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-96-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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Quarterly Report for April, May, June, 1964

Engineering Order No. 814-00-009

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ABSTRACT

Changes in the loading technique for the Henkin test to be used when investigating high temperature explosives are reported. Data comparing times-to-explosion for lead, aluminum "foil", insulated lead, and insulated aluminum "foil" plugs are included.

A preliminary temperature-time curve for an HMX sample in the Henkin bath at 272°C. is reported.

The pyrolysis apparatus has been reworked to increase its sensitivity and the results of the changes are readily apparent in the pyrolytic decomposition curves that are reported.

Thermal data are reported for some "tailormade" samples of HMX, samples of PETN from different manufacturers, and the six simulated samples of Bridgewater HMX. Differences within these groups are detected by one or another of the tests.

Work has begun on a two column gas chromatograph to be used in obtaining chemical reactivity data for use in specifications.

PREVIOUS APPLICABLE WORK

Thermal tests (DTA, Henkin, and pyrolysis) have been used to evaluate differences between conventional explosives, mixtures of inerts and explosives, and new explosives. Data from the tests have been reported.

DISCUSSION

This quarter's thermal work was apportioned, as usual, between apparatus (check out, calibration, interpretation of results, modifications, building) and data gathering. Since some of the data were obtained with modified instruments, we will discuss instrumentation first.

HENKIN TEST

The particular loading technique now used for the Henkin test has an upper temperature limit which is set by the melting point (327°C.) of the lead plug. Some work now being done here with du Pont's high temperature explosives (EL-511 and Tacot) has made it desirable to extend the upper temperature limit of the test. A check on the physical properties of different metals and alloys indicated that aluminum would be a likely cap sealing material to use. The most malleable type of aluminum will not deform sufficiently to make a seal when pressed at 7000 to 8000 psi; however, a satisfactory sealing plug (about one inch long) was made by pressing wadded aluminum foil strips (6 inches x 1/4 inch x 0.001 inch) in the blasting cap at 7000 psi. The explosives systems sealed with the aluminum "foil" plugs gave times-to-explosion (T.E) that were much longer than those sealed with the conventional lead plugs; this difference was believed to be due to the differences in the thermal properties of aluminum and lead. To verify this, an experiment with HMX was conducted using four different loading techniques. The first was the normal method using a gas check and lead plug; the second used a gas check and aluminum "foil" plug; the third used a gas check, cork, and lead plug; and the fourth used a gas check, cork, and aluminum "foil" plug. The results of this experiment are given in Table I.

Table I

Time-To-Explosion for HMX Using Various Loading Techniques

	<u>262°C.</u>	<u>271°C.</u>
1" Lead Plug	40.6 48.4 44.8 48.2 45.3†	25.6 21.8 33.8 29.6 27.0†
Al Foil Plug	506 665 540 623 577†	273 217 218 235 234†
Cork + 1" Lead Plug	48.4T 44.2 46.6 50.2T 47.2†	23.8 24.2 29.4 32.2 27.0†
Cork + 1" Foil Plug	46.8 54.2 58.6 42.2 49.6†	27.8 28.8 31.6 33.8 30.3†

†The harmonic mean of the times
T blew plug out top

A second experiment to determine how much the cork would decompose and affect the test at high temperatures was conducted with a cork as the sample. When loaded in the normal way and tested at 270°C., the lead plug was blown out the top of the blasting cap in 700 to 800 seconds. This indicated considerable decomposition of the cork, and that cork would not be satisfactory for the insulating material.

Some preliminary tests were made in an attempt to measure the temperature of the H.E. sample as a function of time in the Henkin test. The technique used to measure the sample temperature consisted of drilling a 0.140 inch diameter hole through two gas checks and the interposed 1-inch lead sealing plug. A #30 copper-constantan thermocouple was threaded through the holes as shown in Figure 1. The gas check on top of the lead plug kept the lead plug from extruding into the hollow punch when the assembly was pressed at 7000 psi. The thermocouple was connected to the Y-axis of a Mosely recorder; the X-axis was time in seconds. A typical run with HMX is shown in Figure 2.

PYROLYSIS APPARATUS

It was reported that some changes would be made on the pyrolysis apparatus to increase the sensitivity and accuracy. This has been done. The 1/4-inch copper tubing has been replaced with 1/8-inch stainless steel, the positioning of the components has been changed to reduce tubing lengths, and the original thermal conductivity detector was replaced with a thermostated Gow Mac Model 9285 T.C. detector. The only change in the operating procedure was to reduce the gas pressure from 15 to 10 psi with a flow rate of approximately 25 cc/minute.

To calibrate the temperature scale, a special plug (Figure 3) was made for the pyrolysis block. A hole was drilled through the plug to permit insertion of a two-hole thermocouple insulator (0.16-inch O.D.). The insulator and thermocouple leads were cemented to the plug with "Sauereisen" cement for a gas tight seal.

DTA

The controlled atmosphere/pressure DTA is ready for operating at higher pressures (up to 2000 psi), but due to the priority of other projects, no thermographs were run this quarter. Its design does work satisfactorily for controlling the pressure; however, it could be improved to allow better flushing of the sample; i.e., better controlled atmosphere. It should supply enough information to determine if an improved design would be worthwhile.

GAS CHROMATOGRAPHY

The necessary electronics for the single-column chromatograph are almost complete and the other components are ready for assembly. However, since Pantex has been asked by IRL to build a two-column system, the design of the single-column chromatograph will be altered such that a second column can be added with a minimum of effort. This plan should allow Pantex time to get one operating properly before the additional components for the second column are ready to add. The design will be similar to the Royal Research instrument, but will not duplicate it.

THERMAL DATA

Pyrolytic analysis of Holston's simulated Bridgewater HMX's were rerun with the modified pyrolysis apparatus. These samples carry the LRL raw material numbers A-311 through A-316 and the LRL code designations of HS_cC, HS_cF, BS_aC, BS_aF, BS_cC, BS_cF, respectively. The code designation meanings are:

- H - The HMX was prepared by the normal Holston process with the simmering done at 85% acid concentrations.
- B - The HMX prepared by a Bridgewater-like process; i.e., simmer at 70% acid concentration, decant, wash and boil in H₂O before final recrystallization.
- S_c - Recrystallize from cyclohexanone.
- S_a - Recrystallize from acetone.
- C - Coarse particle size, direct from crystallizer. Close to Grade A distribution.
- F - Fine particle size, ground by centrifugal pump. Grade B modified for LX-04-1 distribution.

The pyrolograms are shown in Figures 4 through 9.

Extensive thermal analyses were requested by LRL personnel on a three-sample HMX series to see if differences between them could be detected. The samples were A-311 (described above), specially processed/purified (by Dave Coleman, LRL) A-311, and 99.9 + % pure HMX. The first two samples had LRL designations 1-U and 1-P. Some additional experiments were performed on two other materials for further comparison; namely, U.S. Grade II, Class E, HMX and Bridgewater HMX. The Henkin data and DTA and pyrolysis curves are given in Table II and Figures 10 through 26.

Table II

Time-To-Explosion for HMX of Varying Types

<u>HMX</u>	<u>230°C.</u>	<u>240°C.</u>	<u>250°C.</u>	<u>260°C.</u>	<u>270°C.</u>	<u>280°C.</u>
99.9% Pure	596	328	201	105	40.0	26.0
	571	352	217	106	48.1	23.4
	556	337	202	121	45.6	22.6
	588	325	208	113	49.3	23.6
	525	298T	209	110	49.3	28.0
	567†	327†	207†	110†	46.2†	24.6†
Unprocessed A-311	260	127	87.4T	58.0	27.2	17.6
	263	146	87.0	55.8	30.8	16.4
	258	138	88.8	53.3	30.7	17.0
	297	430NE	105T	55.6	34.6	18.6
	400NE	480NE	86.0	52.0	30.8	19.8
	269†	137†	90.3†	54.8†	30.7†	17.8†
Processed A-311	384	219	147	74.4	33.8	24.4
	416	239	157	77.1	33.2	19.4
	399	223	151	81.2	40.2	22.3
	425	249	150	73.6	40.7	20.2
	386	222	150	79.6T	41.0	23.6
	401†	230†	151†	77.0†	37.4†	21.8†
Class E Grade II	238	300NE	88.0	49.0	26.6	14.9
	221	147T	82.4	47.8	29.8	15.2
	263	127	83.6	47.2	29.2	16.6
	243	142	83.2	45.5	29.6	17.6
	236	135	82.5	45.2	31.0	22.2
	239†	137†	83.9†	42.9†	32.2†	16.9†

†The harmonic mean of the times

T blew plug out top

NE No explosion

LRL representatives also asked that PETN samples of different origin be subjected to a critical thermal analysis. Again, the purpose was to see if the thermal tests could differentiate between them. The samples were du Pont A-326, Mound's detonator trade and Trojan's special. Henkin, DTA, and pyrolysis results are shown in Table III and Figures 27 through 38.

FUTURE WORK; COMMENTS; CONCLUSIONS

The data (Table I) for the experiment to determine why the system sealed with the aluminum plugs gave longer time-to-explosion (T.E.) than those sealed with the lead plugs indicates that it was a result of heat transferred from the sample. The T.E. for systems using a cork insulator between the sample and aluminum plug were in reasonable agreement with the T.E. obtained using a lead plug. Also, there was no significant difference in the T.E. for the insulated lead plug systems, and those that were not insulated from the sample. This indicates that the lead plug does not transfer a great amount of heat from the sample and consequently is a good plug material. During the evaluation of cork insulators for "high" temperature work, it was discovered that corks decomposed so much (at 270°C.) that they blew the lead plugs out the top of the caps. Obviously, cork is not the best choice for the insulating material; other materials such as NaCl will be tested in the future.

Preliminary attempts to monitor the temperature of the sample during Henkin tests show only a slight, constant increase in temperature after the initial rise due to insertion in the bath. A greater increase (ours were barely detectable, if real) in temperature was expected just before the explosion; the large increase

Table III

Time-to-Explosion for PETN of Varying Types

<u>PETN</u>	<u>170°C.</u>	<u>180°C.</u>	<u>190°C.</u>	<u>200°C.</u>
Du Pont	183	86.6	30.5	14.6
	166	77.2	31.2	16.0
	500NE	79.4	30.8	15.7
	183	87.4	31.6	16.8
	178	77.8	30.5	16.2
	177†	81.4†	30.9†	15.8†
Trojan	151	80.6	33.6	17.7
	149	84.6	35.2	16.8
	207	81.2	33.9	17.6
	325	81.2	35.5	17.8
	179	80.4	32.4	17.3
	186†	81.6†	34.1†	17.4†
Mound	196	82.0	34.0	17.5
	287	80.4	33.9	16.0
	330NE	78.8	31.9	17.2
	201	76.8	33.2	17.8
	157	78.0	32.6	18.0
	201†	79.2†	33.1†	17.3†

†The harmonic mean of the times
NE no explosion

accompanying the explosion was not observed because of the long response time of the recorder (1/2 second) and thermocouple. In the future, an attempt will be made to use an oscilloscope to observe the temperature just before the time of explosion.

The pyrolytic decomposition curves (Figures 4 through 9 for the samples of Holston-made simulated Bridgewater HMX (A-311 through A-316) show some differences. We are not sure to what these differences are attributable; but suspect mother liquor gives the lower temperature peaks and RDX is associated with the peak appearing just before complete pyrolysis (see Figures 39 and 40 for Bridgewater and Bridgewater + 2% RDX). The peak positions shift in some samples for unknown reasons. LRL personnel have asked that complete Henkin data be obtained on these samples to help elucidate the results obtained so far.

The Henkin test data (Table I) for the HMX sample (unprocessed A-311, processed A-311, Grade II, Class E, and 99.9% pure) show differences between samples. The effect of particle size has not been determined and was not controlled in this experiment. The data appear to segregate the samples according to purity. The Class E and unprocessed A-311 gave similar, relatively short times; the processed A-311 gave intermediate times and the 99.9% pure sample gave considerably longer times. Pyrolysis curves for these materials are somewhat different and about the same thing may be said here as was said about the pyrolysis data for the A-311 samples. The only significant differences in the DTA thermograms (Figures 18 through 26) are with the processed A-311 sample. The two first runs that were made showed a small endotherm at about 207°C. (see Figure 19). The thermograms

(Figure 20) made from two to three weeks later do not show the endotherm; but do show a shoulder on the major endotherm at 190°C. This has been reproduced a number of times.

The Henkin test data and DTA thermograms (Table I and Figures 27 through 38) for the different PETN samples show no significant difference between samples. The Henkin data are not as complete as would be desired because for temperatures below 170°C. a large number of the caps leaked, resulting in no explosion times. The pyrolytic decomposition curves show some impurity in the du Pont and Mound samples, but not in the Trojan samples.

In addition to the work already described and routine tests on in-plant samples, experiments are planned to study the effects of particle size on the Henkin test, to investigate the effect of RDX impurities in HMX, to complete the calibration and determine the optimum operational conditions for the pyrolysis apparatus, and to complete the building of the two-column chromatograph.

Figure 1