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B. 20

THERMAL PROPERTIES OF EXPLOSIVES

The purpose of this project is to determine the thermal properties of explosives and to continue the development and evaluation of thermal tests.

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DISCLAIMER

ABSTRACT

Henkin's test data are reported for comparisons of the following: dry-to-moist samples, PBX 9404 in brass and gold-plated blasting caps, Holston HMX with Bridgewater HMX, LX-04-1 and LX-04-1 + Ucon oil, and PETN, LX-04-1 and Extex. The time-to-explosion curves for HMX and PBX 9404 are also given.

A description of the pyrolysis apparatus and the method of calibrating the sample temperature to the response of the thermal conductivity detector are reported. The pyrolytic decomposition curves of several standard explosives and six specially prepared HMX samples (LRL raw material #A-311 through A-316) are included.

A controlled atmosphere D.T.A. is described and the thermograms of PETN with an atmosphere of air at 85 psi, nitrogen at 85 psi and 200 psi are given. The thermograms indicate that PETN becomes more sensitive as the pressure increases.

Chemical reactivity data are reported for Comp B, Comp B-3, Comp C-4, HMX, PBX 9011, PBX 9205, Tetryl and TNT. Also, test results are reported for LX-04-1 and Comp B-3 heated at 150°C for 22 hours, LX-02-1 heated at 100°C for 22 hours, and pressed pellets of PBX 9404 and PBX 9404 + powdered lead.

PREVIOUS APPLICABLE WORK

A number of techniques for measuring and evaluating the effect of thermal stimuli upon explosives have been installed, and in some cases modified and developed from the work of others.[1,2,3] These include DTA, Henkin and pyrolysis tests. Gas chromatograph results have been reported, as was an extensive experiment comparing the Henkin and gas chromatograph tests. Other work such as measuring the thermal diffusivity of explosives and the glass transition of plastic materials have been included in this report.

DISCUSSION

Henkin Test

An experiment was started to determine the effects of the moisture content of explosives on the Henkin test results. The explosives used in this experiment were PBX 9404 and HMX. The explosives (thinly spread on a dish) and the unloaded blasting caps were kept in a controlled atmosphere chamber of known humidity (8% and 60%) for 24 hours. The H.E.'s were measured into the blasting caps and immediately sealed with a cork until the gascheck and lead plug could be put into the cap and pressed. At this time, only one temperature has been used to make comparison of dry-to-wet samples (Table I). The moist ones sometimes blew the lead plug out the top rather than rupturing the side of the case, while the dry ones did not.

The comparison of the time-to-explosion in brass blasting caps and gold-plated blasting caps has been completed for PBX 9404. The blasting caps were sent to a vendor who did the gold plating and the gaschecks were vacuum plated at Pantex.

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[1] Pantex Quarterly Report for April, May, June, 1963
[2] Pantex Quarterly Report for July, August, September, 1963
[3] Pantex Quarterly Report for October, November, December, 1963

The brass and gold-plated caps were randomly sequenced and tested at different temperatures. The data with the harmonic means of the time-to-explosion are given in Table II. A graph of the harmonic mean as a function of the reciprocal of the absolute temperature is shown in Figure 1. The gold caps are probably less reactive than the brass, but the differences may not be worth a change.

LRL sent Pantex samples of ball-milled and unmilled Bridgewater HMX and requested a series of tests to compare the sensitivity of Bridgewater and Holston HMX's. The Henkin test was used as one means of comparing the thermal sensitivity of the two Bridgewater samples with Holston HMX. The time-to-explosion data (Table III) show a difference between the milled and unmilled Bridgewater samples. Because of this difference, the time-to-explosions at different temperatures were determined for Bridgewater samples of known particle sizes, as well as for samples of Holston HMX (Grade II, Class A, B, D & E). These are given in Tables IV and V, respectively. The time-to-explosion curve, determined from the harmonic mean of the times for Class E HMX, is given in Figure 2.

An in-plant problem involving sensitivity and compatibility of a liquid was undertaken. The liquid was Ucon oil, which is to be used in a new press pumping station. It is reported here because it is quite different from any system previously used, and contrary to past experience, the Henkin results do not agree with the D.T.A. results. To load the blasting caps, two drops of Ucon were put into the cap, followed by 50 mg of LX-04-1. This was sealed in the usual manner (using gascheck, lead plug and pressing to 7000 psi). Included are the Henkin data (for LX-04-1 and LX-04-1 + Ucon) and D.T.A. thermograms (for various H.E. and H.E. + Ucon) in Table VI and Figures 3 through 9, respectively.

The formulation facility has made a sample of Extex, a LASL explosive, and to check its thermal sensitivity, a comparison of the Henkin data (Table VII) for Extex, LX-02-1 and PETN were made. These are of interest to this report because the samples are different from any that have been used before and, again, the Henkin results do not agree with the D.T.A. results (Figures 10, 11 and 12).

Pyrolysis Apparatus

The pyrolysis apparatus was constructed quite similar to the instrument design by Rogers, Yasuda and Zinn[1]. A complete description will be given in this report for the convenience of the reader.

The pyrolysis block (Diagram I) is the heart of the instrument and was constructed with a cooling jacket to permit more rapid cooling after a pyrolysis run. In order to be able to heat the sample at fairly high rates (7.9°C/min), two 750-watt cartridge heaters (Watlow Firerod Heaters #L3E5A) were used in place of the two 180-watt cartridge heaters suggested by Rogers. The heating rate of the pyrolysis block was controlled by a linear temperature programmer (F & M Scientific Corporation, Model 40).

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[1]Rogers, R. N.; Yasuda, S. K.; and Zinn, John; Anal. Chem. Vol. 32, 672 (1960)

The combustion chamber (Diagram II) is completely different from Rogers and uses a $1\frac{1}{2}$" x 3" x 6" stainless-steel block to hold two 750-watt cartridge heaters and a 7-inch nickel pipe filled with a mixture of firebrick and copper oxide (a 1 to 1 volume mixture of 20 to 40 mesh copper oxide and 40 to 60 mesh firebrick is used). A small hole is drilled in the stainless steel block for a thermocouple which monitors its temperature.

The thermal conductivity detector (Diagram III) was constructed according to the design suggested by Felton[2] and uses model airplane glow plugs (Champion Model VC-1). The power supply for the thermal conductivity detector is a 6 - 12 volt battery charger and the output voltage is controlled by a powerstat. The bridge circuit for the detector is shown in Diagram IV.

The gas flow rate is monitored by a Brooks flowmeter (Sho-Rate 150). Diagram V is a block diagram of the components of the pyrolysis apparatus.

The procedure for operation is as follows: After the combustion chamber has been set to operate at approximately 700°C, a 10-mg sample (or for mixtures 10 mg of explosive and 10 mg of "inert" are used) is weighed into a small stainless steel boat and put in the pyrolysis chamber. The helium carrier is turned on and the needle valves are set to maintain a 15-psig pressure with a flow of approximately 25 cc/min. The voltage on the thermal conductivity detector is set at 1.5 volts, the bridge circuit is balanced, and when it will remain balanced, i.e., when all the air is purged from the lines, the temperature programmer is turned on to start the cycle.

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[2]Felton, H. R.; Buehler, A. A.; Anal. Chem. Vol. 30, 1163 (1958)

After a run is completed, the bridge voltage and pyrolysis-block heaters are turned off and air is allowed to flow through the pyrolysis block and combustion chamber to regenerate the copper oxide. Air is also blown through the cooling jacket of the pyrolysis block until the temperature has dropped to approximately 100°C; at which time, cold water can be used to complete the cooling of the block.

The pyrolysis apparatus has become operational this quarter. The pyrolysis curves (Figures 13 and 14) and D.T.A. thermograms (Figures 10 and 15) of PETN and EL-511 Beta were used to correlate the sample temperature with the thermal conductivity detector responses.

LRL sent Pantex six samples of specially prepared HMX (made for LRL by Holston, LRL raw material No. A-311 through A-316) and requested the pyrolytic decomposition curves. The samples were made with variations in the normal Holston process. For these six samples, the pyrolytic decomposition curves, D.T.A. thermograms, and Henkin data are given in Figures 16 through 21, Figures 22 through 27 and Table VIII, respectively. These may be compared to the Henkin (Tables III, IV and V), pyrolysis (Figures 28, 29 and 30) and D.T.A. (Figures 31, 32 and 33) data for Holston-made Bridgewater process, milled and unmilled HMX's. A number of pyrolysis curves of standard explosives have been generated. They are not included in this report, but have been incorporated into our H.E. Thermal Handbook for reference, and available upon request.

<u>D.T.A.</u>

The design of the controlled atmosphere/pressure D.T.A. is considerably different from normal D.T.A.'s; therefore, a description of the apparatus will be given.

The pressure chamber (Diagram VI) was designed for 3000 psi at 500°C and is a stainless steel tube (1.490" O.D. and 12" long) using an "O" ring seal between it and the base plate. The base plate provides the gas inlet and outlet systems (an outlet enables runs to be made using a gas flow) and has a series of holes to permit water to be circulated near the "O" ring to keep it cool; however, the base plate remains cool with this design and the water has not been used.

The thermocouples (see insert in Diagram VI) were made with #28 gauge glass-insulated chromel-alumel thermocouple wires; to insure that the thermocouple could not be grounded near its junction, a one-inch piece of creamic thermocouple insulator was used inside the stainless steel tubing. The thermocouple wires were silver soldered to a 1/16" thick brass plate which was, in turn, silver soldered to the end of a 1/8" piece of stainless steel tubing of the desired length (10 inches). The 1/8" tubing was fastened to the base plate with Swagelok fitting.

The sample holders (see insert in Diagram VI) were made from a $\frac{1}{4}$" length of 3/16" stainless steel tubing which fits tightly on the thermocouple tubes. The furnace consists of a $1\frac{1}{2}$" I.D. by 12" long combustion tube helically wound with a 1/8" coil of 20-gauge of nichrome heater wire (total resistance of the heater is 25 Ω). The furnace is mounted in a container filled with Vermiculite insulation. The furnace assembly can easily be removed from the pressure chamber to reduce the cooling time.

The electronics include a Moseley Autograf X-Y Recorder and a Leeds & Northrup Microvolt Amplifier #9835b.

A few thermograms were made with the controlled atmosphere D.T.A. using PETN with atmosphere of air and nitrogen. These are given in Figures 34 through 39.

A large number of thermograms have been generated with the conventional D.T.A. this quarter, including all samples for which Henkin data and/or pyrolysis curves were requested. There are too many curves for all of them to be published here; only those of specific interest to LRL have been included.

Chromatographic Work

The second heating bath for the chemical reactivity test was used this quarter to obtain data (Table IX), requested by LRL, for LX-04-1 and Comp B-3 at 150°C and LX-02 at 100°C. Some new chemical reactivity data (Table X) has been generated this quarter using pressed pellets of PBX 9404 and PBX 9404 + powdered lead. The chemical reactivity data for Comp B, Comp B-3, Comp C-4, HMX (~ 98% pure), PBX 9011, PBX 9205, Tetryl and TNT to be used in the LRL H.E. Handbook are given in Table XI. Also included, are reactivity data for comparison of Bridgewater, Holston and UK-simulated (A-311 - A-316) HMX (Table XII). To increase the output (samples per day) of the gas chromatograph, a separate manifold has been constructed so that the sample tubes can be evacuated and filled with helium without interfering with the chromatograph in any way.

Some of the new sample tubes are in operation and seem to be satisfactory. The volume in the sample tube has been reduced by inserting a glass rod (3/8" diameter and 3 3/8" long) in the space above the samples.

The construction of a single-column chromatograph at Pantex has started. Most of the components will be like those in the existing chromatograph which will enable new ideas for the existing chromatograph to be tried and tested on the single-column chromatograph. Plans are to have the single-column chromatograph in operation by the end of June.

Glass-Transition Temperature

Last quarter, the Glass-Transition Temperature (Tg) of Plexiglas, using a change in capacitance method, was determined to be approximately 140°C. This quarter, an apparatus was constructed to measure a volume change as a function of temperature. The apparatus (Diagram VII) consisted of a 2-mm capillary tube glued to a steel sample chamber. After the sample has been placed in the chamber, it is filled to the desired height with mercury. The assembly is placed in a controlled-temperature bath (using mineral oil for the liquid and controlled by a Fisher Temperature Control, Model 44), and the height of the mercury column is measured at a given temperature with a cathetometer (Gaertner, Model M-911). The coefficient of volume expansion of mercury is virtually constant (1.8116×10^{-4}/°C) for temperature range 30°C to 170°C. Therefore, the volume change of the sample is directly proportional to the change in the height of the mercury column. Ample time was allowed at each temperature for the sample and bath temperatures to equalize. Figure 40 is a graph of the mercury column height as a function of the sample temperature. No sudden transitions or inflection points, which would be indicative of Tg, are observed.

FUTURE WORK; COMMENTS; CONCLUSIONS

The final detailed report of the comparison between the Henkin and gas chromatograph chemical reactivity tests has been published and distributed to LRL and LASL.

The data (Table I) for the experiment, to determine the effect of the moisture content of an explosive on the results of the Henkin test, indicate that samples of HMX and PBX 9404 containing moisture had more explosions that blew the lead plug out the top of the cap rather than rupturing the side of the cap. It is believed that sometimes the vaporization of the moisture causes enough pressure to move the gascheck and lead plug; this causes the sealing system to be weakened. When no leaks occur, the explosion blows the plug out the top; if a large leak occurs, there is no explosion. In future tests, other temperatures and environment humidities will be tried.

The time-to-explosion data for PBX 9404 (Table II and Figure 1), in the brass blasting caps and the gold-plated caps, suggest that some difference in reactivity of the H.E. with the two metals occurs. The gold caps are the more inert since the times-to-explosion in them are essentially the same or less than in brass over the usable temperature range. Whether this difference is sufficient to warrant a change to gold-plated caps is debatable. For PBX 9404, in light of our present method of data interpretation, our belief is that the difference does not justify the use of the gold-plated caps. Other H.E.'s and mixtures may be more sensitive to this parameter however, and if "absolute" times-to-explosions are required, the change may be practical and necessary.

We plan to experiment with several more H.E. systems to help resolve this question.

The change in the slopes in the 220°C to 230°C region of the time-to-explosion of PBX 9404 (Figure 1) is most likely due to the explosive being a dual explosive system. The pure explosive, HMX, does not display this transition.

For Holston HMX, ballmilled and unmilled Bridgewater HMX, the time-to-explosion (Table III) were determined at three temperatures (240°C, 248°C, and 250°C). There was no significant difference in the times for the Holston and unmilled Bridgewater HMX; but the milled Bridgewater HMX times were approximately twice those of the Holston or the unmilled samples. The D.T.A. thermograms (Figures 31 and 33) and pyrolysis curves (Figures 28 and 30) for these samples show no great differences in the thermal sensitivity of Holston and Bridgewater HMX's. Plans are to increase the sensitivity of the pyrolysis apparatus in a further attempt to distinguish between these materials.

Because of the difference in the times-to-explosion of the milled and unmilled Bridgewater HMX, an experiment was conducted to run samples of varying particle sizes. The data from this experiment is given in Table IV. The experiment involving time-to-explosion measurements of Bridgewater and Holston HMX's as a function of particle size is incomplete. In general, however, the Bridgewater times decrease as size increases (Tables III, IV), while Holston HMX remains relatively constant (Tables II, V). This apparent variation in thermal sensitivity in the former may be due to parameters other than size alone. Such things as external surface area, particle roughness, distribution of inclusions or thermal conductivity effects (caused by different crystal packing) may be partially or dominantly responsible.

Two particular in-plant problems have caused some concern about the effect of liquids on thermal test results. The first involved a new type of oil (Ucon) to be used in a pumping station for a press. The D.T.A. thermogram of PBX 9404 + Ucon oil (Figure 7) suggests that the explosive is rendered less sensitive by the presence of the oil. The exotherm caused by nitrocellulose is less evident (in amplitude) even though it seems to begin about 10°C earlier. The decomposition of, or at least the resulting heat evolution from, the nitrocellulose occurs differently and does not contribute in the normal way to the HMX decomposition. On running LX-04-1 + Ucon in the D.T.A. (Figure 5), the run-away exotherm is about 25°C earlier than the LX-04-1 (Figure 4); but time-to-explosion data (Table VI) does not show this degree of enhanced reactivity. Comparing D.T.A. thermograms (Figures 8 and 9) of HMX and HMX + Ucon, it is noted that the HMX phase transition normally obtained at 190°C has changed to 182°C for the HMX + Ucon, which suggests that HMX is somewhat soluble in the Ucon. This is not seen in the above cases where the HMX is plastic bonded.

A possible similar effect was noted when LX-02-1, Extex, and PETN were compared. The D.T.A. thermograms (Figures 10, 11 and 12) would indicate that LX-02 and Extex were slightly more thermally sensitive than PETN; however, the Henkin data (Table VI) describes PETN as more thermally sensitive than either LX-02-1 or Extex. Since liquids are often used in our work, better methods or measuring its reactivity with explosives and interpreting the results will be incorporated into our near future work.

PETN and EL-511 Beta were selected to correlate the sample temperature with the pyrolysis apparatus thermal conductivity detector response (i.e., to correct for the thermal lag). PETN was used because it has a fast rising peak; EL-511 Beta was used because its pyrolytic peak occurs at a high temperature. Better temperature calibration is necessary for future work; however, since all the data described in this report was treated in the same way, the relative positions of the peaks may be compared. The pyrolytic decomposition curves (Figures 16 to 21) for the the six specially prepared HMX's (LRL raw material #A-311 through A-316) does not show a difference in these samples; because of this, it is believed that the sensitivity of the instrument should be improved. The $\frac{1}{4}$" tubing will be changed to 1/8" stainless steel tubing and its over-all length decreased. A thermostated hot wire detector will be substituted for the glow plugs.

Thermograms of PETN (Figures 34 to 39) indicate that increasing pressure on the sample decreases the temperature at which rapid decomposition starts. There is a difference of 3° to 4°C between the thermograms at atmospheric pressure and 85 psig, and 4° and 5°C between atmospheric pressure and 200 psig. They also suggest that the composition of the surrounding gas (at least in the case of air or nitrogen) has little or no effect on the thermograms. Future experiments will be conducted at a wider range of pressures to determine the functionality of the runaway temperature with pressure. Gas composition effects will be investigated further.

The gas chromatograph reactivity data (Table IX) for pelleted PBX 9404 and

PBX 9404 + lead are not particularly different from those obtained from loose samples. The various HMX's (Holston, Bridgewater, Bridgewater simulated) are not distinguishable by chromatograph analysis (Table XII).

The purpose of the reduction of the volume of the gas chromatograph sample tube (by inserting a 3/8" x 3 3/8" glass rod into the sample chamber) was to improve sample injection by increasing the product gas/helium ratio in the sample tube. This, when flushed out with more carrier gas, would be in a narrower band in the flow stream.

When the single column chromatograph is in operation (by the end of June), methods to improve resolution, to reduce time necessary to make a run, and to optimize sampling techniques will be tried in an effort to improve the existing chromatograph. Other experimental applications of the new chromatograph is to rapidly pyrolyze an explosive sample and analyze its products; also, to analyze gas samples of explosives taken at different temperatures of a slow pyrolysis run.

TABLE I

Time-To-Explosion for HMX & PBX 9404[1]

(Time - seconds)

	245°C.
HMX (dry)	88.6
	93.2
	91.7
	88.3
	100.6
HMX (8% humidity)	200.0[2]
	94.8[3]
	86.0
	82.0[3]
	96.4
	88.6
HMX (60% humidity)	90.3[3]
	200.0[2]
	90.9
	98.4
	89.5[3]
	111.3
	225°C.
PBX 9404 (dry)	97.4
	92.9
	83.8
	90.7
	119.0
	90.2
PBX 9404 (8% humidity)	89.1
	86.2[3]
	96.1
	92.8
	97.0[3]
PBX 9404 (60% humidity)	78.6
	92.5
	98.0
	96.9[3]
	92.0[3]

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[1]A 0.2 second interval counter was used to record these data.
[2]No event.
[3]Blew the lead plug out the top of the cap.

TABLE II

Time-To-Explosion for PBX 9404
In Brass and Gold Plate Caps[1]

(Time - seconds)

	195°C.	200°C.	210°C.	220°C.
Brass Caps	1133.6	559.2	289.7	127.0
	862.3	551.7	310.8[2]	132.5
	893.6	534.7	265.7	146.6
	938.5	570.4	235.2	138.8
	937.2	651.8	284.9	130.2
	944.6[3]	570.9[3]	274.8[3]	134.7[3]
Gold Caps	1069.3	840.8	307.2	152.2
	1904.9	975.1	261.4	141.0
	1140.4	847.2	319.0	161.5
	1079.8	686.2	448.8	126.8
	1167.5	1407.5	484.1	139.0
	1213.7[3]	898.8[3]	344.6[3]	143.1[3]

	225°C.	230°C.	240°C.	250°C.
Brass Caps	107.8	67.7	16.0	10.8
	87.2	30.0	24.6	14.6
	91.4	27.2	19.8	13.3
	93.0	24.8	18.2	12.8
	88.6	26.0	18.4	16.2
	93.1[3]	30.6[3]	19.0[3]	13.3[3]
Gold Caps	102.6	23.7	16.0	13.6
	38.5	29.6	17.6	14.4
	96.1	27.7	19.8	15.6
	97.1	27.6	19.4	15.0
	101.5	36.2	23.0[2]	16.4
	75.4[3]	28.4[3]	18.9[3]	14.9[3]

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[1] A 0.2 second interval counter was used to record these data.
[2] Blew the lead plug out the top of the cap.
[3] The harmonic mean of the times.

TABLE III

Time-To-Explosion for HMX[1]

(Time - seconds)

	240°C.	248°C.	255°C.
Holston HMX	121.9	79.6	49.1
	137.8	80.9	47.8
	127.0	82.0	48.2
	126.8	90.0	43.7
	121.8	80.8	50.5
	126.8[2]	82.5[2]	47.7[2]
Milled Bridgewater HMX	206.8	172.3	89.0
	227.2	143.8	114.9
	228.8	172.2	94.8
	211.9	134.9	85.4
	211.4	154.0	99.4
	216.9[2]	154.0[2]	95.7[2]
Unmilled Bridgewater HMX	104.3	72.0	53.1
	107.4	79.7	55.3
	118.0	77.3	54.0
	100.1	79.6	53.3
	109.8	87.0	57.4
	107.6[2]	78.8[2]	54.6[2]

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[1] A 0.2 second interval counter was used to record these data.
[2] The harmonic mean of the times.

[redacted]

TABLE IV

Time-To-Explosion for Bridgewater HMX of Varying Particle Sizes[1]

(Time - seconds)

		230°C.	260°C.
Milled HMX	10μ	756.2[2]	120.2
		609.4[2]	120.5
		639.8	122.6
		662.8[3]	121.1[3]
	44μ	723.4	124.8
		695.8	124.8
		672.2	146.0
		696.7[3]	131.2[3]
	62μ	613.6	111.1
		626.6	128.2
		638.1	151.2
		626.1[3]	128.1[3]
Unmilled HMX	62μ	510.0[2]	62.2
		586.7	87.0
		530.7	96.0
		540.7[3]	79.0[3]
	125μ	404.1	85.8
		850.0[2] (plug moved)	88.2
		513.2	88.2
		441.4	
		448.6[3]	87.4[3]
	350μ	357.3	62.8
		301.8[2]	69.0
		422.6[2]	75.2
		353.4[3]	68.6[3]

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[1] A 0.2 second interval counter was used to record these data.
[2] Blew the lead plug out the top of the cap.
[3] The harmonic mean of the times.

[redacted]

TABLE V

Time-To-Explosion for Holston-Made Grade II HMX[1]

(Time - seconds)

	220°C.	230°C.	240°C.	250°C.	260°C.	270°C.
Class E HMX	468.6	238.8	121.2	75.2	42.4	24.8
	409.4	203.2	125.0	70.8	38.1	23.4
	460.5	222.2	116.0	68.8	44.8	24.2
	440.4	223.7	190.2	74.2	46.0	21.6
	600.0	251.4	129.4	72.0	41.4	24.6
	660.0					
	402.2					
	434.7[2]	226.7[2]	132.1[2]	72.1[2]	42.4[2]	28.7[2]
Class A HMX		216.2			47.7	
		218.8			47.4	
		202.0			47.8	
		228.4			65.6	
		216.7			59.4	
		216.7[2]			52.6[2]	
Class B HMX		285.5			48.0	
		288.8			54.0	
		294.0			55.2	
		282.9			54.3	
		326.0			63.6	
		294.7[2]			54.6[2]	
Class D HMX		199.8			47.8	
		224.3			49.2	
		207.3			46.7	
		178.8			46.2	
		202.2			52.4	
		201.4[2]			48.4[2]	

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[1] A 0.2 second interval counter was used to record these data.
[2] The harmonic mean of the times.

TABLE VI

Time-To-Explosion for LX-04-1 & LX-04-1 + Ucon[1]

(Time - seconds)

	240°C.	251°C.	262°C.	270°C.
LX-04-1	268.8	145.6	74.2	35.6
	248.8	193.4	70.4	27.8
	316.4	170.8	76.0	
LX-04-1 + Ucon	482.6	145.6[2]	55.6	32.2
	512.8[2]	146.2[2]	55.8	34.6
	417.4[2]	151.6	55.0	40.2[2]

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[1] A 0.2 second interval counter was used to record these data.
[2] Blew the lead plug out the top of the cap.

TABLE VII

Time-To-Explosion for PETN, LX-04-1 & Extex[1]

(Time - seconds)

	135°C.	140°C.	145°C.	155°C.	165°C.	172°C.	175°C.	180°C.
PETN	716.9	477.5	171.4	79.8	40.4	31.6	25.8	20.4
	716.2	327.8	174.5	83.0	38.0	29.6	25.0	22.6
	179.8		182.6	69.5	38.5	29.2	25.2	22.4
	656.4		183.8	98.6	39.0	33.4	24.9	20.8
	721.1		178.4	90.7	41.1		24.5	
	688.6		164.0					
	620.1		166.7					
			177.0					
			177.6					
	696.9[2]	388.8[2]	174.9[2]	83.1[2]	39.4[2]	30.9[2]	25.1[2]	21.5[2]
LX-02-1						343.6		41.2
						254.2		42.8
						339.9		45.8
						137.6		40.1
Extex						186.8		64.4
						178.6		68.0
						187.0		72.6
						185.8		72.2

----

[1] A 0.2 second interval counter was used to record these data.
[2] The harmonic mean of the times.

TABLE VIII

Time-To-Explosion for HMX[1]

(Time - seconds)

HMX (LRL)	230°C.	245°C.	260°C.
A-311	700.0[3]	106.4[2]	50.1
H S_c C	224.1	96.6	45.5
	242.4	102.8[2]	46.6
	195.6	129.9[2]	49.4
	198.8	107.8	48.8
	188.7		
A-312	247.4	134.3	51.1
H S_c C	253.0	131.8	41.1
	308.8	124.8	37.9
	238.8	126.2	50.6
	226.5	120.8	59.0
A-313	246.3[2]	134.0	25.5
B S_a C	297.0	161.6	23.7
	236.0[2]	122.8[2]	19.4
	238.2	131.8[2]	36.4
	248.0	106.3[2]	30.6
A-314	238.4[2]	97.9	24.8
B S_a F	247.1[2]	102.8	26.8
	253.0[2]	91.8	29.4
	242.2	106.8	31.7
	238.2	110.0	46.0
A-315	200.7[2]	107.0	27.9
B S_c C	245.2[2]	110.1[2]	28.0
	229.8	110.8[2]	33.0
	273.8	111.1	38.7
	233.6	105.5	43.5
A-316	254.0	139.7	28.6
B S_c F	270.6	95.6	27.5
	256.2	121.6	29.7
	216.2	114.0	31.5
	208.2	113.2[2]	35.6

----

[1] A 0.2 second interval counter was used to record these data.
[2] Blew the lead plug out the top of the cap.
[3] No event.

TABLE IX

Gas Chromatograph Results
(Gas Evolved in Microliters @ S.T.P.)

Date	$N_2 + O_2$	NO + CO	N_2O	CO_2	Total
			LX-04-1 (B2-673-62) @ 150°C.		
4/2/64	10.2	9.46	54.8	15.2	89.7
	10.4	9.46	61.2	18.0	99.1
	15.7	7.03	42.2	18.2	83.1
			CB-3 @ 150°C.		
3/31/64	197	277	366	216	1060
	571	907	998	755	3230
	193	245	325	191	954
4/2/64	599	1730	1083	953	4365
			LX-02-1, Lot 188 @ 100°C.		
3/20/64	18.9	20.0	0.97	5.43	45.3
3/24/64	23.6	19.3	0.62	2.24	45.8
	13.1	13.0	0.31	3.95	30.4
	16.8	25.2	0.82	5.00	47.8

TABLE X

Date	$N_2 + O_2$	NO + CO	N_2O	CO_2	Total
			PBX 9404 Pellets @ 120°C.		
1/31/64	40.4	165.4	23.7	117.2	346.7
	31.2	160	23.0	103.0	317.6
	33.8	177	29.1	133	372.9
	45.8	190	27.4	155	418
			PBX 9404 + Lead Pellets @ 120°C.		
1/30/64	60.1	270	113	127	571
1/31/64	71.4	293	133	128	626
2/11/64	50.8	218	76.4	111	456
	61.1	256	79.9	112	509
1/16/64	69.0	280	89.7	128	567
12/30/63	77.0	243	101	109	530
12/20/63	56.4	246	84.4	131	517

Table XI

DATE	$N_2 + O_2$	NO + CO	N_2O	CO_2	TOTAL
	Comp B, Grade A (517-6401-001-001) @ 120°C				
4-8-64	10.8	16.9	8.47	7.74	43.9
	8.20	18.4	9.43	7.06	43.1
4-7-64	16.5	20.3	10.8	11.0	58.6
	18.4	18.7	11.4	10.7	59.2
4-3-64	10.5	18.6	10.9	7.58	47.7
	18.3	15.3	8.40	10.3	52.3
	CB_3 @ 120°C				
2-13-64	6.10	11.6	19.1	14.3	51.1
	11.7	8.84	10.3	9.60	39.9
3-20-64	11.2	10.3	12.0	10.8	44.3
	15.4	1.14	6.85	14.1	37.5
3-26-64	8.74	4.30	8.53	11.3	32.9
	8.10	11.0	11.9	8.37	39.3
1-31-64	10.3	5.43	4.83	5.14	25.7
4-7-64	9.78	14.2	4.60	5.88	34.5
	9.26	12.1	10.3	11.5	43.1
	Comp C-4 (495-15-9-56) @ 120°C				
4-1-64	9.92	3.78	2.03	6.84	22.6
	17.2	5.40	1.20	5.31	29.1
4-2-64	13.3	4.18	1.33	5.95	24.7
	17.0	4.32	1.47	4.17	27.0
4-3-64	17.1	3.24	0.99	4.35	25.7
	12.2	3.92	1.52	7.93	25.6
	HMX, Grade VI, Class B, Lot SRA-62, @ 120°C				
3-19-64	7.80	2.80	10.6	7.80	29.0
	11.9	5.20	8.30	6.40	31.8
3-18-64	4.90	6.00	6.00	5.60	22.5
	11.6	2.80	9.20	3.80	27.4
3-17-64	11.1	2.60	9.00	5.70	28.4
	28.6	4.70	8.50	3.80	45.7

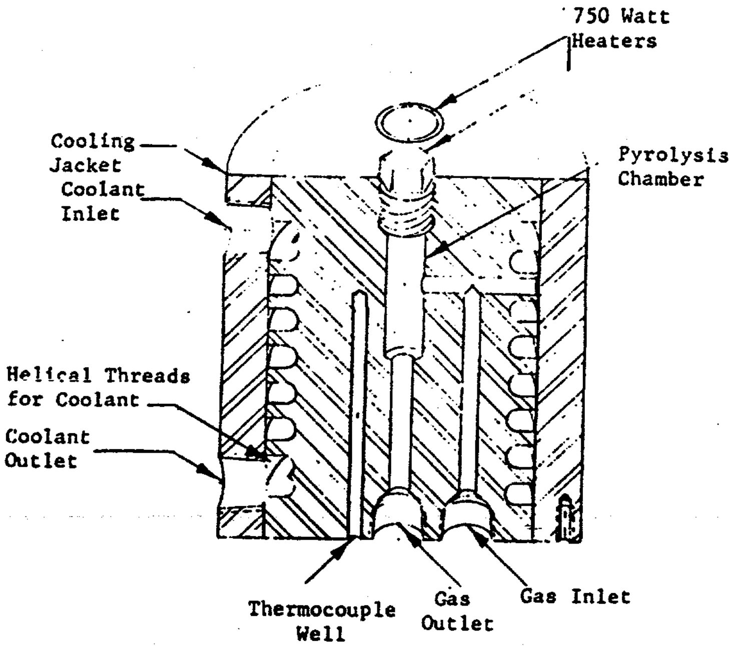
Table XI (Continued)

DATE	$N_2 + O_2$	NO + CO	N_2O	CO_2	TOTAL
		PBX 9011, Batch #656-4 HDSR137-63 @ 120°C			
3-24-64	12.8	0.00	2.74	6.75	22.3
	8.55	0.00	4.08	7.80	20.4
3-25-64	9.64	1.22	3.28	9.78	23.9
2-11-64	6.98	0.79	5.70	9.78	23.3
3-31-64	10.8	0.00	3.80	10.9	25.5
3-25-64	16.2	2.20	2.67	4.72	25.8
		PBX 9205 (57155A0901) @ 120°C			
3-17-64	14.5	2.24	6.79	3.89	27.5
	12.5	4.03	6.06	4.84	27.5
3-18-64	12.2	2.01	5.33	4.28	23.8
	8.5	2.90	4.10	4.10	19.6
3-19-64	9.20	1.00	6.80	4.90	21.9
	9.84	4.68	5.30	8.05	27.9
		Tetryl Pellets @ 120°C			
3-19-64	16.2	17.5	1.00	9.10	43.8
	10.6	9.90	1.00	9.80	31.3
3-18-64	13.2	11.4	1.00	8.40	34.0
	11.4	15.7	0.40	7.40	34.9
3-17-64	13.8	13.0	0.80	6.10	33.7
	12.1	16.6	0.90	10.6	40.2
		TNT @ 120°			
1-31-64	6.90	6.30	0.00	7.80	21.0
2-12-64	26.2	5.00	0.70	7.10	39.0
3-20-64	14.8	3.96	0.74	11.5	31.0
	4.74	5.76	0.96	6.30	17.8
2-13-64	11.5	6.95	0.00	5.31	24.2
	17.7	8.86	0.00	8.53	35.1
3-26-64	12.8	3.28	0.00	4.97	20.9
3-31-64	14.4	5.67	0.00	7.83	24.9
	4.52	5.33	0.00	7.95	17.8
		LX-02-1, Lot 188 @ 120°C			
3-20-64	53.1	94.6	18.0	33.9	200
3-17-64	47.2	118	19.2	38.2	233
	66.6	112	25.0	44.5	248

Table XII

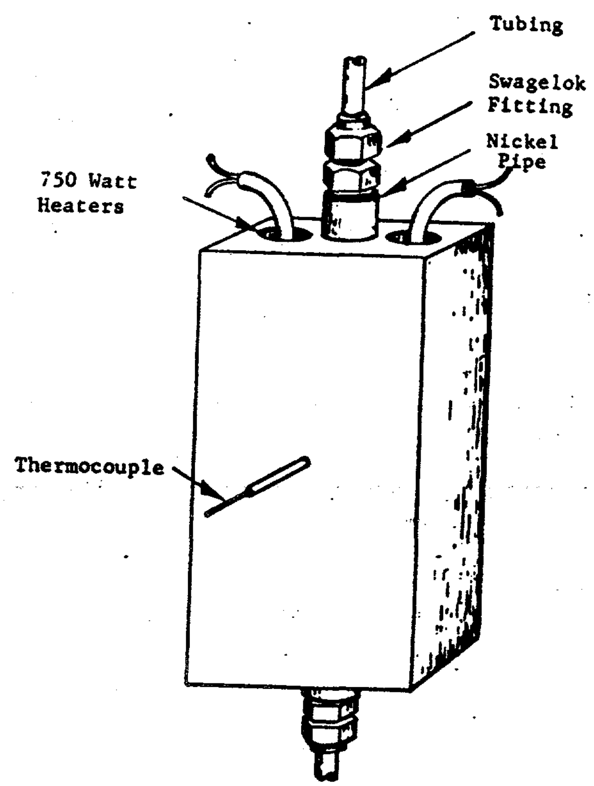
DATE	$N_2 + O_2$	NO + CO	N_2O	CO_2	TOTAL
		Holston HMX, A-311, SR187-63 @ 120°C			
4-15-64	19.8	2.08	7.55	2.76	32.2
	14.2	1.56	5.92	4.65	26.3
	7.70	1.84	8.45	6.59	24.6
		Holston HMX, A-312, SR188-63 @ 120°C			
4-15-64	9.54	4.55	9.44	4.05	27.6
	12.7	2.79	10.4	7.54	33.5
	22.6	3.27	14.6	6.45	46.9
		Holston HMX, A-313, SR189-63 @ 120°C			
4-15-64	9.63	0.62	4.27	3.88	18.4
	32.2	2.75	5.22	3.12	43.3
	15.3	1.88	4.58	6.72	28.5
		Holston HMX, A-314, SR190-63 @ 120°C			
4-16-64	8.69	3.50	8.73	6.48	27.4
	9.92	1.69	11.2	7.30	30.1
	11.8	2.10	10.1	6.59	30.6
		Holston HMX, A-315, SR191-63, @ 120°C			
4-9-64	20.0	2.47	4.78	1.82	29.0
	10.7	2.27	5.28	5.71	24.0
	10.8	1.82	5.85	7.83	26.3
		Holston HMX, A-316, SR192-63 @ 120°C			
4-16-64	12.0	2.60	7.86	4.00	29.4
	23.5	3.98	15.3	6.93	29.6
	14.3	3.76	12.1	5.52	35.7
		Bridgewater HMX, PC #A-004-SA2623 Unmilled @ 120°C			
4-10-64	11.9	0.00	1.76	3.89	17.5
	35.9	0.00	1.27	7.46	44.6
4-16-64	15.1	1.17	2.71	3.92	22.9
		Bridgewater HMX, PC #A-004, SA2623, Ball Milled @ 120°C			
4-10-64	8.90	0.00	5.96	7.65	22.5
	19.4	0.00	9.43	9.94	38.8
4-16-64	7.79	0.00	9.67	7.24	24.7
		Holston HMX Cl.A, Lot 123-63 (6) @ 120°C			
4-16-64	11.6	1.36	11.6	7.93	32.4
	8.53	2.34	12.8	6.98	30.7
	16.3	2.53	16.1	6.81	41.7

DIAGRAM I



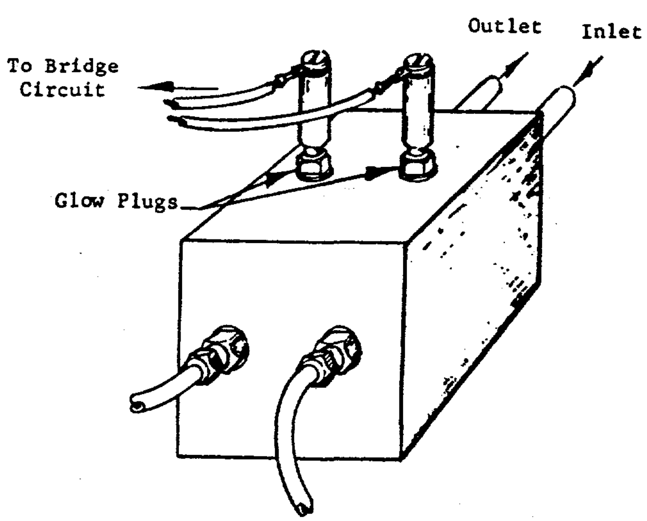
PYROLYSIS BLOCK

DIAGRAM II



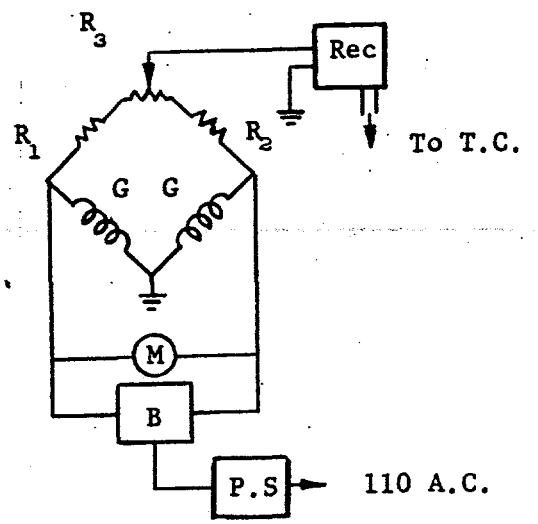
COMBUSTION CHAMBER

DIAGRAM III



THERMAL CONDUCTIVITY DETECTOR

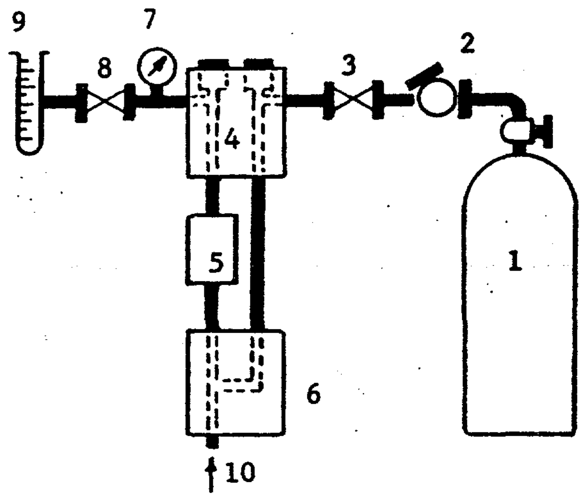
DIAGRAM IV



, R_2	Resistors 50Ω
	Helipot, 10turn 25Ω
G	Glow Plugs VG-1
	Voltmeter (0 - 3 volts)
	6 - 12 volt Battery Charger
S.	Powerstat
:c.	X-Y Recorder

RMAL CONDUCTIVITY DETECTOR CIRCUIT

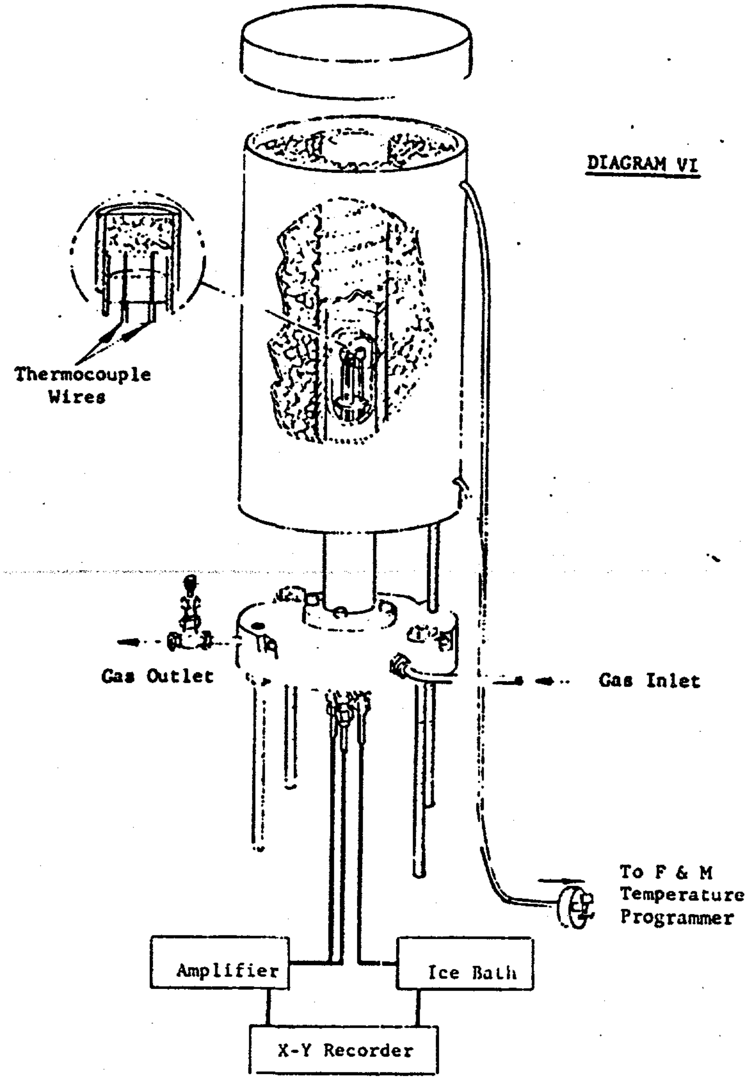
DIAGRAM V



1. Carrier gas (helium)
2. Regulator
3. & 8. Needle Valve
4. Thermal Conductivity Detector
5. Combustion Chamber
6. Pyrolysis Block
7. Pressure Gage
9. Flowmeter
10. Sample Inlet

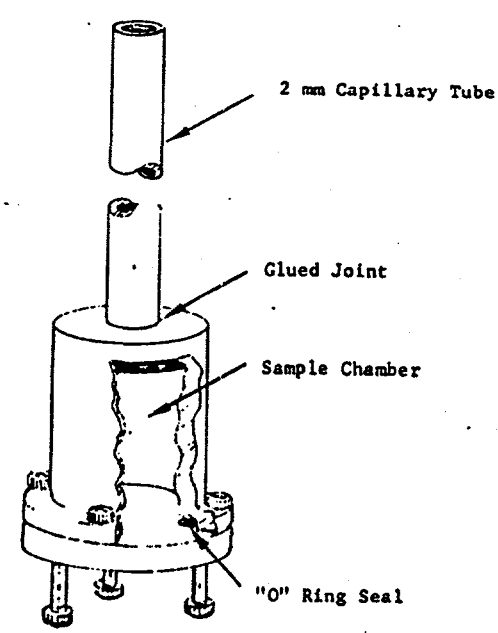
BLOCK DIAGRAM OF PYROLYSIS APPARATUS

DIAGRAM VI



CONTROLLED ATMOSPHERE DTA

DIAGRAM VII



GLASS TRANSITION APPARATUS

[redacted]

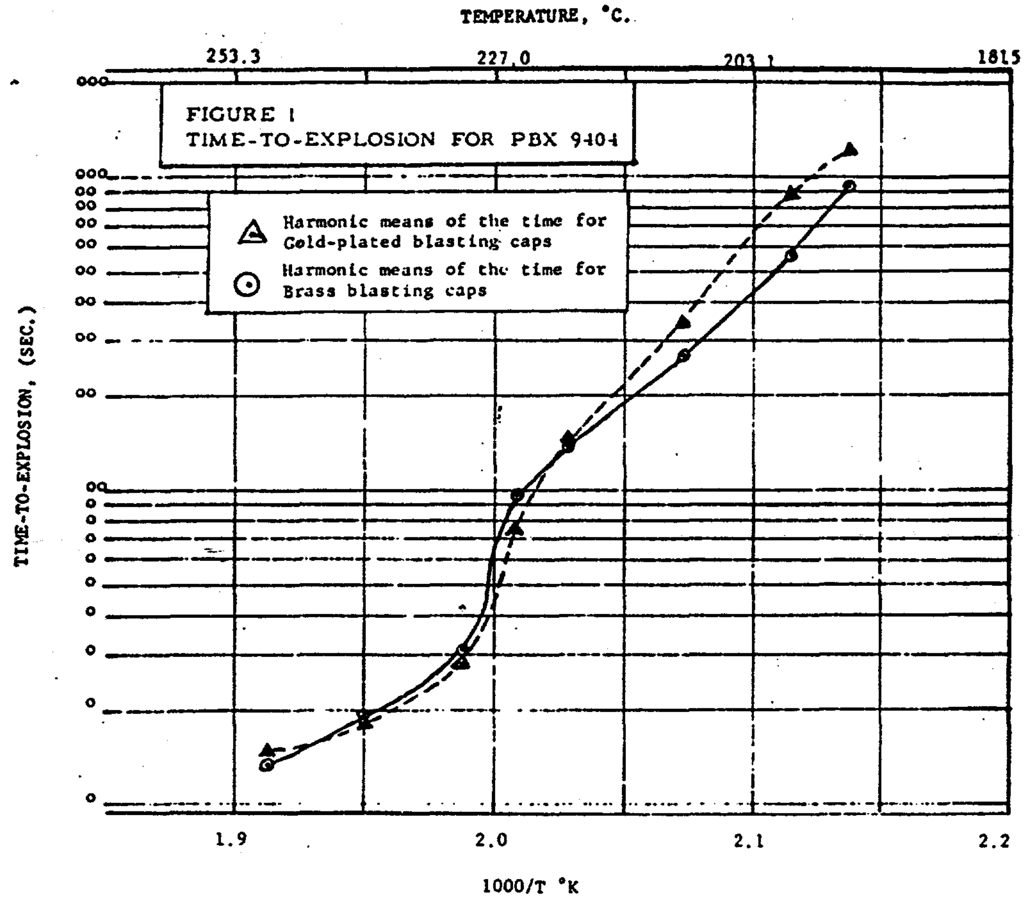


FIGURE 1
TIME-TO-EXPLOSION FOR PBX 9404

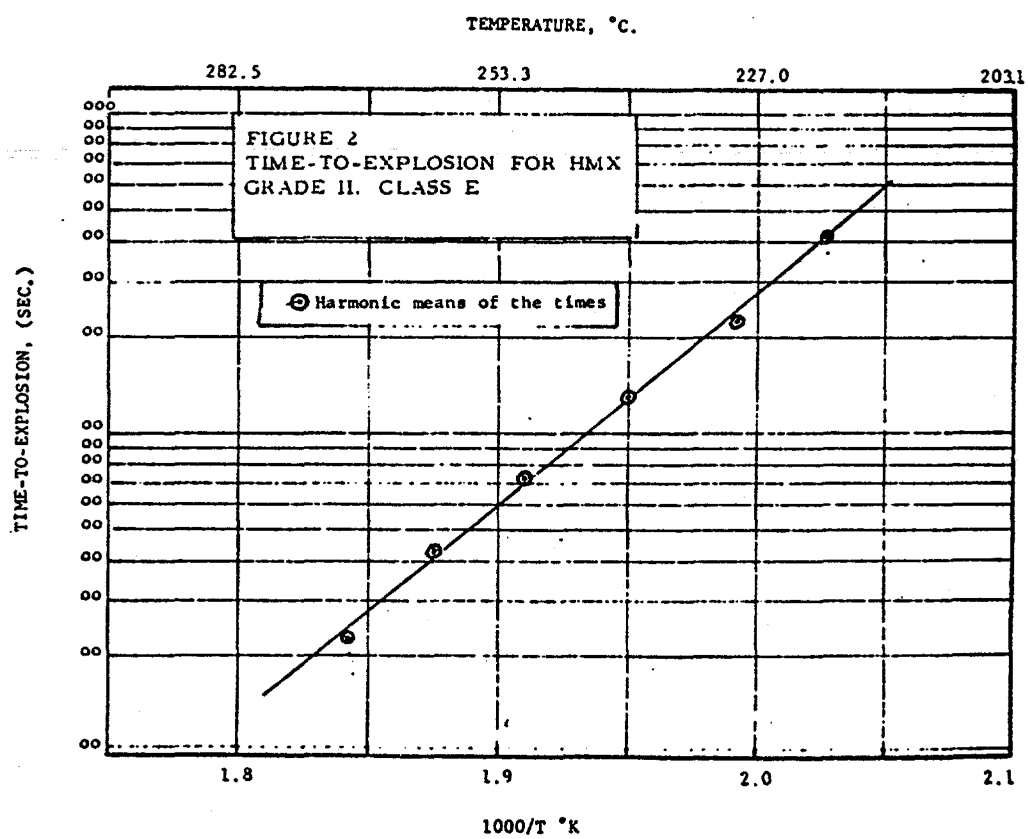
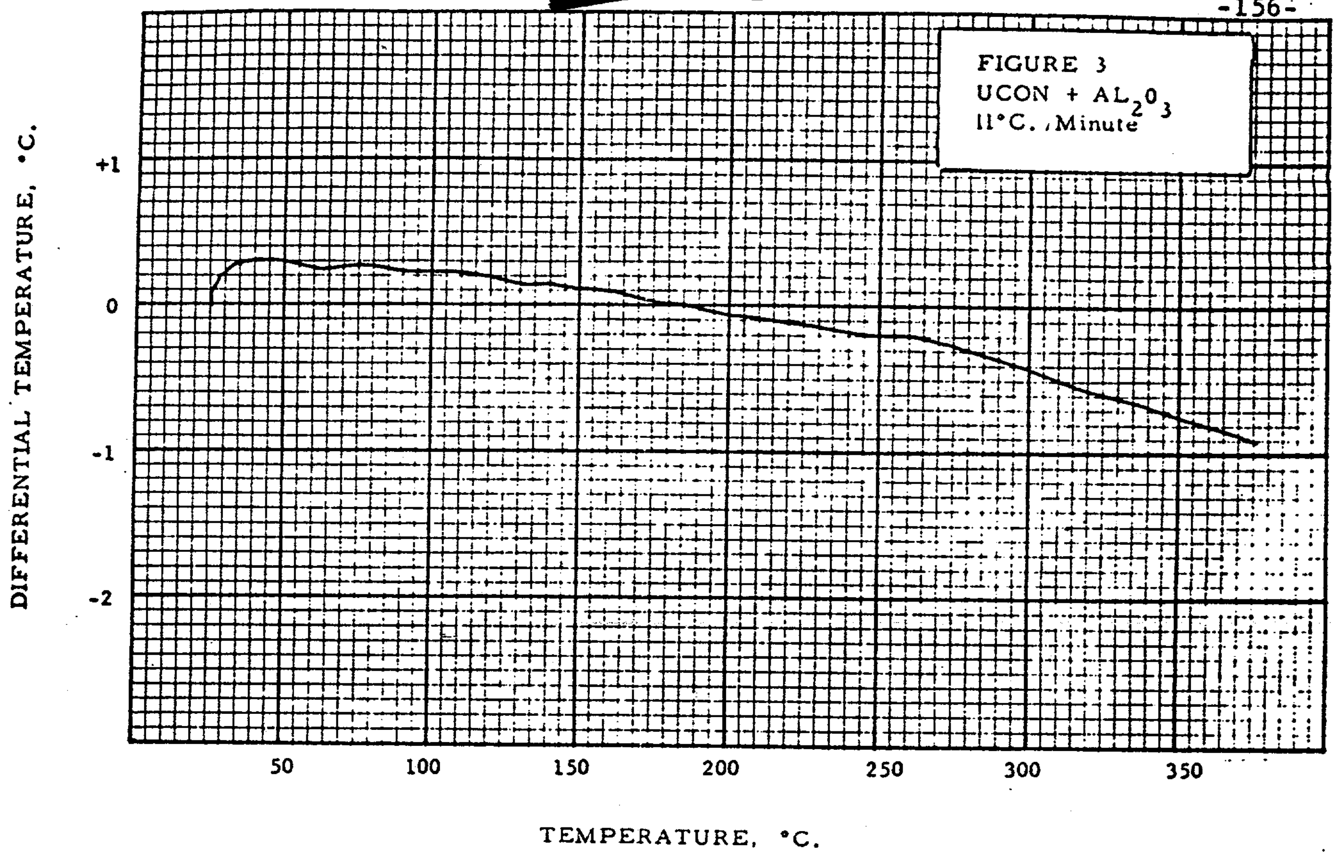
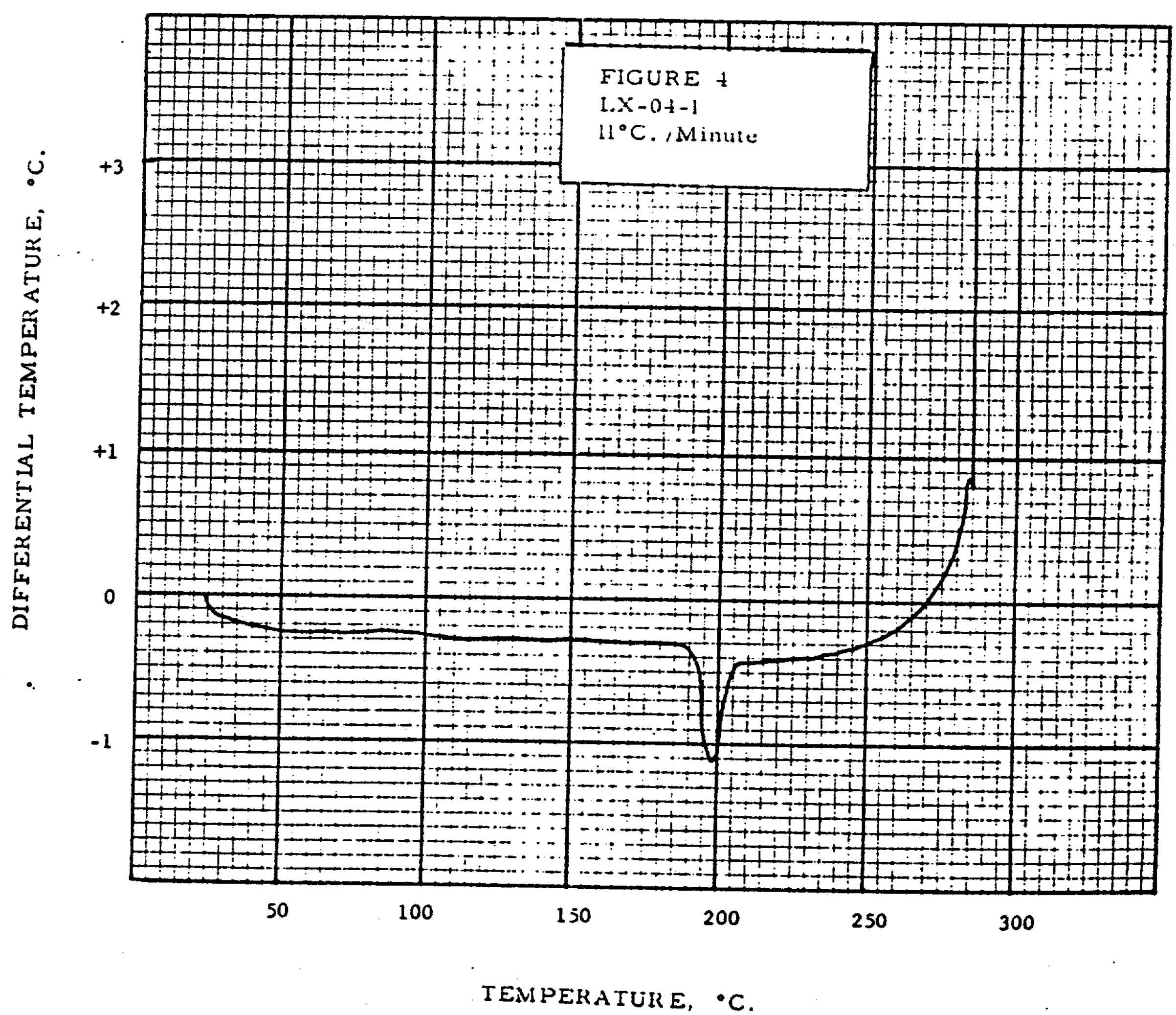


FIGURE 2
TIME-TO-EXPLOSION FOR HMX GRADE II. CLASS E

[redacted]





[redacted]

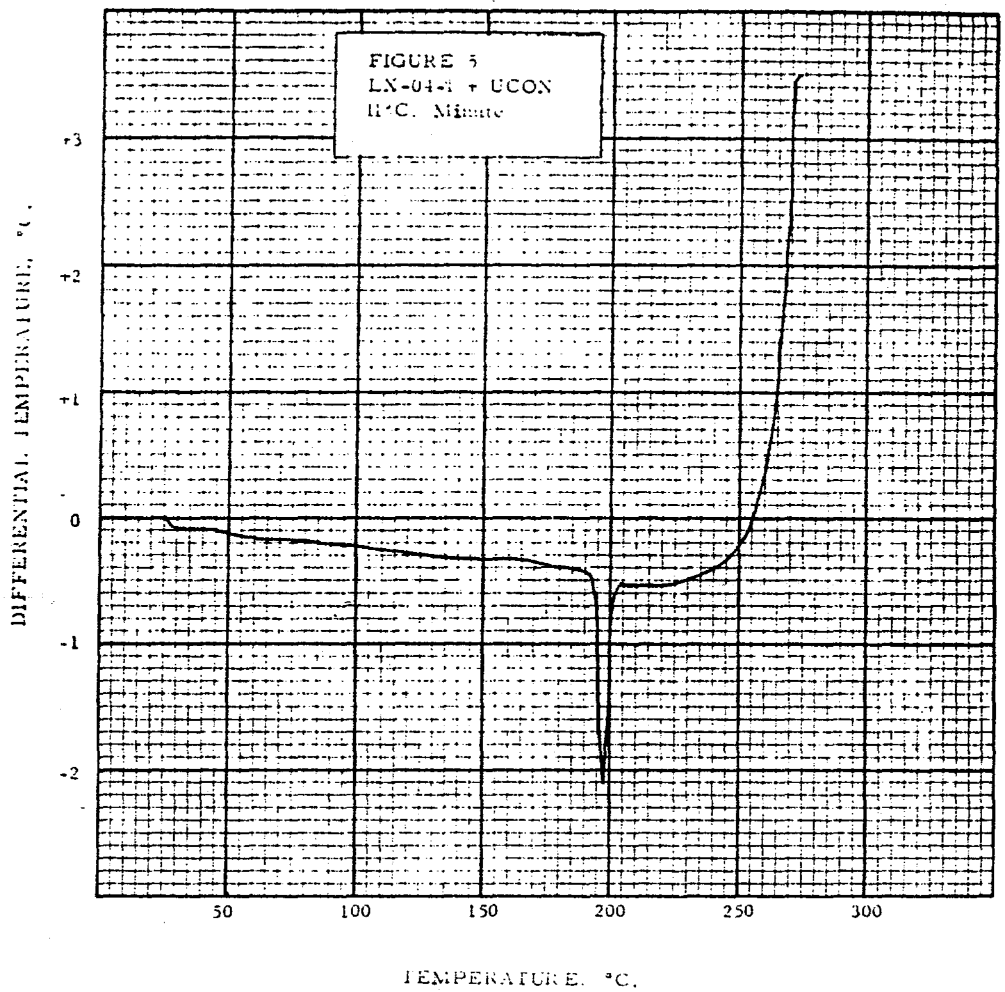


FIGURE 5
LX-04-1 + UCON
11°C/Minute
DIFFERENTIAL TEMPERATURE, °C.
+3
+2
+1
0
-1
-2
50
100
150
200
250
300
TEMPERATURE, °C.

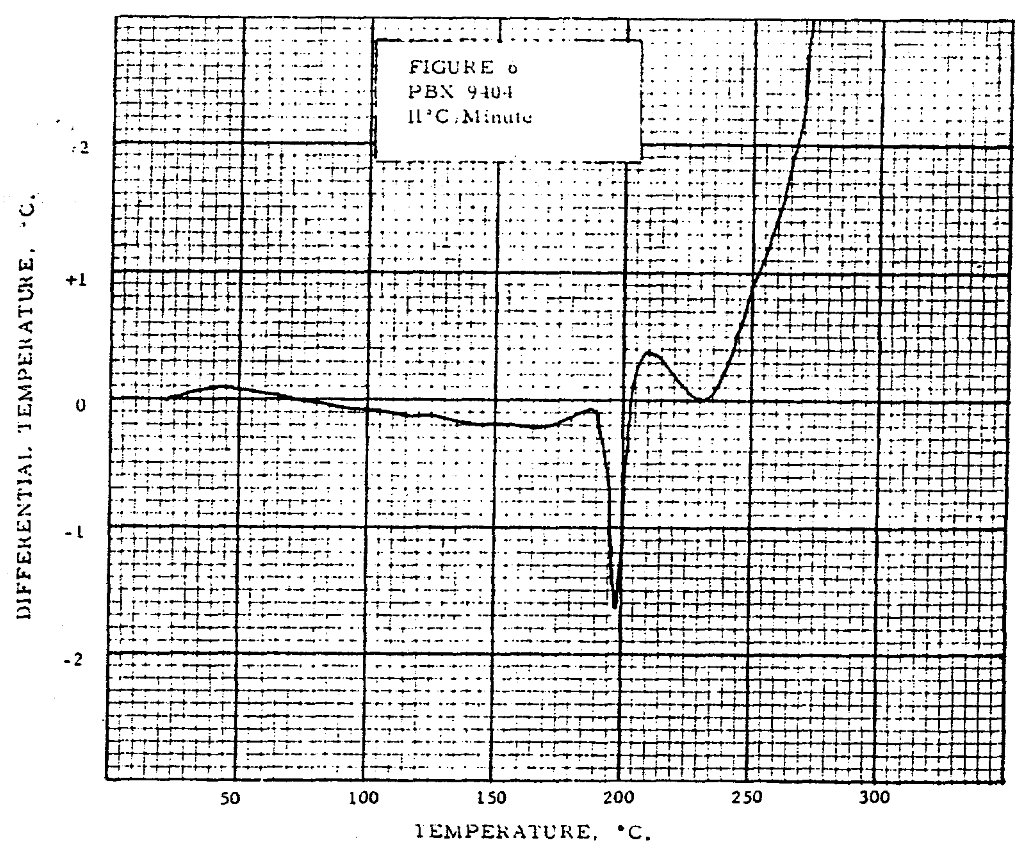


FIGURE 6
PBX 9404
11°C/Minute
DIFFERENTIAL TEMPERATURE, °C.
+2
+1
0
-1
-2
50
100
150
200
250
300
TEMPERATURE, °C.

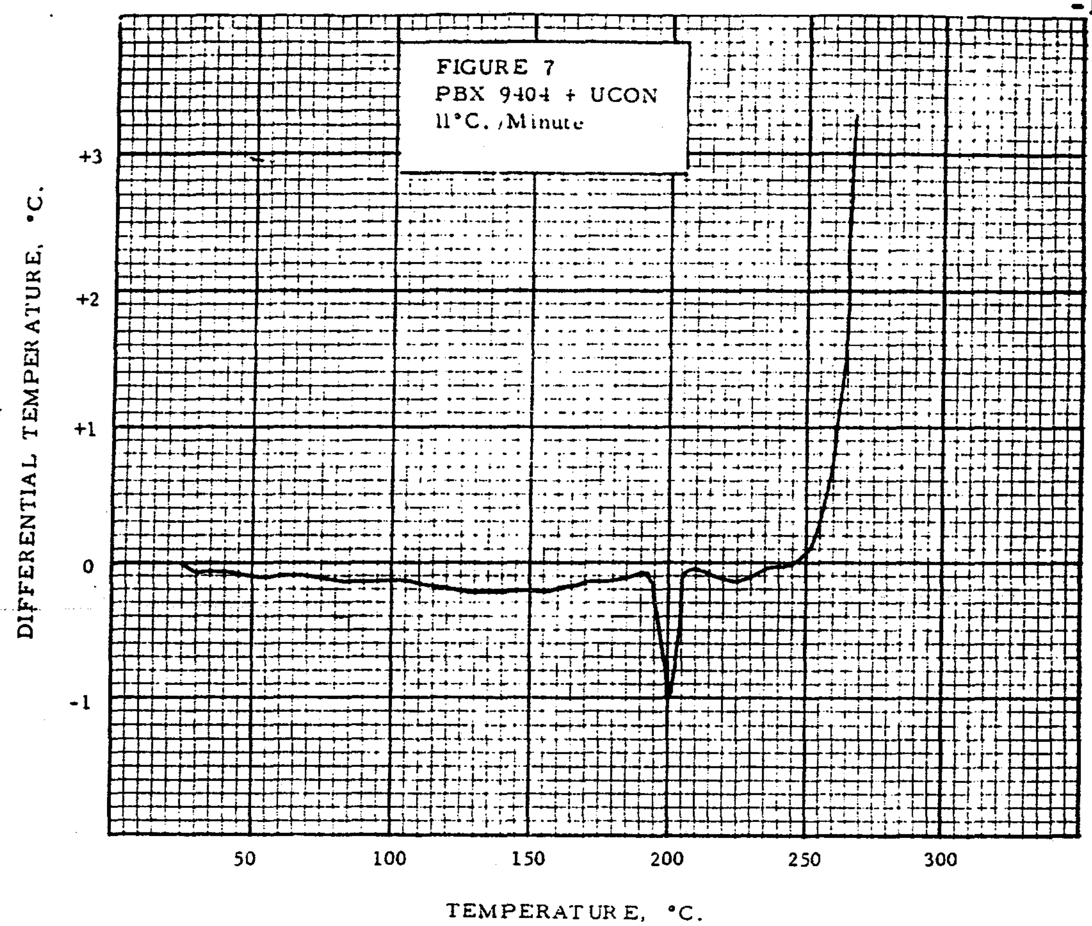


FIGURE 7
PBX 9404 + UCON
11°C. /Minute
DIFFERENTIAL TEMPERATURE, °C.
+3
+2
+1
0
-1
50
100
150
200
250
300
TEMPERATURE, °C.

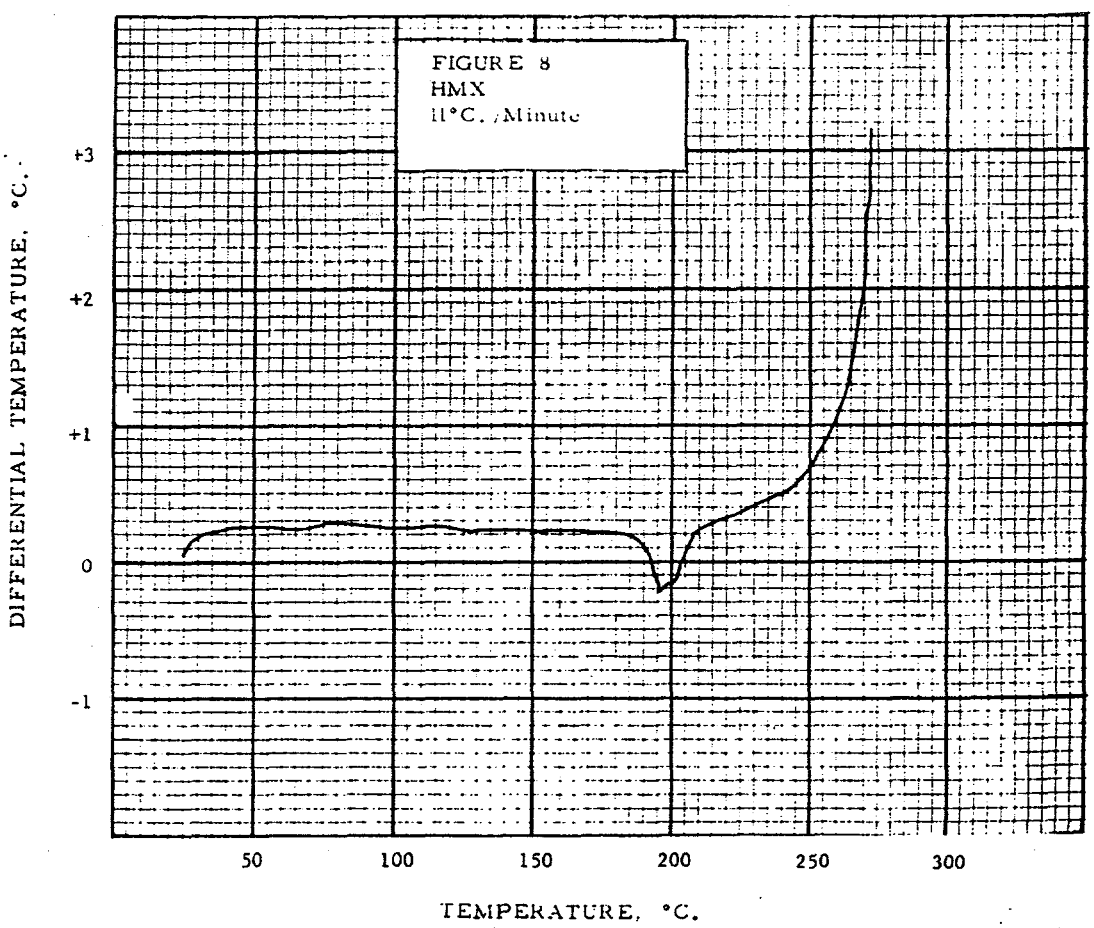


FIGURE 8
HMX
11°C. /Minute
DIFFERENTIAL TEMPERATURE, °C.
+3
+2
+1
0
-1
50
100
150
200
250
300
TEMPERATURE, °C.

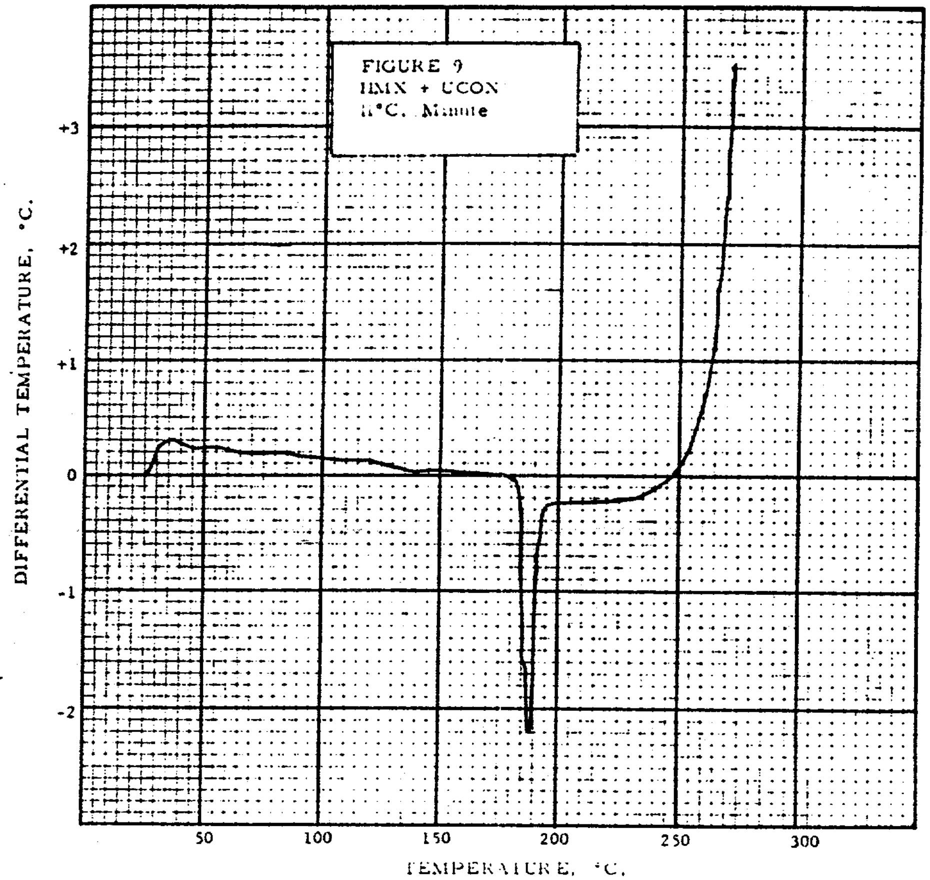


FIGURE 9
HMX + UCON
11°C./Minute

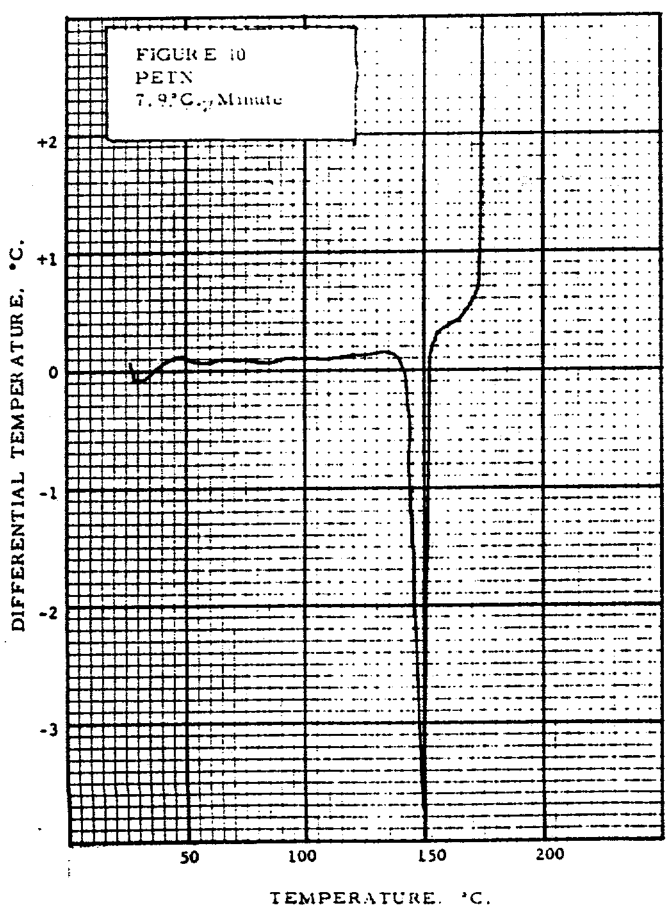


FIGURE 10
PETN
7.9°C./Minute

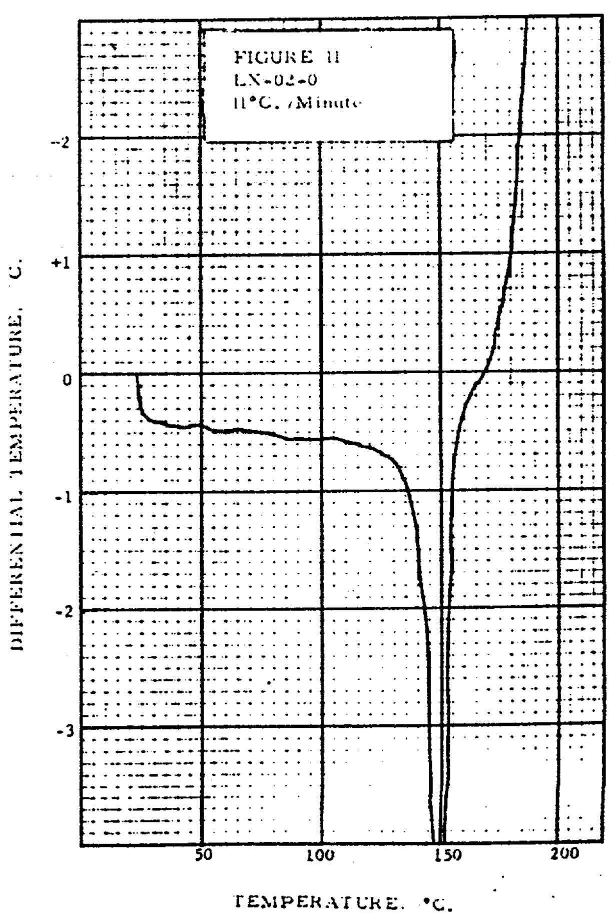


FIGURE 11
LX-02-0
11°C./Minute

[redacted]

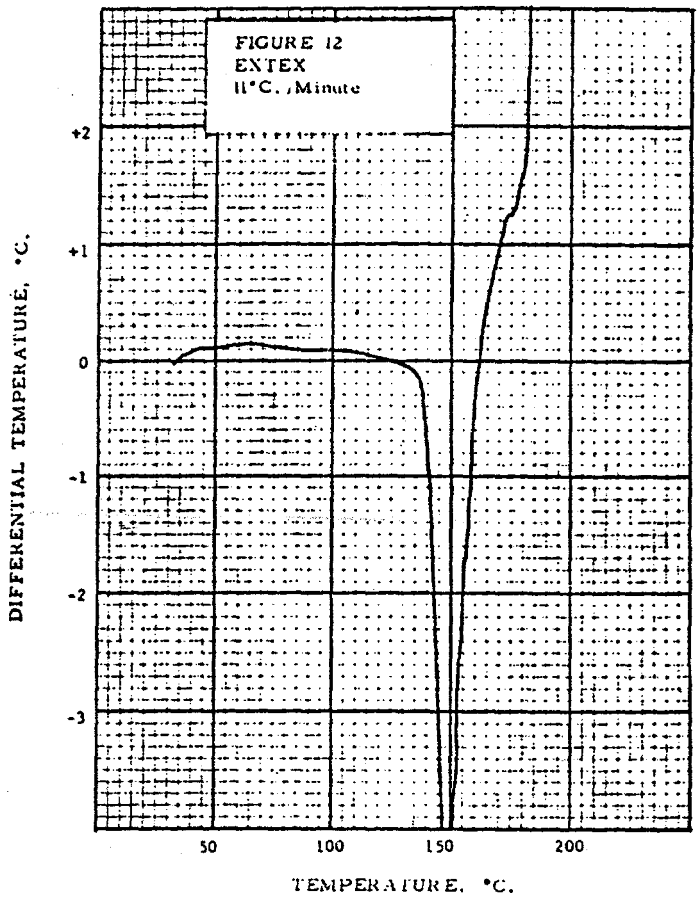


FIGURE 12
EXTEX
11°C./Minute

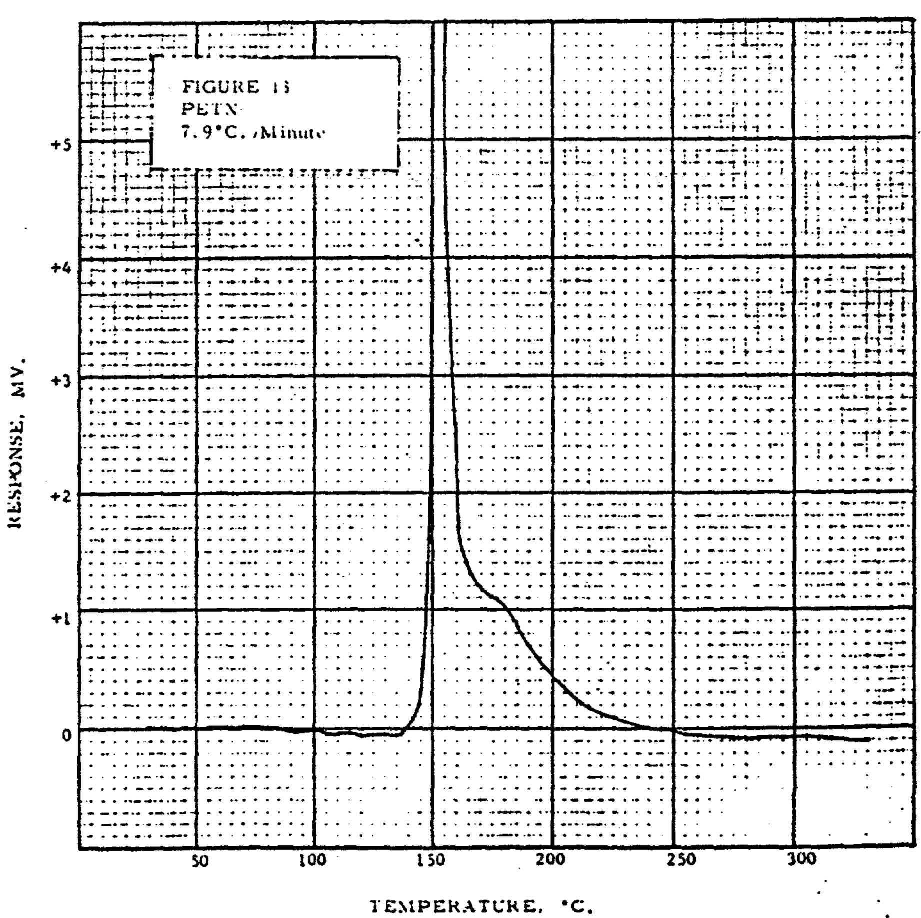


FIGURE 13
PETN
7.9°C./Minute

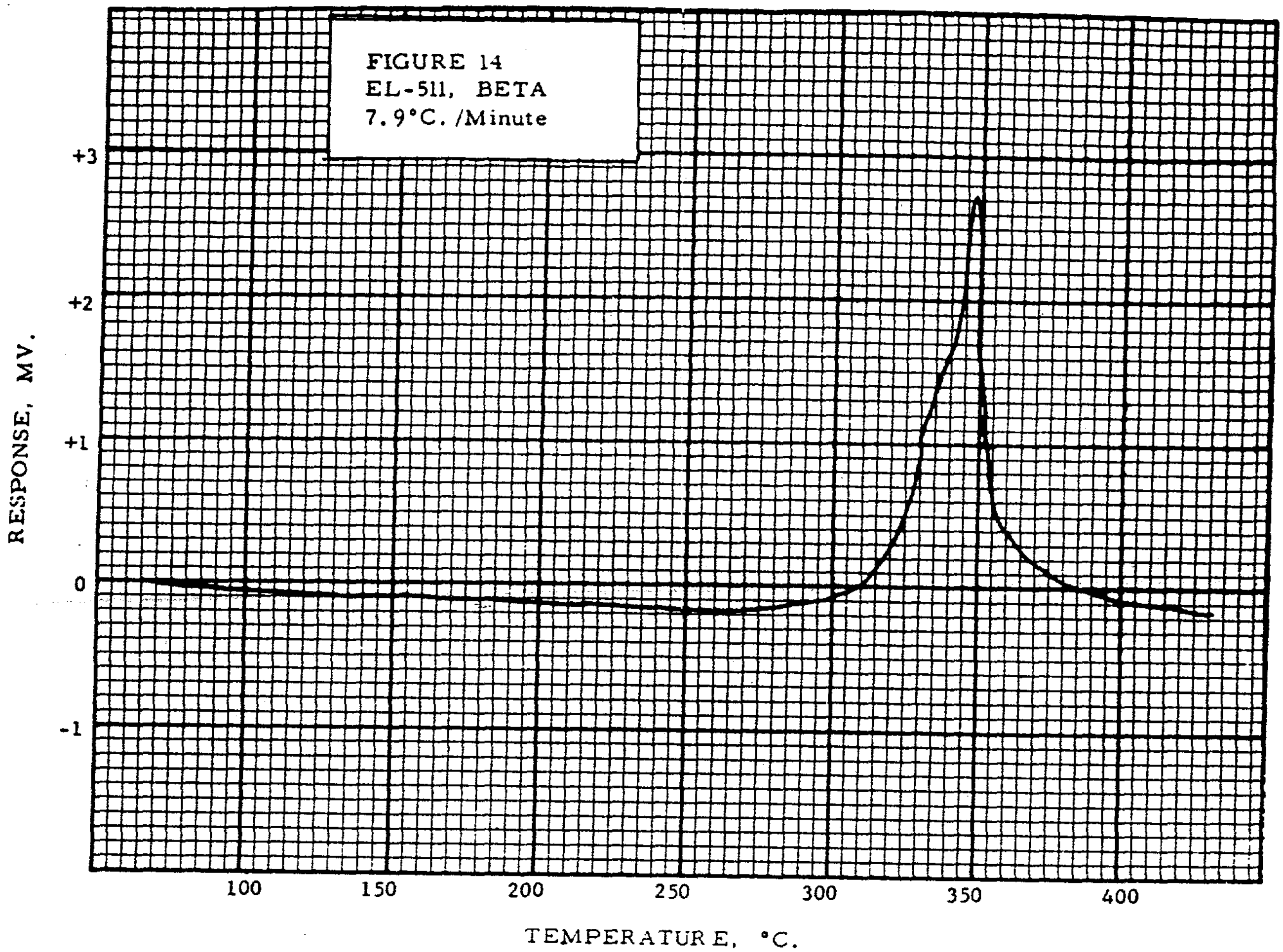


FIGURE 14
EL-511, BETA
7.9°C. /Minute
RESPONSE, MV.
+3
+2
+1
0
-1
100
150
200
250
300
350
400
TEMPERATURE, °C.

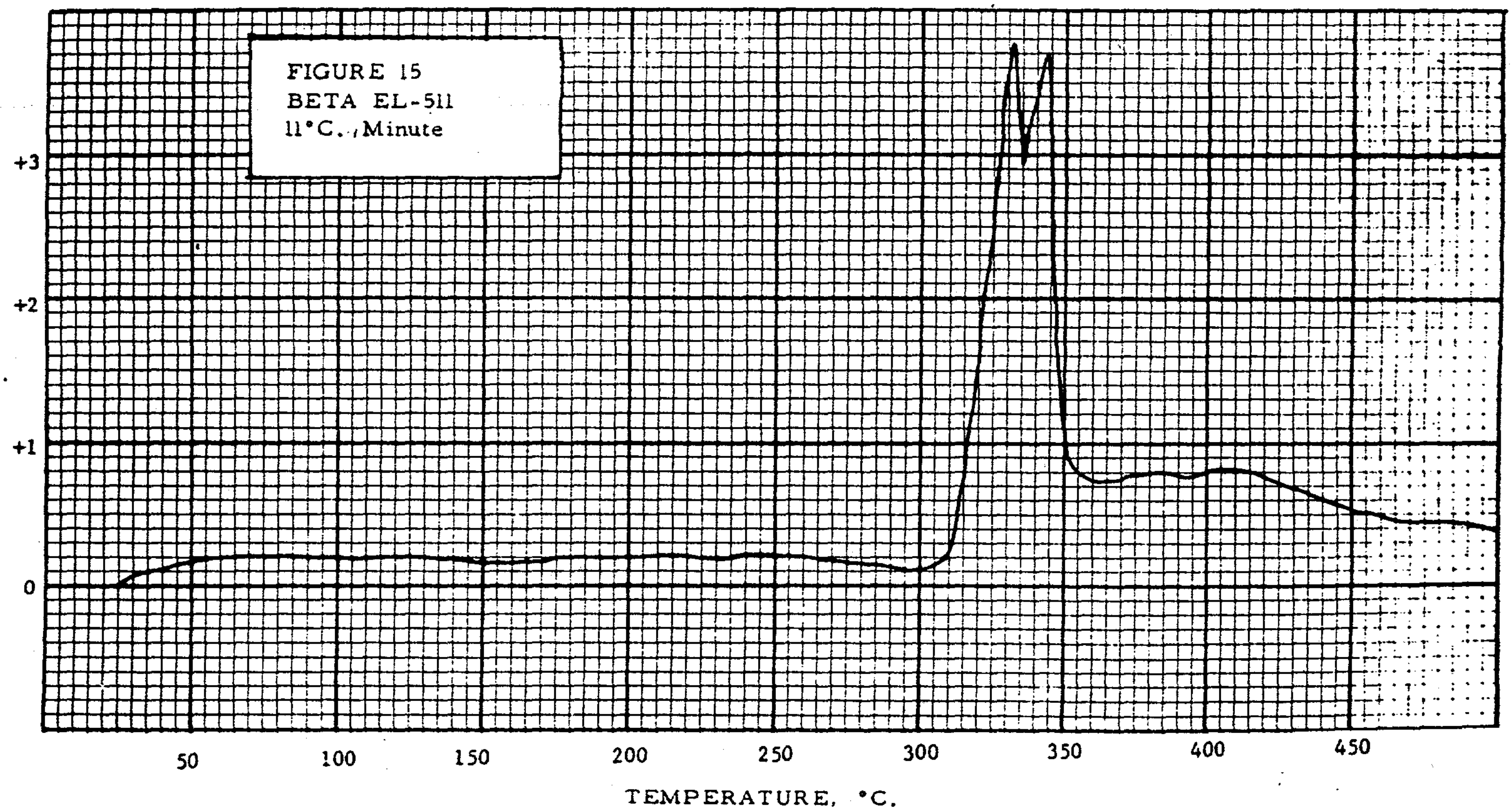


FIGURE 15
BETA EL-511
11°C. /Minute
+3
+2
+1
0
50
100
150
200
250
300
350
400
450
TEMPERATURE, °C.

[redacted]

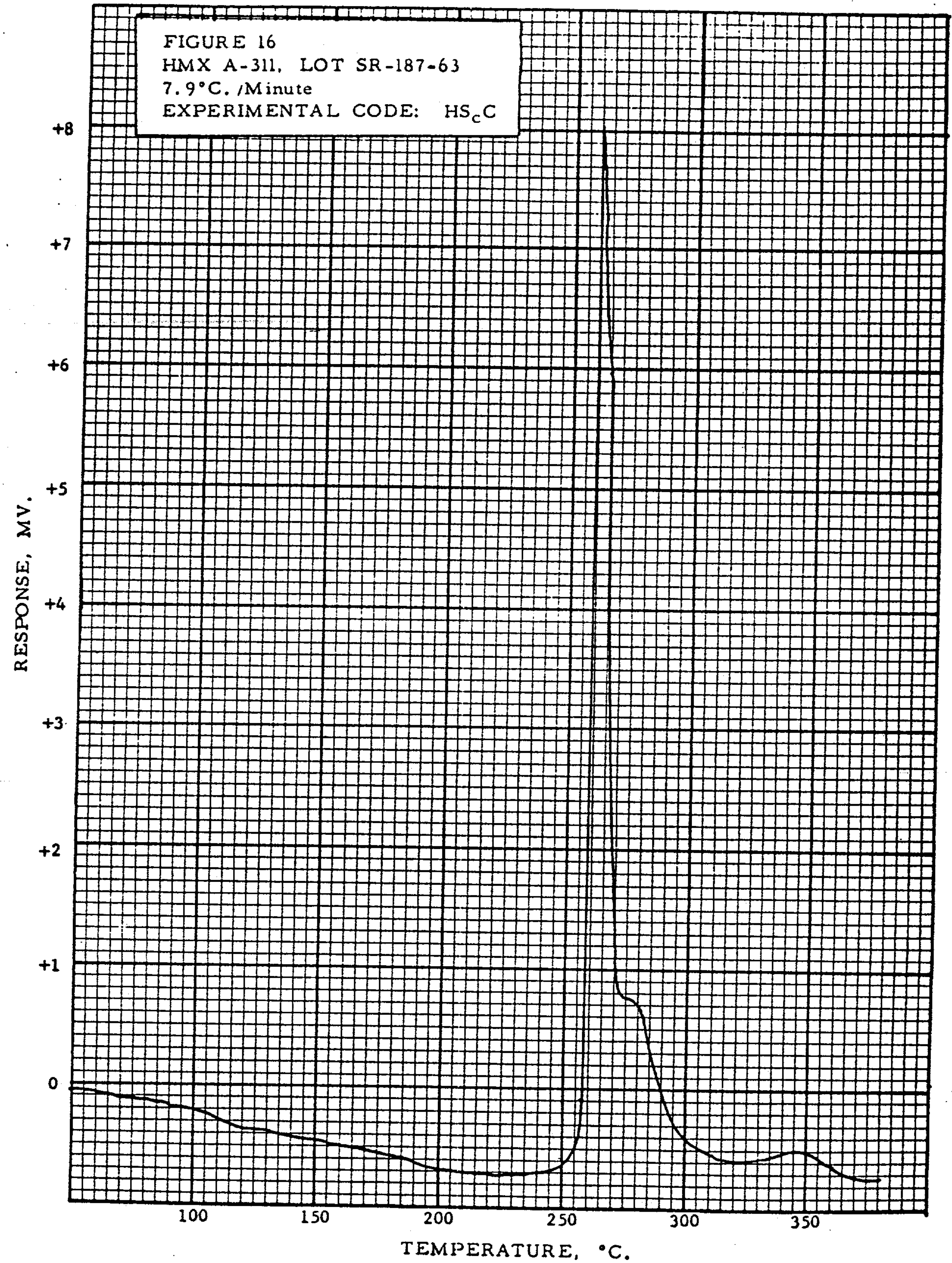


FIGURE 16
HMX A-311, LOT SR-187-63
7.9°C. /Minute
EXPERIMENTAL CODE: HS_cC

[redacted]

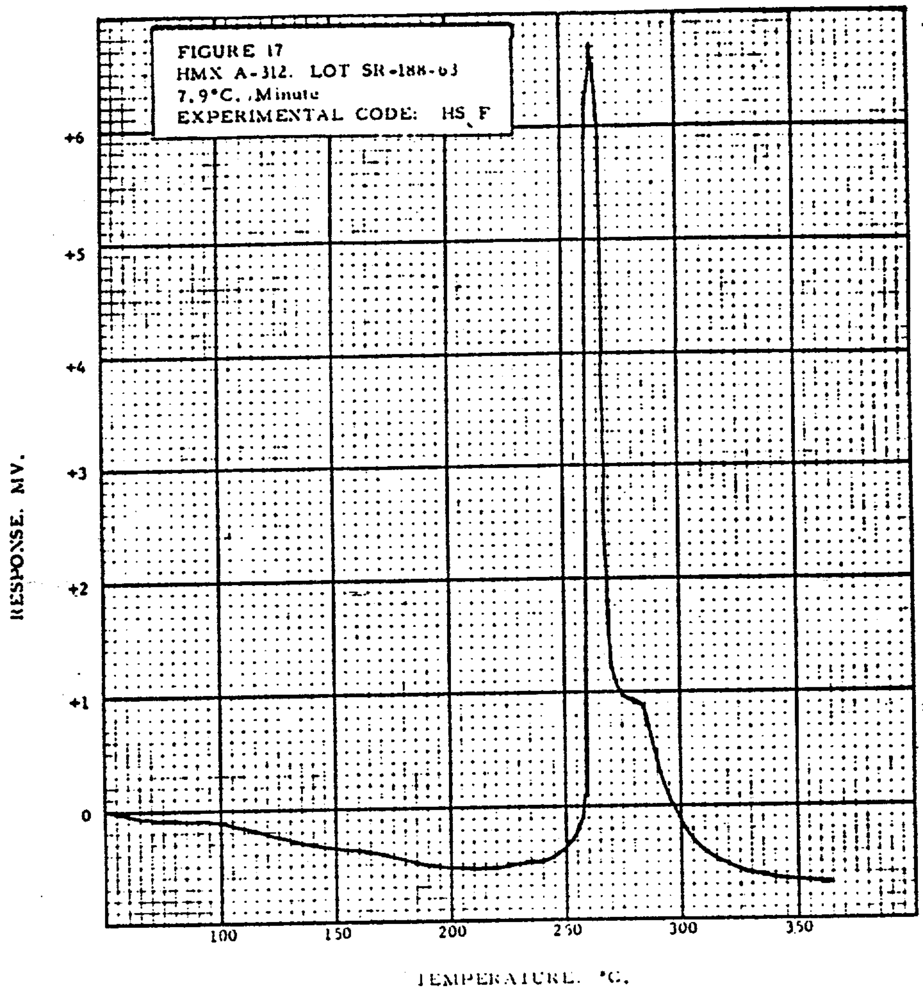


FIGURE 17
HMX A-312. LOT SR-188-63
7.9°C./Minute
EXPERIMENTAL CODE: HS F

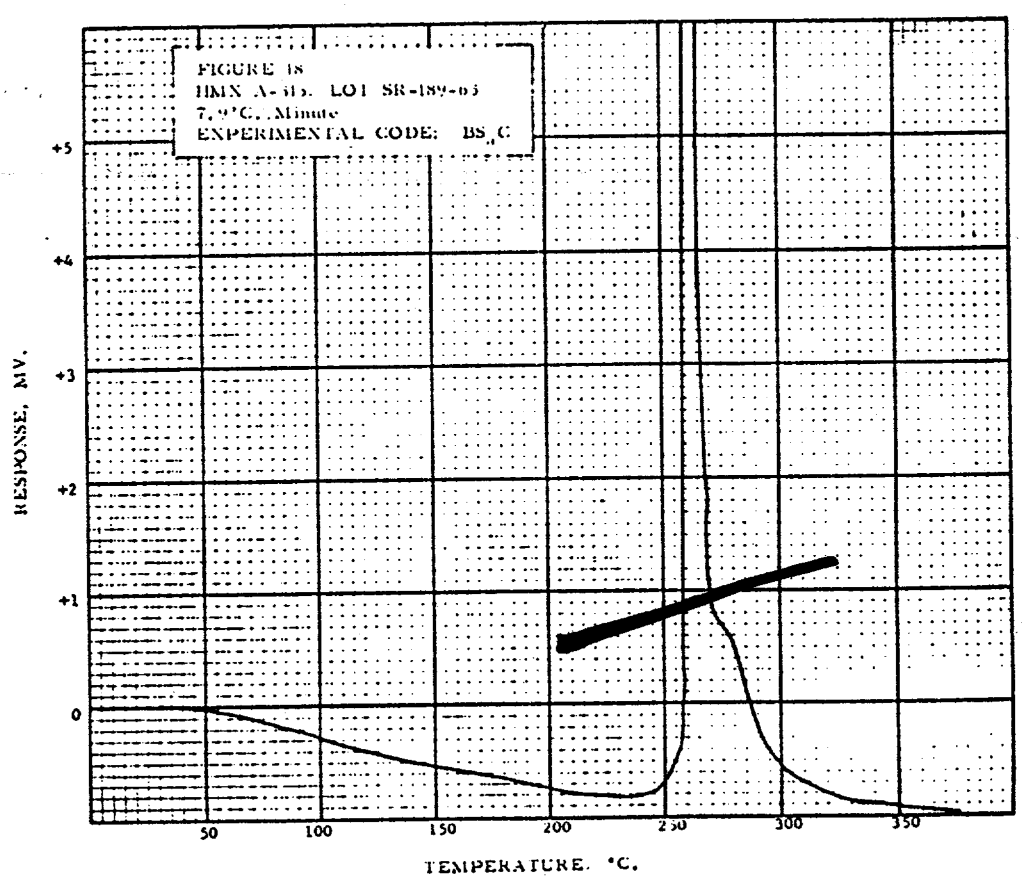


FIGURE 18
HMX A-315. LOT SR-189-63
7.9°C./Minute
EXPERIMENTAL CODE: BS C

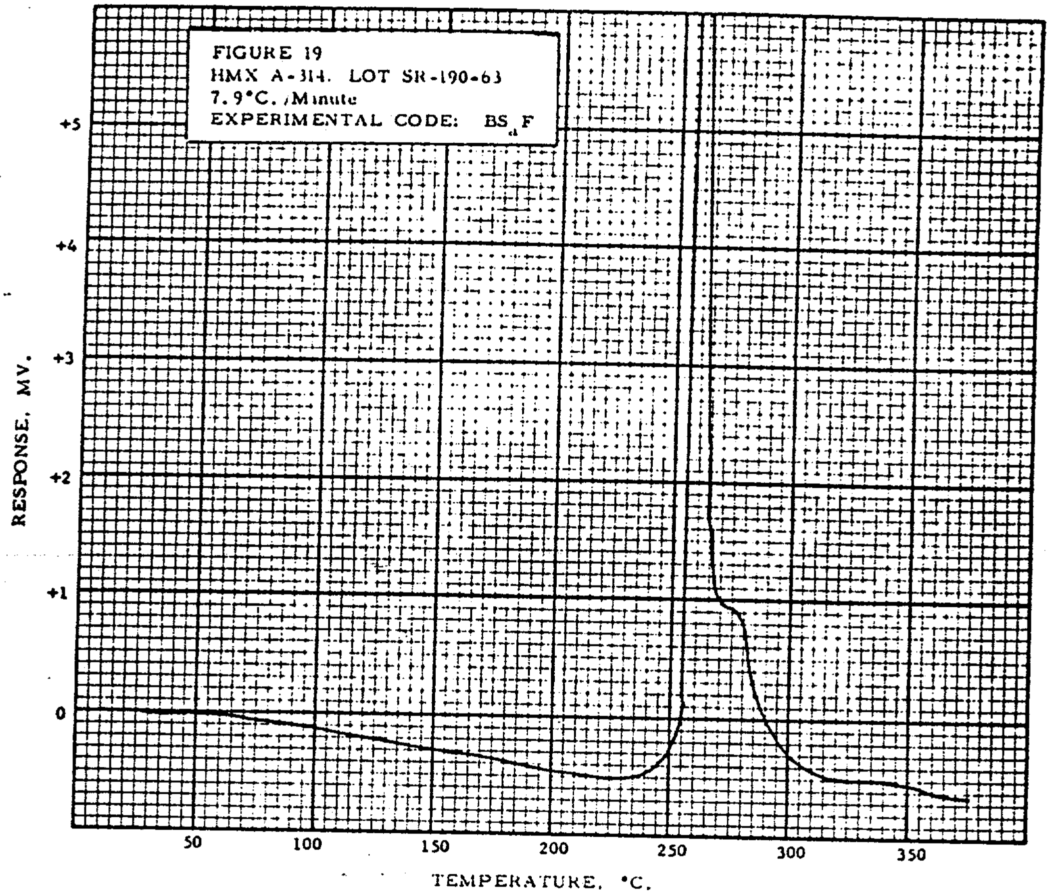


FIGURE 19
HMX A-314. LOT SR-190-63
7.9°C. /Minute
EXPERIMENTAL CODE: BS_aF

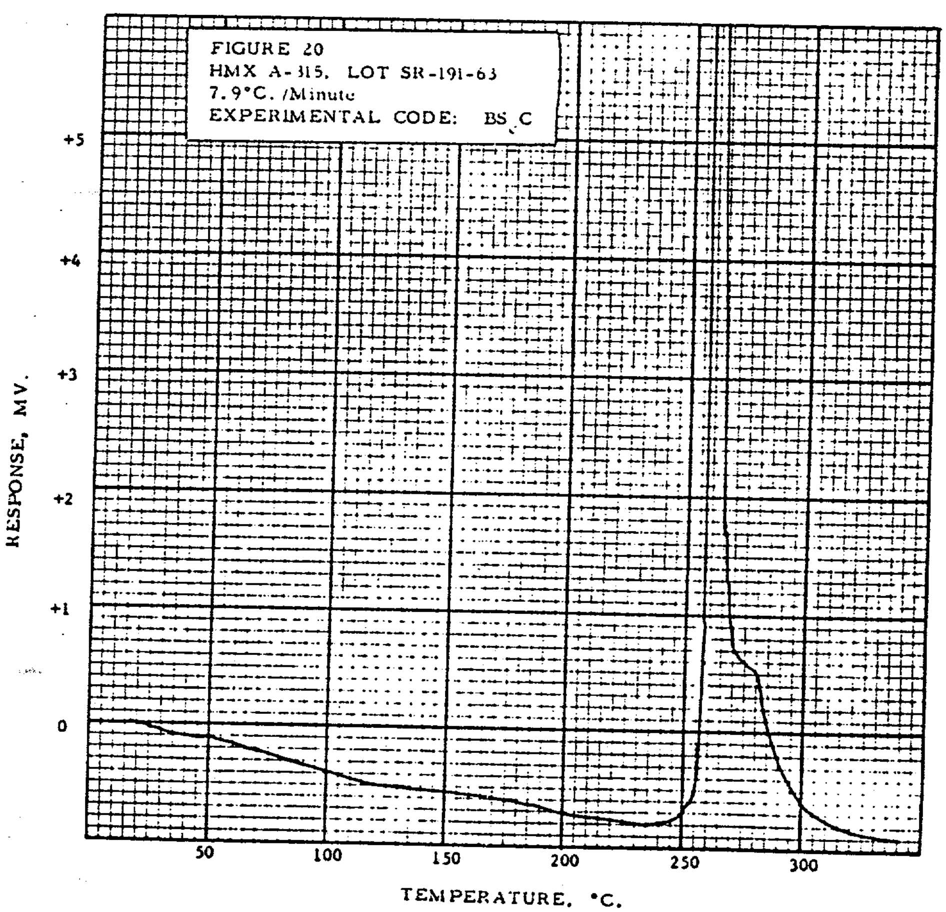


FIGURE 20
HMX A-315. LOT SR-191-63
7.9°C. /Minute
EXPERIMENTAL CODE: BS_cC

[redacted]

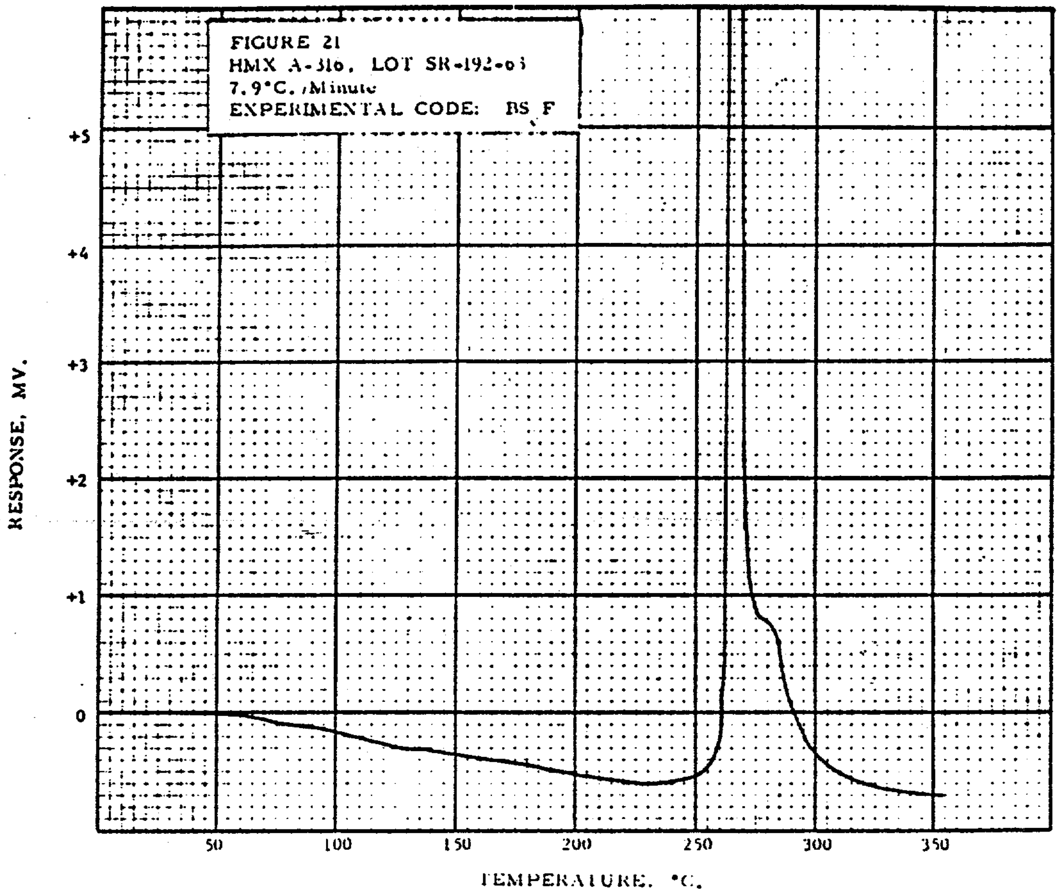


FIGURE 21
HMX A-316, LOT SR-192-63
7.9°C./Minute
EXPERIMENTAL CODE: BS F

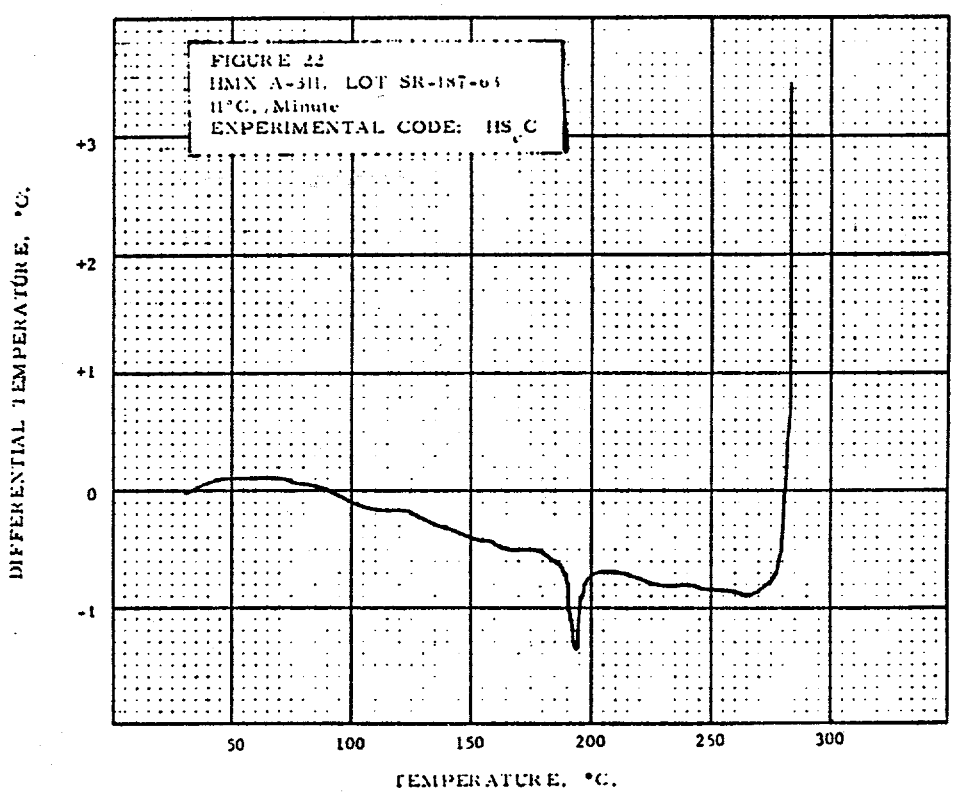


FIGURE 22
HMX A-311, LOT SR-187-63
11°C./Minute
EXPERIMENTAL CODE: HS C

[redacted]

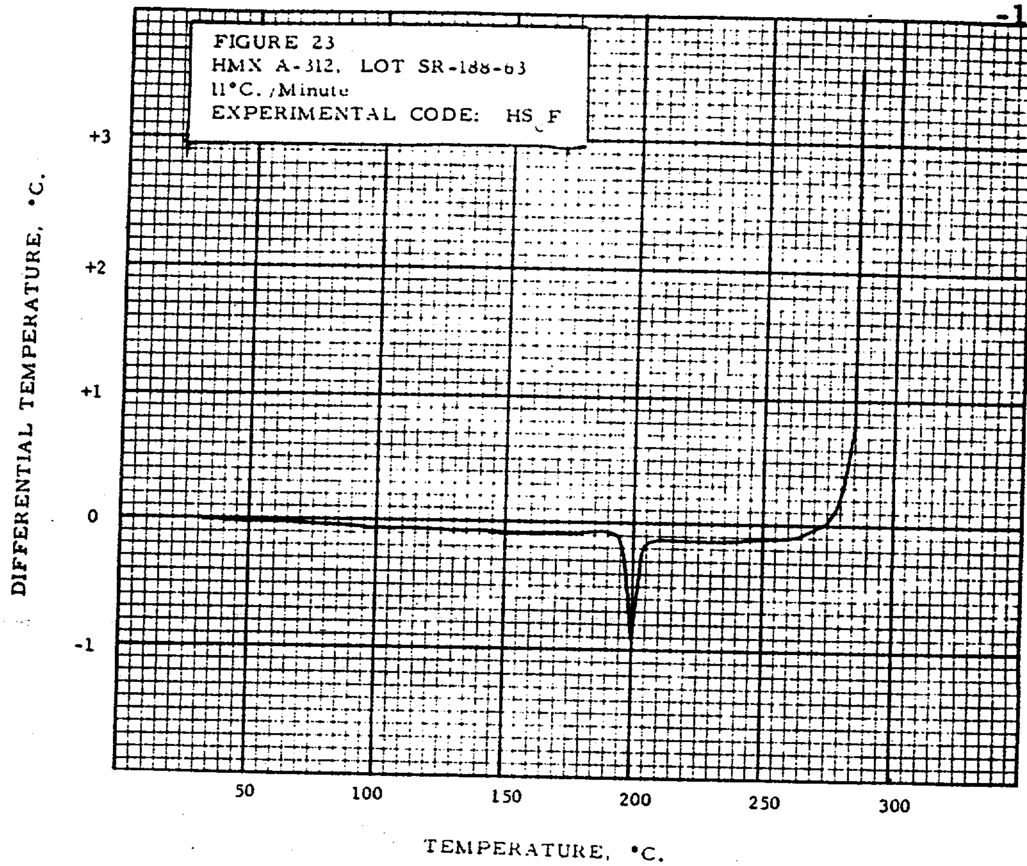


FIGURE 23
HMX A-312, LOT SR-188-63
11°C./Minute
EXPERIMENTAL CODE: HS_cF

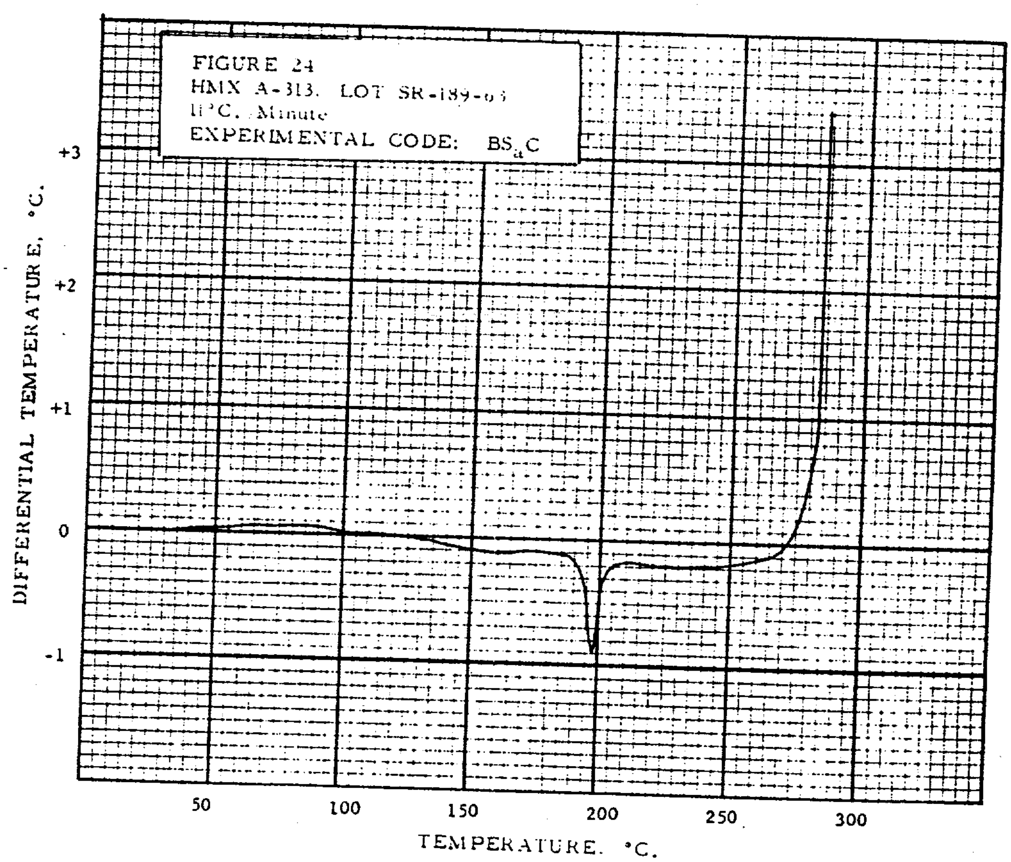


FIGURE 24
HMX A-313, LOT SR-189-63
11°C./Minute
EXPERIMENTAL CODE: BS_aC

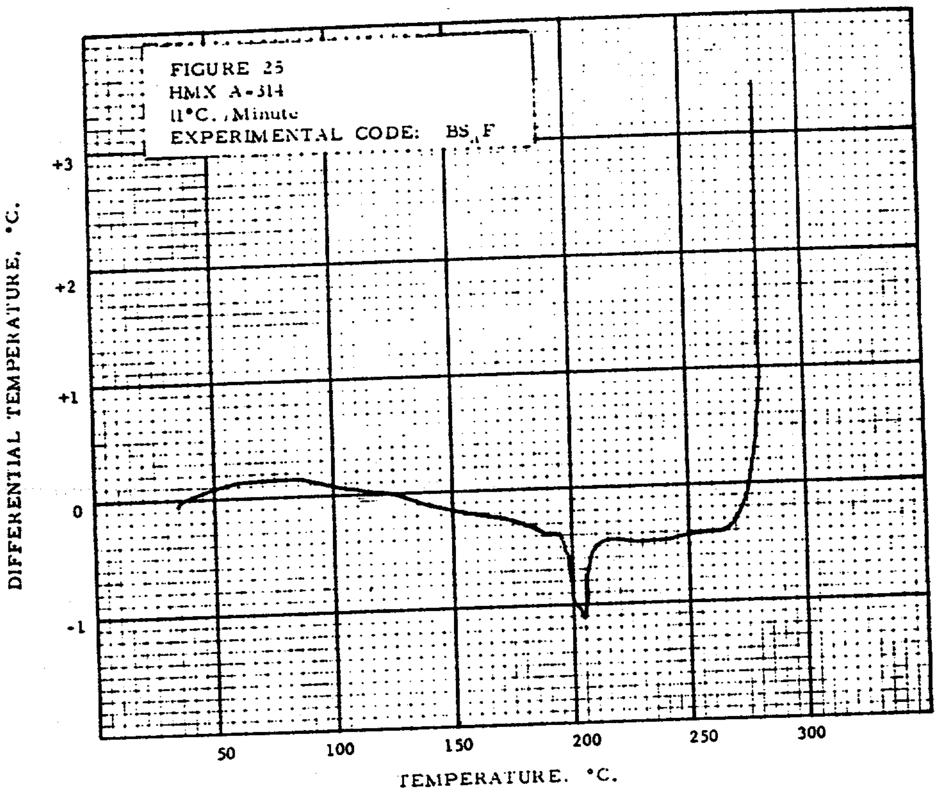


FIGURE 25
HMX A-314
11°C. /Minute
EXPERIMENTAL CODE: BS$_A$F

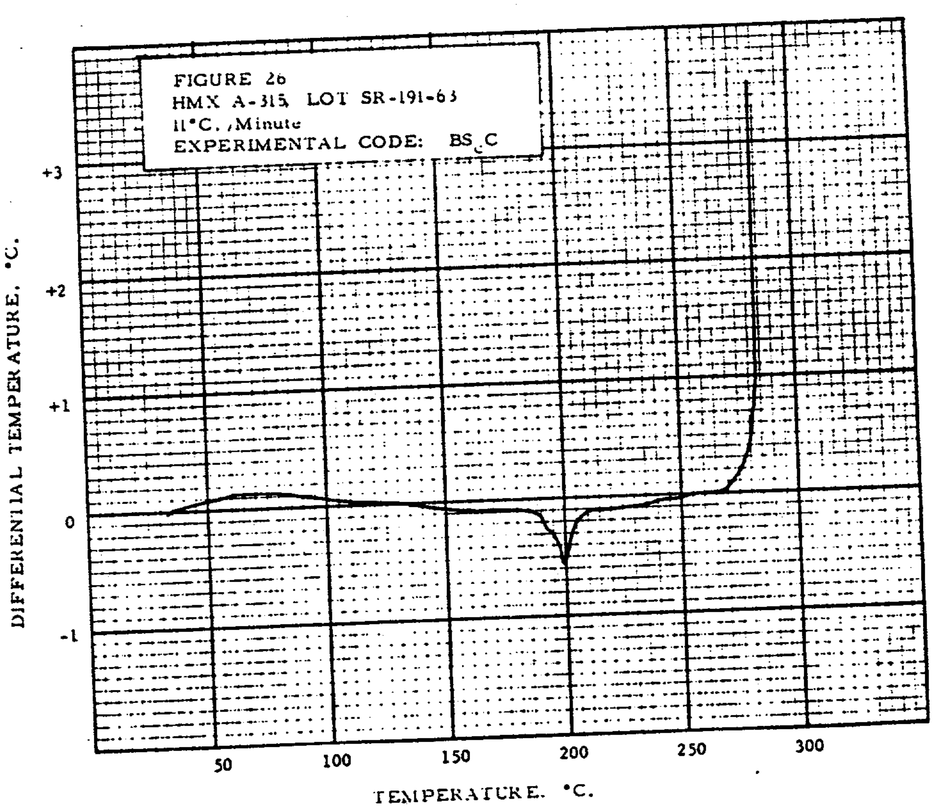


FIGURE 26
HMX A-315, LOT SR-191-63
11°C. /Minute
EXPERIMENTAL CODE: BS$_C$C

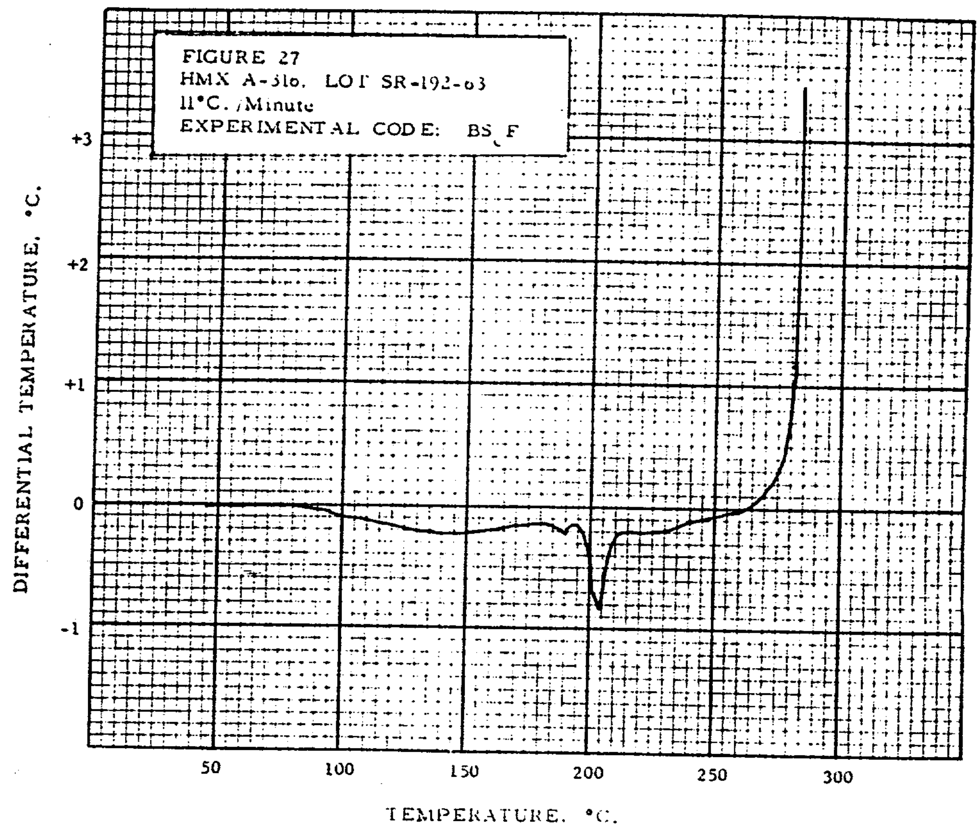


FIGURE 27
HMX A-3l6. LOT SR-192-63
11°C. /Minute
EXPERIMENTAL CODE: BS F
DIFFERENTIAL TEMPERATURE, °C.
+3
+2
+1
0
-1
50
100
150
200
250
300
TEMPERATURE, °C.

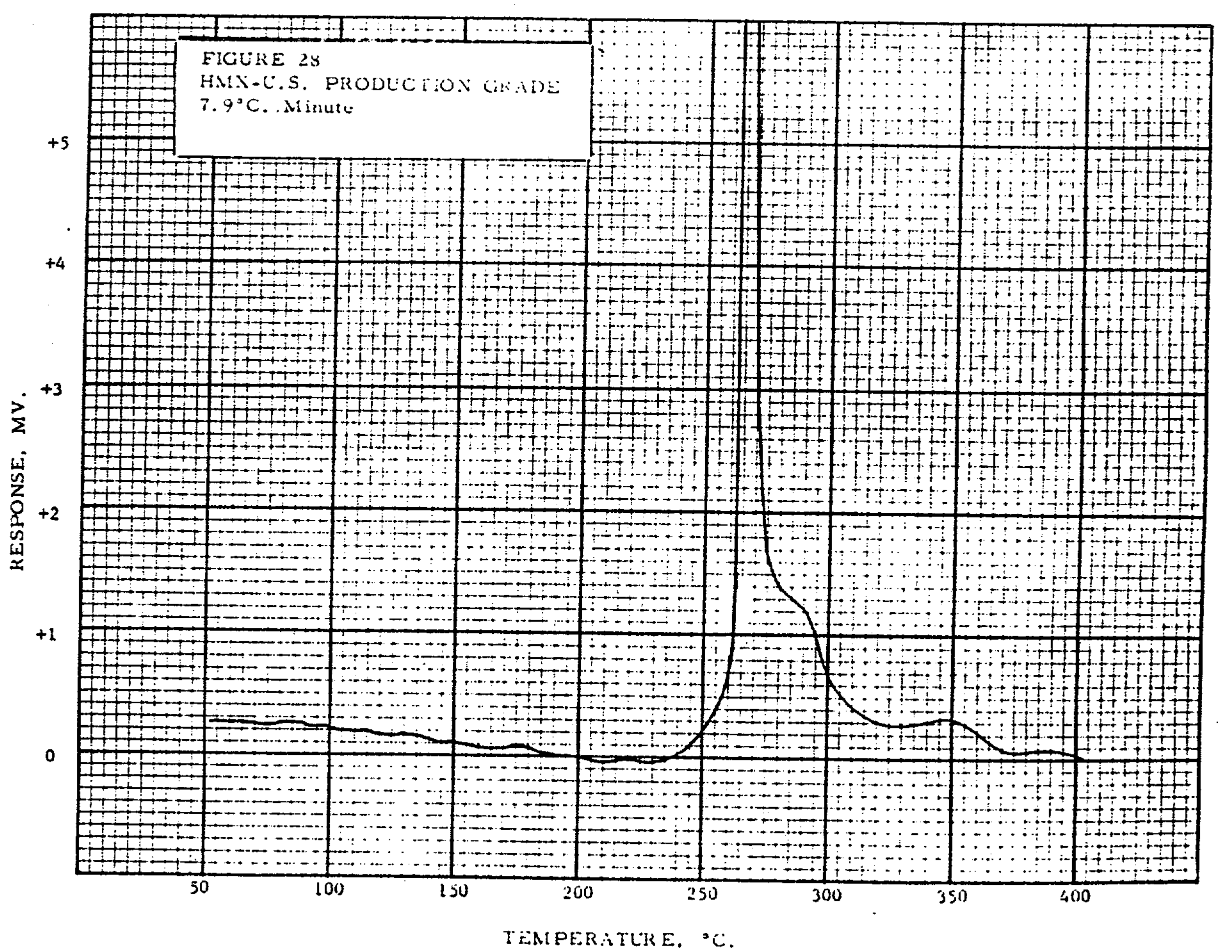


FIGURE 28
HMX-C.S. PRODUCTION GRADE
7.9°C. /Minute
RESPONSE, MV.
+5
+4
+3
+2
+1
0
50
100
150
200
250
300
350
400
TEMPERATURE, °C.

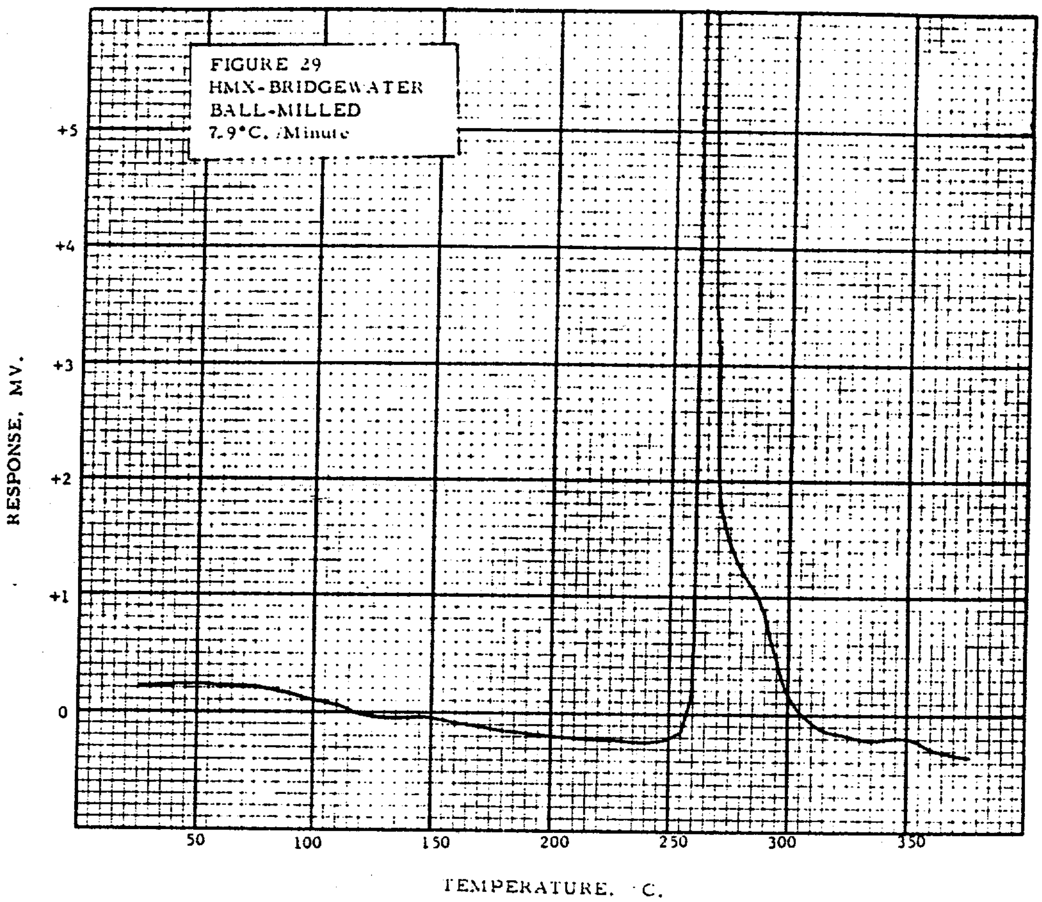


FIGURE 29
HMX-BRIDGEWATER
BALL-MILLED
7.9°C. /Minute
RESPONSE, MV.
+5
+4
+3
+2
+1
0
50
100
150
200
250
300
350
TEMPERATURE, °C.

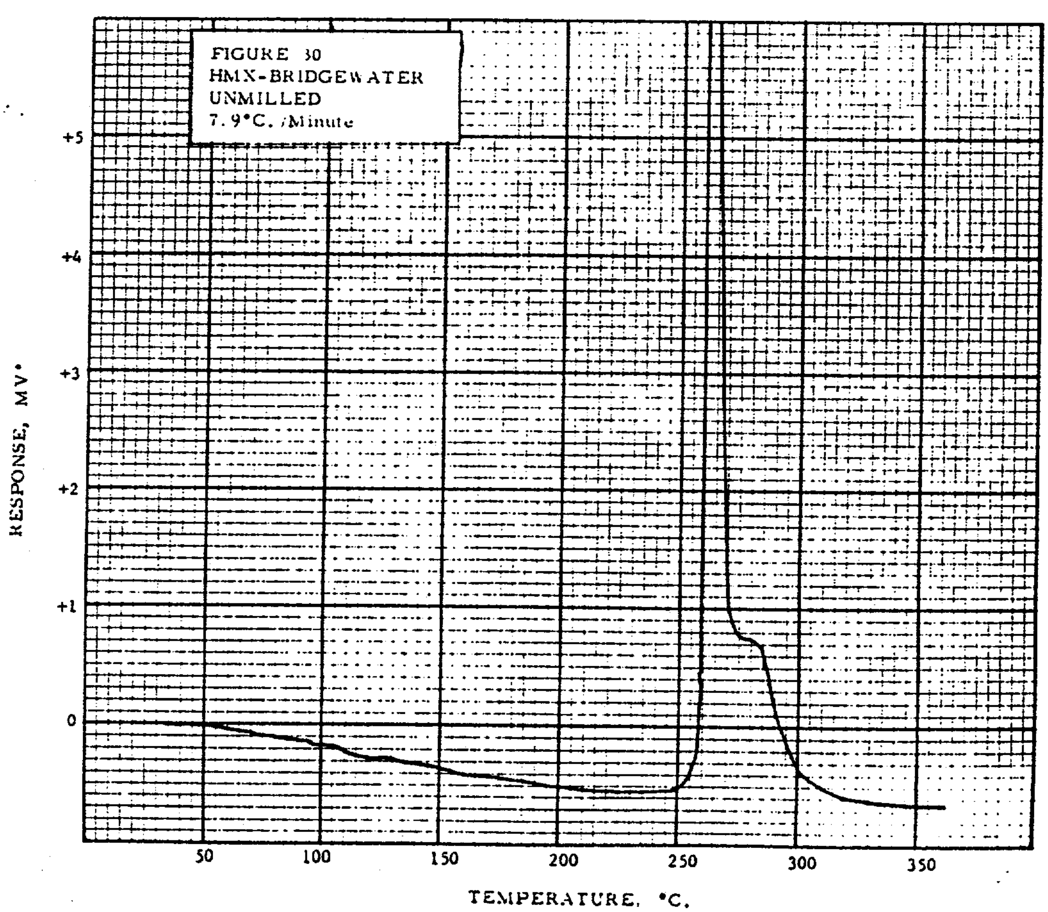


FIGURE 30
HMX-BRIDGEWATER
UNMILLED
7.9°C. /Minute
RESPONSE, MV.
+5
+4
+3
+2
+1
0
50
100
150
200
250
300
350
TEMPERATURE, °C.

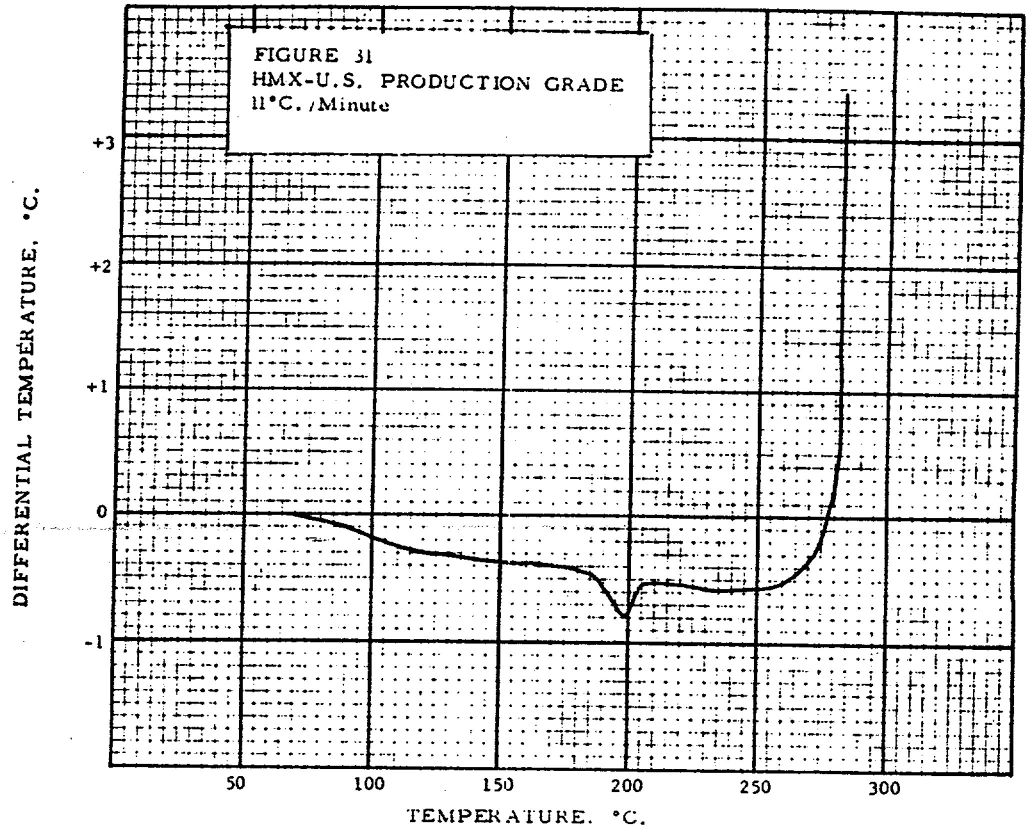


FIGURE 31
HMX-U.S. PRODUCTION GRADE
11°C. /Minute
+3
+2
+1
0
-1
DIFFERENTIAL TEMPERATURE, °C.
50
100
150
200
250
300
TEMPERATURE. °C.

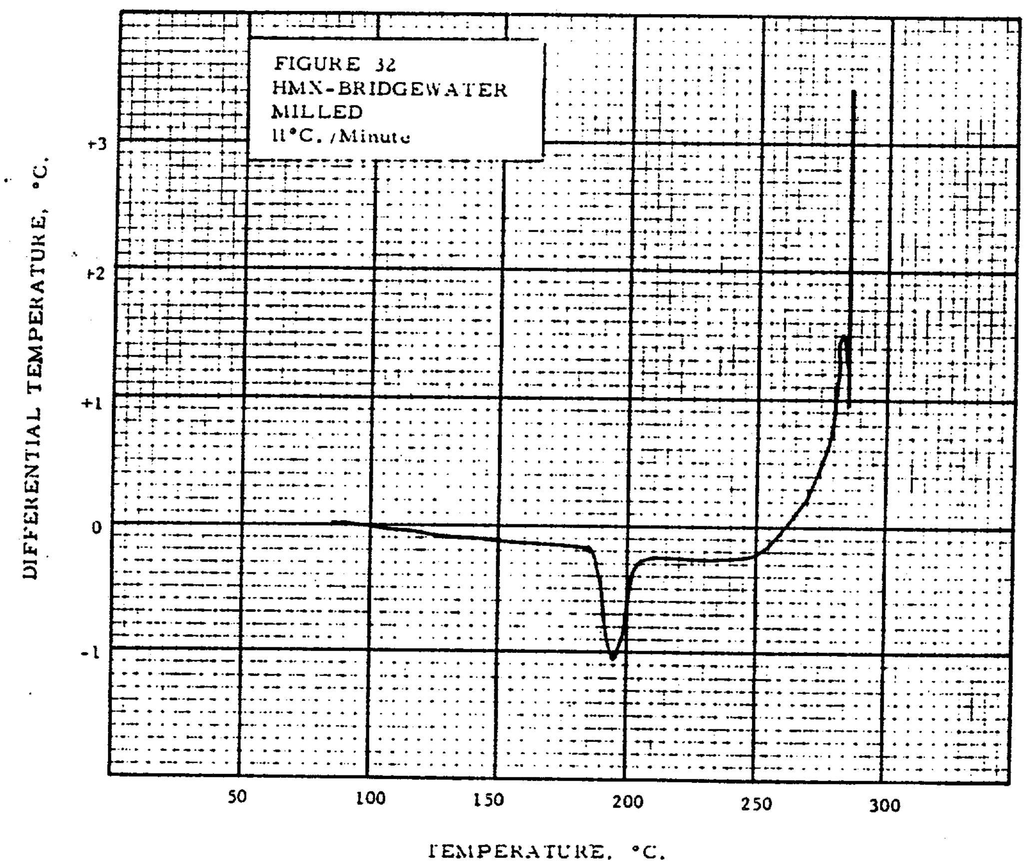


FIGURE 32
HMX-BRIDGEWATER
MILLED
11°C. /Minute
+3
+2
+1
0
-1
DIFFERENTIAL TEMPERATURE, °C.
50
100
150
200
250
300
TEMPERATURE. °C.

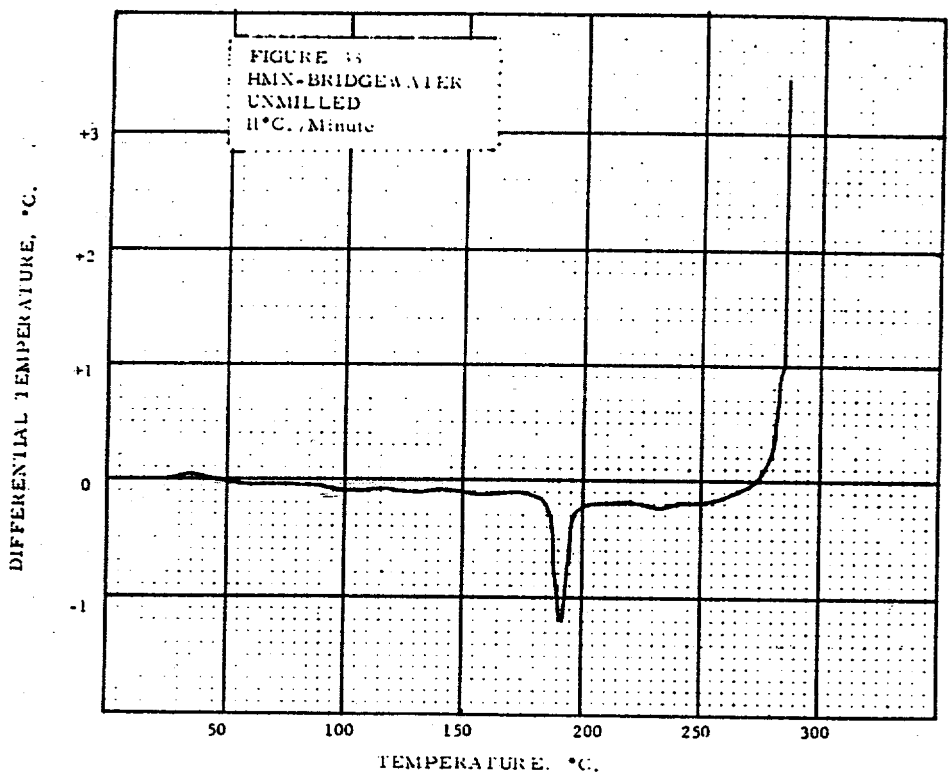


FIGURE 53
HMX-BRIDGEWATER
UNMILLED
11°C./Minute
DIFFERENTIAL TEMPERATURE, °C.
+3
+2
+1
0
-1
50
100
150
200
250
300
TEMPERATURE. °C.

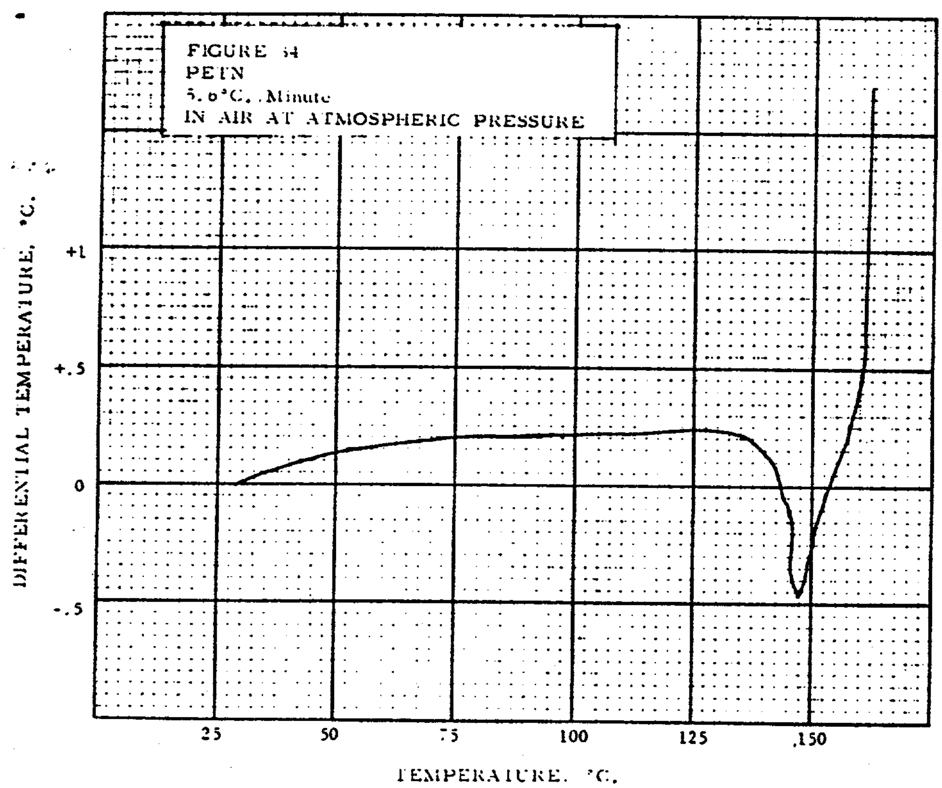


FIGURE 54
PETN
5.6°C./Minute
IN AIR AT ATMOSPHERIC PRESSURE
DIFFERENTIAL TEMPERATURE, °C.
+1
+.5
0
-.5
25
50
75
100
125
150
TEMPERATURE. °C.

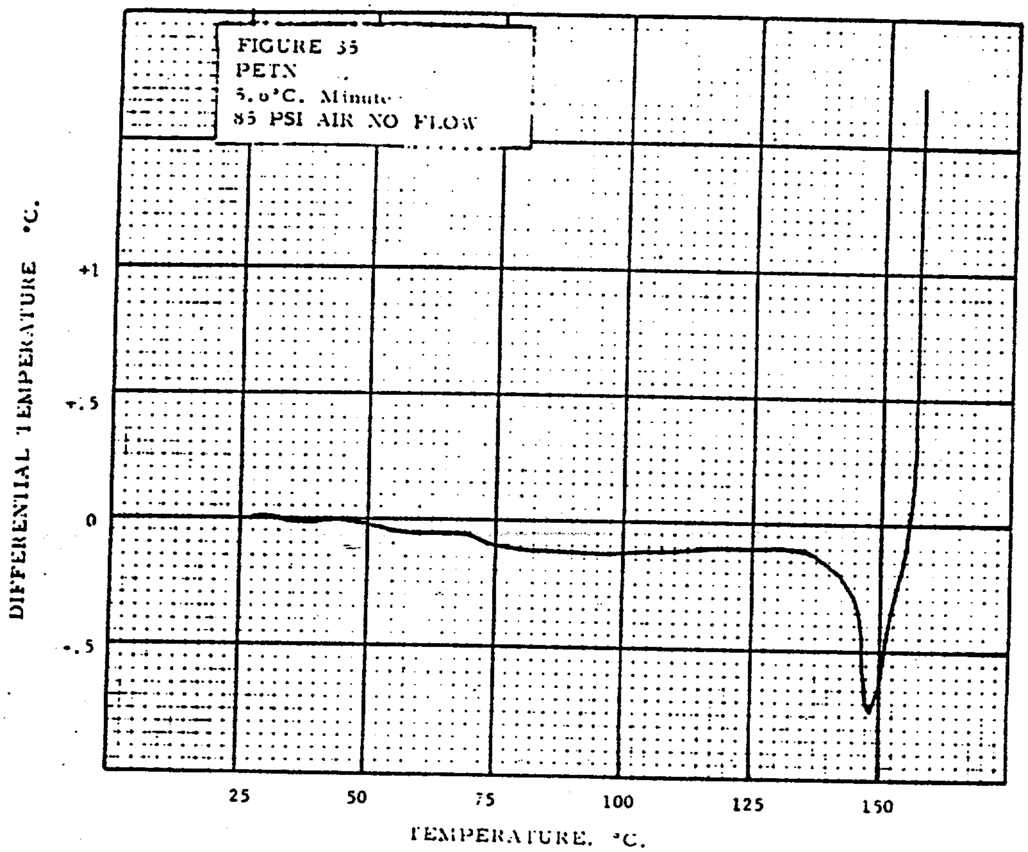


FIGURE 35
PETN
5.6°C. Minute
85 PSI AIR NO FLOW

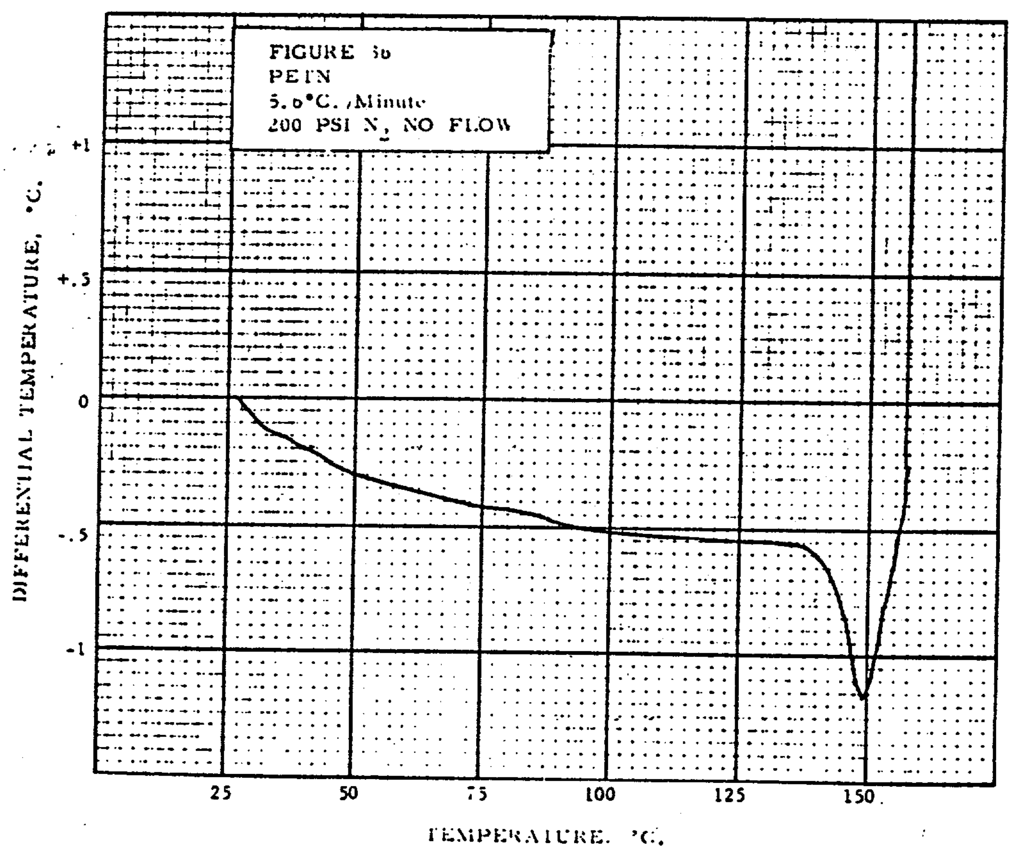


FIGURE 36
PETN
5.6°C. /Minute
200 PSI N_2 NO FLOW

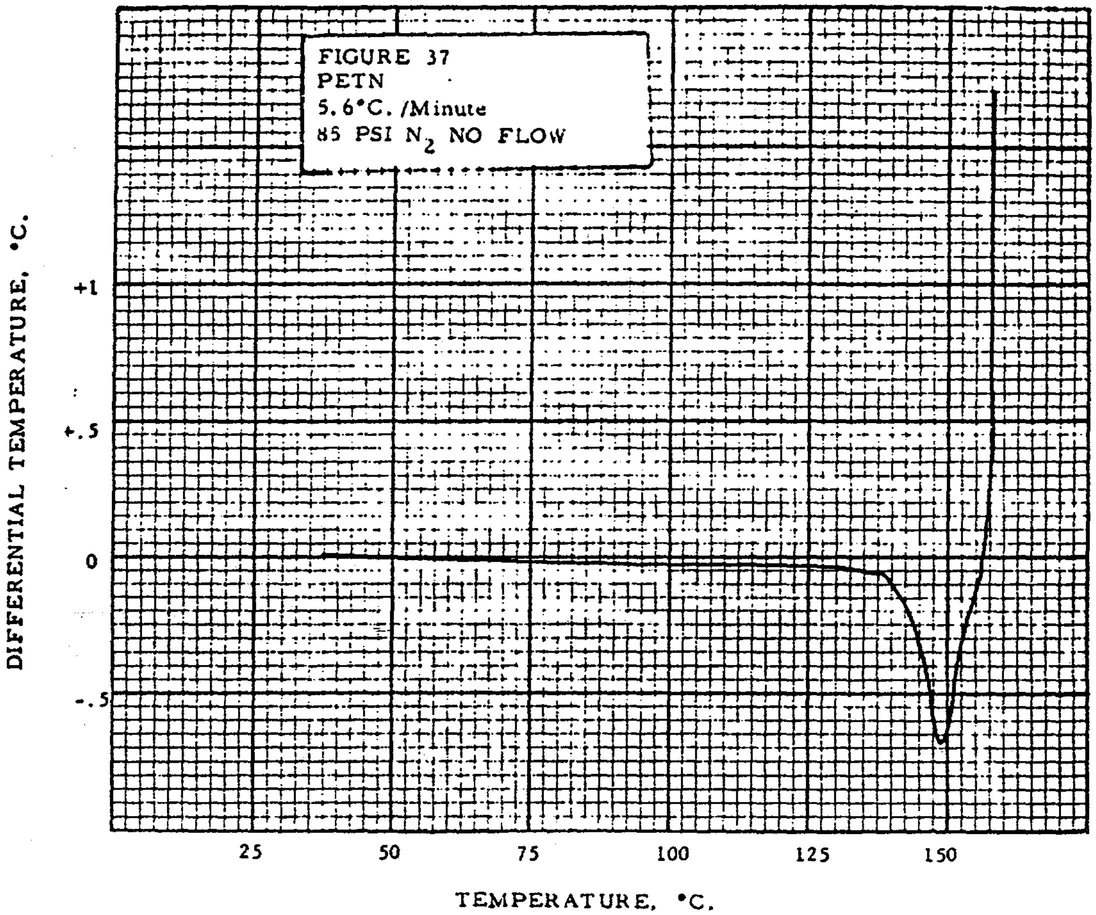


FIGURE 37
PETN
5.6°C. /Minute
85 PSI N_2 NO FLOW

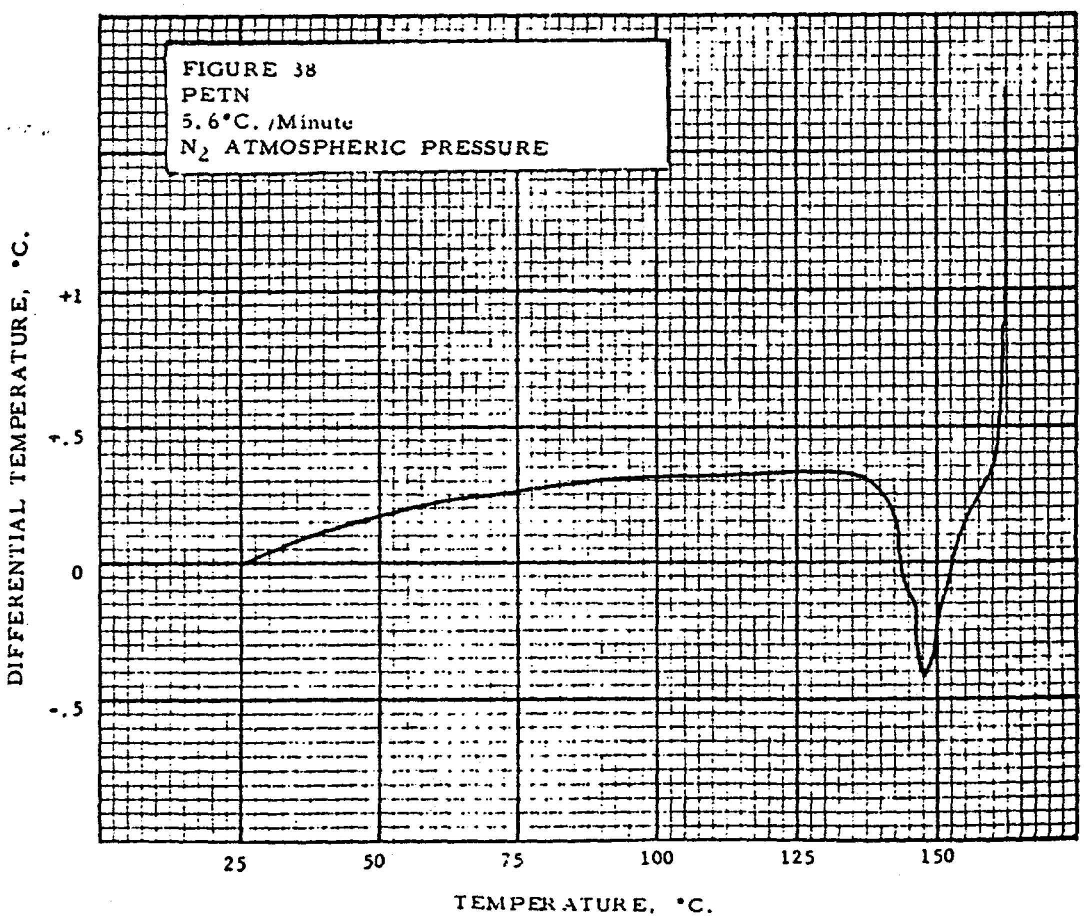


FIGURE 38
PETN
5.6°C. /Minute
N_2 ATMOSPHERIC PRESSURE

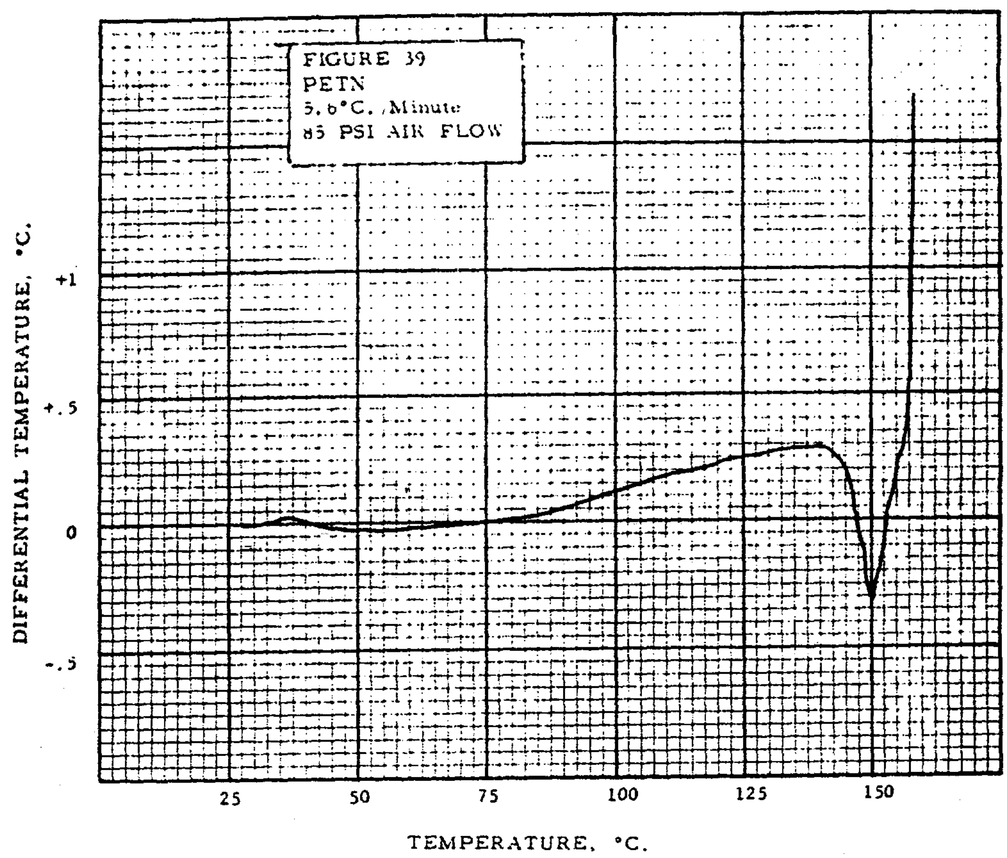


FIGURE 39
PETN
5.6°C./Minute
85 PSI AIR FLOW
DIFFERENTIAL TEMPERATURE, °C.
+1
+.5
0
-.5
25
50
75
100
125
150
TEMPERATURE, °C.

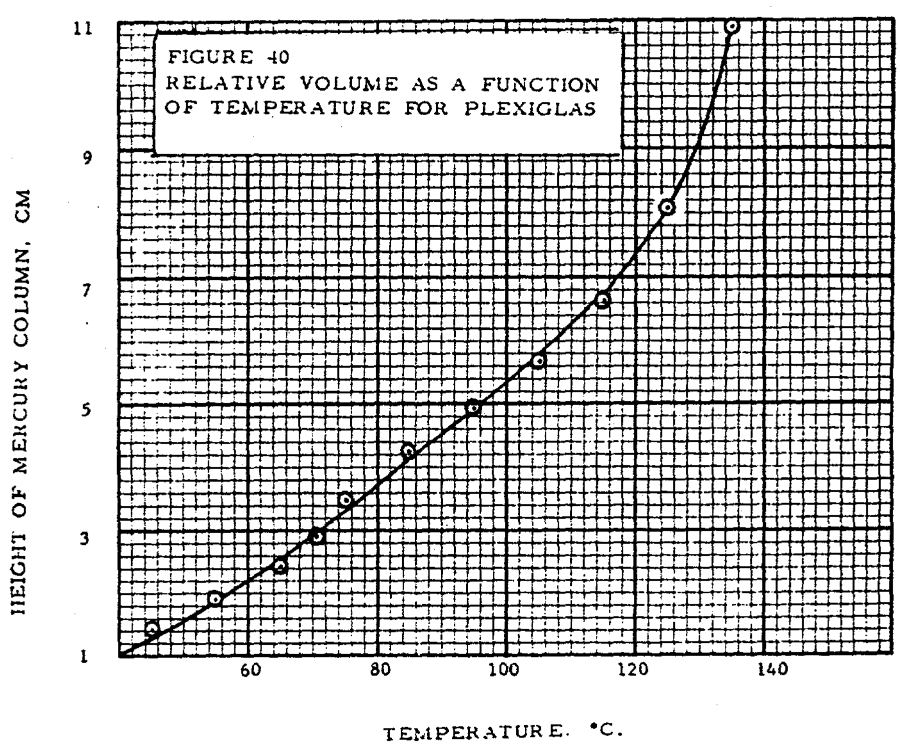


FIGURE 40
RELATIVE VOLUME AS A FUNCTION
OF TEMPERATURE FOR PLEXIGLAS
HEIGHT OF MERCURY COLUMN, CM
11
9
7
5
3
1
60
80
100
120
140
TEMPERATURE. °C.