 149.0

s N/35

NE 55

s N/33

s N/33

s 103.1

s N/27

s N/25

59.7

s N/56

54.9

44.1

not crimped
brass

290-1p
350

alum

alum

alum

18-19-63

40

[illegible]

8-20-6:

(41)

9404

2322 acc.

215

SN338

SN305

SN329

SN345

SN293

SN298

crimped

506.1

96.1

SN197

227.4

247.6

289.1

338

295.6

285.7

493.2

9404 + Pb

crimped

crimped

crimped

crimped

crimped

215

SN598. crimped

SN325

SN497

SN399

9404

N. 5.700

515

NE 700

NE 1150

NE 750

(cracked gas check)

9404 + Pb

8-21-03

(42)

9404

210

S~670 crimped

S~519

"

S~582

"

N.E. 1355

"

S~759

"

S~566 not crimped

S~659 crimped (30 sec dwell)

S~659 not crimped

N.E. 1300 crimped (30 sec dwell)

9404 + PH

N.E. 1135 crimped

N.E. 1300

"

N.E. 1300

"

N.E. 2000 not crimped

430.3 " 1" (10 min of PH)

395 " " (10 min of PH)

8-22-63

43

(R-6
C-9
F-5.2)

218°C

9404

R=6
C=10
F=2.0

238°C

R-6
C-10
F-1.6

233

R-6
C-10
F-0.8

229

S 119

S 111

S 122

S 121

S 128

SN324

SN692

SN341

SN312

SN286

SN375

N.E. 320

S 71

32.7

S 81

S 80

NE 750

162.2

165.5

S 407

175.3

227.9

231.3

N.E. 2800

SN16

SN14

12.5

SN14

26.8

S 36

S 33

S 29

S 41

S 28

96.9

S 144

[illegible]

45-

[illegible]

8-27-63

46

LX-04-1	(R _L C ₁₀ F ₄₆)		270°		(R _L C ₁₀ F ₄₈)		277°		(R _L C ₁₀ F ₅₃)		284°		(R _L C ₁₀ F ₄₀)		260°	
	S		S		S		S		S		S		S		S	
	30		36 av.		18		19 av.		10		11 av.		136		236 av	
	35				25				11				120			
	32				14				10				183			
	38				19				12				312			
	47				20				12				468			
													198			

8-28-63

(47)

LX-04-1

 $\left(\begin{array}{c} R_6 \\ C_{10} \\ F_{3.3} \end{array} \right)$

252.5°

S 428

S 777

S 466

S 935

S 758

673 a.u.

 $\left(\begin{array}{c} R_6 \\ C_{10} \\ F_{4.1} \end{array} \right)$

261.5°

S 253

S 197

S 262

S 164

S 192

214 a.u.

8-29-63

48

LX-04-1

(R6
210
F4.1) 262.5

84 au.
S 66
S 141
S 68
S 76
S 68

8-30-63

(49)

(R6
C10
F2.7)

246°

LX-04-1

S 596

S 821

Top 558

S 656

S 611

S 841

705

av

(R6
C10
F2.2)

242°

S 518

S 1110

S 1551

Top 702

S 972

S 552

941

av

9-3-63

50

(R6)
C10
F3.8

LX-04-1

260-
261

S

402

S

123

S

225

S

247

S

183

S

101

S

92

S

89

S

85

S

92

glue

92 av.

236 av.

1982

1967

231.5

235°

9404

130

162 av.

141

96 av.

190

167

91

130

83

173

86

81

9-6-63

(57)

R6
C10
F2.7

9404

245.5°

246

36.2

18.5

14.7

11.9 not crimped

19.9

T 14.0

T 13.8

T 13.5

T 14.4

T 13.5

T 15.2

T 13.5

9-9-63

242

242

T 15.3

not crimped

T 19.3

" "

T 22.0

Crimped

T 26.6

" "

T 16.5

" "

? 19.9

21.9

T 16.0

242.5

13.8

T 14.6

17.9

52

Date	Temp	7-12	9-13	9-18	9-20	9-24	9-11
LX-04-1	✓ 243°C	243.4	264°	264°	264	242	242.5
1	594.4	8	243.4	75.01	56.95	1014.7	767.9
10	539.8	11	131.8	270.46	218.710	1130.7	733.6
15	481.2	13	69.7	71.4	85.215	1093.8	704.1
20	709.5	18	53.4	117.2	78.523	702.2	724.8
28	735.9	20	88.2	54.201	62.525	616.7	737.0
LX-04-1 + Pb							
2	81.1	3	22.3	21.8	9.44	92.7	127.4
9	55.2	4	15.8	18.3	17.49	87.4	106.3
11	91.2	15	19.7	21.0	17.214	84.3	149.9
21	100.6	17	12.7	25.8	17.418	142.8	149.4
25	59.1	22	22.4	14.8	17.1	111.1	126.8
3	53.6	5	112.9	58.1	37.23	849.6	497.0
8	576.4	10	196.3	42.5	103.38	1003.5	477.1
16	528.1	12	374.3	144.1	125.813	848.8	483.3
24	495.4	19	288.2	204.4	273.822	447.7	586.2
26	402.3	23	98.6	42.8	36.724	767.1	609.2
LX-04-1 + Silashin							
4	175.6	1	44.1	37.4	32.62	195.6	149.8
7	218.5	6	17.3	40.2	21.27	196.2	191.4
13	98.1	14	22.2	55.1	25.612	228.3	252.1
18	312.8	16	38.8	50.8	24.817	159.9	199.7
23	160.7	25	37.7	36.8	22.620	110.7	151.0
5	17.5	3	15.0	10.5	8.61	19.6	18.7
6	17.1	7	12.9	11.9	9.06	17.9	21.7
14	18.5	9	8.5	18.4	8.411	18.9	21.4
19	16.4	21	9.9	16.8	10.216	18.4	18.2
27	19.2	24	11.2	11.7	9.014	19.4	30.9
LX-04-1 + E-poxy							
633111							

242.5
 242.5
 242.5

(53)

LRL	Date		9-10	9-16	9-17	9-19	9-23	9-25							
	Temp.														
9404	1	55.5	5	539.0	1	63.1	3	T	20.6	1	299.4	1	269.5		
	6	39.8	10	281.1	6	64.4	8		25.7	6	275.5	10	216.1		
	11	48.8	15	239.9	11	27.8	13		61.4	11	299.2	15	T	143.4	
	16	39.2	20	241.8	16	62.0	18		51.4	16	700	20	184.8		
	21	52.1	24	306.0	21	32.2	23	T	25.2	21	234.0	25	241.2		
9404 + Pk	2	11.7	4	T	169.9	4		T	16.2	2	586.7	2	111.8		
	7	12.3	9	T	209.3	9		T	12.9	7	106.2	9	148.5		
	12	14.6	14		143.4	14		T	14.6	12	112.2	14	270.5		
	17	16.0	19		157.6	19		T	14.5	17	114.9	19	150.8		
	22	17.2	23	T	143.5	24		T	11.8	22	102.6	24	111.6		
9404 + NaCl	3	81.5	3	NE	1500	2			118.7	3	467.5	3	T	277.2	
	8	85.5	8	NE	1000	7			74.0	8	226.2	8	T	261.0	
	13	102.1	13	NE	1000	12			74.2	13	232.8	13	NE	1000	
	18	60.0	18	NE	1000	17			71.4	18	421.8	18	T	296.2	
	23	68.8	25	NE	1800	22			76.5	23	720.	23	NE	700	
9404 + SILASTIC 30030	4	T	172.8	2	T	428.8	5	T	103.0	2	T	1000	4	T	397.8
	9	T	101.1	7	T	418.0	10		72.5	7	T	403.7	7	T	475.0
	14	T	95.2	12	NE	1000	15	T	58.4	12		338	12	T	374.0
	19	T	61.2	17	T	664.4	20	T	59.5	17	T	215.3	17	T	557.8
	24	T	51.8	22	T	477	25	T	76.4	22	T	246.3	22	T	272.2
9404 + EPOXY 63311	5	T	14.2	1		329.9	3	T	16.3	5	T	23.7	5	T	26.0
	10	T	14.6	6	T	22.0	8	T	16.0	10	T	22.8	6	T	23.2
	15	T	13.5	11	T	26.0	13	T	14.8	15	T	22.5	11	T	25.8
	20	T	17.6	16	T	24.8	18	T	15.5	20	T	21.9	16	T	25.0
	25	T	17.3	21	T	24.6	23	T	16.2	25	T	21.8	21	T	25.1
				Completion		Jot		Date							

(54)

220°

24.4

25.0

23.4

9404 + epoxy

124.8

98.3

112.2

9404 + lead

361.0

—

336.8

9404 + silastic

55

244C

19.5

19.3

24.4

242°

15.8

15.2

15.4

245°

25.0

44.3

34.0

242°

69.0

87.1

47.1

25.0

23.0

loaded with 2 Goodrich 1 long steel
plates and filled with fluorescent cement.
Gell out bottom

2 gc, 2 plugs 300/350
all out bottom
9404 + EPOXY (9-30)

2 gc, 2 plugs 300/350
all out bottom
9404 only (10-1)

9404 + SILASTIC (10-2)
all out bottom

9404 + Plt (10-2)
all out bottom

Date	11-11	11-12	11-13	11-14	11-18
Temp.	220.2	221.2	221	221	220+
9404	1 210.2				
	2 201.4				
	3 190.1				
	4 187.9				
	5 186.4				
	6 600 NE (LEAK)				
	7 213.4				
	8 211.1				
	9 210.8				
	10 186.2				
	11 239.9				
	12 207.1				
	13 2200 (time not stopped)				
	14 600 NE (cap. failed)				
	15 221.5				
	16 214.3				
	17 2200 (time not stopped)				
	18 217.2				
	19 213.6				
	20 206.6				
	21 175.0				
	22 194.4				
	23 196.2				
	24 153.3				
	25 178.2				

temp not accurate

56

57

11-19-63

9404 + epoxy

223°

29.0

28.4

32.2

30.9

31.5

28.4

28.7

30.6

30.5

33.0

aver. 30.4

2 qc, 1" @ 300 lb

Brass
caps

6000 psi

aver. 30.2

1 qc, 1" @ 300 lb

Alum caps

9404 11-26-63

22.0 T

33.8

413.5

273.6

1000 NE

9404 11-19-63

230

505.2

1000 NE

1 qc, 1" ~225 lbs
Al caps 6000 psi

11-20-63

9404 + epoxy

30.6

27.2 T

28.0

9010

PROP. REFLECT

(over)

Holliston

Holistic care

Prod. Receipts

(good)

11

Mad. Rivet

(b) (5) DPP

9010	12-6 210°	500 NE	12-6 224.5°	12-6 220°	12-6 215°	12-9 202.5°	12-9 204°
PROP. REFLECT (grey)							
Holocene			13.9 15.6	26.0 23.0 24.2 26.4 24.0			
Holocene					35.8 32.8 35.2 36.2 35.7 29.6 30.4 31.8 31.0 37.5		
Prod. Reservoir (grey)							
Holocene						118.6 650 NE 500 NE 113.0 115.1	IP 84.2 IP 83.8
Prod. Reservoir (grey)						600 NE IP 500 NE IP	IP 750.1

28

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* double wt.

9010

Hed. 511.1

12-10
204°

360 NE

96.2

101.0

12-10
208°

54.6

60.7

56.8

57.9

62.0

12-10
208°

420 NE

420 NE

420 NE

420 NE

420 NE

12-11
208.5°

43.8

45.1

48.8

48.8

48.8

12-11
205°

66.0

82.5

360

360

360

Prod. Point (gach)

1P 1380 NE

1P 500 NE

1P

1P

1P

1P

2G 1P

1020 NE

540 NE

420 NE

80 *

418 *

540 NE

59 *

420 NE

513 *

420 NE

525 *

300 NE

72.8

70.2

67.8

1P 19 *

1P 19 *

1P 19 *

330

300

360

1P 19 NE

1P 19 NE

1P 19 NE

Production (white)

plump wren

not cleared

with a. s. t. o. n

Discolored

9010

Holocene

NE 1200
NE 300
T 5342

live
and
dead

458.1
464.1
NE 660

12-13
205°

NE 1200
NE 300
T 5342

w
w
w

12-13
205°

397.8
309.0
477.2

84.8
89.2
104.7

97.8
102.7
88.2

temperature
fluctuating
between 20
(net 202-205)

Discarded

459.4
449.0
477.4

$$(v = 75 \text{ m/s})$$
$$(V = 100 \text{ mm}^3)$$
[illegible]

[illegible]

63

		249°C		258°		265°				259.5°		253°
5269-				NE 650								
3270	w 560											
Baratol						60.6				79.0		113.0
						59.9				77.8		112.6
						63.8				78.7		114.6
						64.2				80.8		106.9
						65.6				74.6		123.4
						61.4						
3317						131.0	50 mg			159.5		255.0
INT - Wilson A -						83.4	75 mg			164.5		229.8
Ba(OH) ₂ (50/10/100)						90.0	100 mg			172.5		236.2
						111.4	50 mg			168.3		246.0
						141.6	50 mg			132.0		239.4
						135.0	50					
						131.6	50					
TART						650	NE					
						1195.0						
						1500.0	NE					

64

940H (Doug. H.)

Control

240°

13.4

15.1

14.2

37.0 ✓

13.8

14.2 ✓

220°

169.2

186.6

160.4

178.6

161.1

17.2

17.2

17.2

10275C3101

38.6

23.2 ✓

36.6

37.7

38.9

140.4

152.6

171.2

167.5

131.4

152.6

152.6

152.6

10281C3101

10.6

10.9

13.0

14.0

16.2

125.6

135.4

174.6

143.2

165.5

148.9

148.9

148.9

these were
preserved
(940H)

POP 419

t and $\frac{1}{2}t$

[illegible]

27

Data Sheet 66									
9404	230°		225.5°						
	62.5		97.4						
			92.9						
			83.8						
			90.7						
			119.0						
			90.2						
9404(84% humidity)									
			89.1						
			86.2						
			96.1						
			92.8						
			97.0						
9404(60% humidity)									
			78.6						
			92.5						
			98.0						
			96.9						
			92.0						

(67)

HMX

230°

T 174.6

245°

88.6

93.2

225°

T 256.1

91.7

88.3

100.6

5.26

HMX (8 schmidt)

200 NE line

94.8 T

86.0

82.0 T

96.4

88.6

8.88

HMX (6 schmidt)

90.3 T

200 NE

90.9

98.4

89.5 T

111.3

1.96

Broad canal

[illegible]

04-000000

Date	Time	Lat	Long	Alt	Dist	Wind	Sea	Temp	Humid	Pressure	Remarks
10/10/21	11.0	152.2	140.8	307.2	9						
	17.6	141.0	975.1	261.4	8						
	19.8	161.5	847.2	319.0	7						
	19.4	121.8	686.2	448.8	6						
	23.0	139.0	1407.5	484.1	5						

Brasscap

230°

67.7

30.0

272

872

26.0

250°

10.8

1460

13.3

12.8

16.2

Walter C. Hall

23.7

29.6

17.

2.1.6

Black

13.6

14.4

29.51

15.0

16.7

1950

1133.6

8623

893.6

938.5

937.2

750

07.8

87.2

91.4

93.0

9.85

07.6

305

96.1

11	11
----	----

01.5

		0.556
--	--	-------

[illegible]

191°

Q-7A061
277.1
110082

9

245

20

H11X

H11X

255°

49.1

47.9

47.8

82.7

79.6

80.9

127.1

121.9

137.8

127.0

126.8

121.8

248°

240°

Alkilled Bridgmanite

89.0

96.7

114.9

155.4

172.3

143.8

217.3

206.8

227.2

228.8

211.9

211.4

Unaltered Bridgmanite

53.1

54.6

55.3

79.1

72.0

79.7

107.9

104.3

107.4

118.0

100.1

109.8

79.6

109.8

(72)

HPLX (LRL)	230°		245°		260°	
A-315	200.7 T		107.0		27.9	
B S _a C	245.2 T		110.1 T	N	38.0	
	229.8	6.801	110.9 T	S	33.0	
	273.8		111.1		38.7	
	233.6		105.5		13.5	
A-316	254.0		139.7		28.6	
B S _a F	270.6	8.7	95.6	0	27.5	
	256.2	9.1	121.6	S	29.7	
	216.2		114.0		31.5	
	208.2		113.2 T		35.6	
T - Phone blew out trap						
W - available						
NE - no observation						

74

		240°							240°			
9404												
~25 mg		56.8										
~75 mg		14.2										
old. allug		23.2										
300#		14.4										
24 mg	T	43.5										
48 mg		42.6										
73 mg		12.6										
58 mg		12.0										
53 mg		17.8										
55 mg		13.2	T	38.7	13.8							
57 mg		17.5		29.6								
65 mg		15.6		13.2								
49 mg		39.6		33.6								
50 mg		22.3		25.1								
75 mg				14.1								
99 mg				21.6								
B3-569-61		14.9										
~60 mg		14.4										
		14.8										
B1-521-61		17.2										
~60 mg		14.6										
		17.0										

brass cups

72

[illegible]

8-13-63

(36)

H0404

239

SN 81

SN 68

SN 73

SN 51

SN 88

SN 82

SN 50

SN 55

SN 37

SN 43

SN 15

SN 20

S 15.6

28.9

NE

500

14.5

NE

100

NE

100

290-2P

260-200

blue

290-1P

260

blue

190-200-P

260

blue

290-2P

260-390

120-9-blue

190-200-P

200-200-blue

4933A

exp 044