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CLASSIFICATION OF LIQUID GUN PROPELLANTS AND RAW MATERIALS FOR TRANSPORTATION AND STORAGE

Prepared by

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US ARMY ARMAMENT RESEARCH AND DEVELOPMENT COMMAND
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ABERDEEN PROVING GROUND, MARYLAND

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) Nine materials have been tested and classified on the basis of the military explosive classification system as described in Department of the Army Technical Bulletin C1, TB 700-2. Since the test methods described in TB 700-2 are designed for solids, rather than liquids, a test program was designed in which nine tests (relating specifically to unique problems and situations encountered in the liquid phase) (continued)		

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were substituted for the specified TB 700-2 tests. Where the originally proposed tests were considered to be overly rigorous, additional tests were performed to better simulate the TB 700-2 protocol. Concordance of the tests with those described in TB 700-2 was described, and a testing protocol was developed.

This protocol was applied to the classification of the nine materials and each material was classified according to the TB 700-2 classification scheme. The three fuels (IPAN, TMAN, TEAN) and the three oxidizers (HAN 2.8 M, 11 M, 13 M) were considered unrestricted under TB 700-2; LGP 1776, LGP 1845 and NOS 365 were classified Military Class 3 (DOT Class B).

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INTRODUCTION

This report summarizes the results of an experimental program performed for ARRADCOM, CML/Ballistics Procurement Division, Aberdeen Proving Ground (Edgewood Area), Maryland, by Hazards Research Corporation, Rockaway, New Jersey, under Contract Number DAAK11-78-C-0024. Contact with Aberdeen Proving Ground was maintained through Mr. Kenton E. Travis, DPB.

The purpose of this program was to evaluate the hazardous properties of constituents and formulations of candidate liquid gun propellants for the purpose of classification in transportation.

Department of the Army Technical Bulletin C1, TB 700-2 provides a protocol for classification purposes; however the test procedures are designed for solids, rather than reactive materials in the liquid phase. Therefore, the test procedures used in this program were adaptations of procedures used for liquid propellants and reactive materials as practiced by Hazards Research Corporation.

During the development of the experimental program, methods were selected to provide as close a correlation as possible with the protocol specified for solids in TB 700-2. Although there is not an ideal one-to-one correlation between tests, as more than one "liquid" test may be required to yield data comparable to one "solid" test, or vice versa, the overall evaluation results provide essentially the same information for classification purposes.

EXPERIMENTAL PROGRAM

Materials

The following materials were supplied by ARRADCOM for use in the test program:

- (1) 11 Molar HAN
- (2) 13 Molar HAN
- (3) 2.8 Molar HAN
- (4) TEAN
- (5) IPAN
- (6) TMAN
- (7) NOS 365
- (8) LGP 1776
- (9) LGP 1845

Description of Experiments

Task 1 - Trauzl Tests

This test is primarily a measure of the explosive power of the sample material, although it also provides information on the ease of initiation. In the test a glass vial containing a weighed quantity of the sample is placed in a lead cylinder (1/2" wall) adjacent to a No. 8 blasting cap. The cap is electrically activated and the volume increase of the cylinder noted. The volume increase (less that obtained in a blank run) divided by the mass of sample gives the specific expansion in cc/g.

Samples were run in duplicate in one and two gram loadings.

Task 2 - JANAF Thermal Stability

The JANAF thermal stability test is the standard test designed by the ICRPG for testing the thermal sensitivity of propellants. The test fixture is a stainless steel cylinder 0.22 inches in diameter by 1-1/2 inches long, closed at the bottom with a shielded thermocouple and compression fitting. The fixture is charged with 0.5 cc of sample and closed at the top with a stainless steel diaphragm

0.003 inches thick. The assembly is then placed in a bath which is heated at a constant rate of 100°C/minute. A second thermocouple and an X-Y recorder are connected with the sample thermocouple so as to yield a plot of differential temperature (sample temperature minus bath temperature) versus bath temperature. Exothermic reactions appear as positive peaks, endothermic reactions as negative peaks. Results are reported in terms of the temperature at which significant thermal activity is observed.

Task 3 - Impact Tests

a) Liquids

The impact test used for oxidizers and propellants was the standard ICRPG test for liquids. In this test a small sample of liquid (0.03 ml) to be tested is enclosed in a cavity formed by a steel cup, an elastic ring, and a steel diaphragm. A piston rests on the diaphragm and carries a vent hole which is blocked by the steel diaphragm. A 2 kg. weight is dropped onto the piston. A positive result is indicated by puncture of the steel diaphragm accompanied by a loud noise or severe deformation of the diaphragm and evidence that the sample was completely consumed. Data is reported as the height which yields a 50% probability of initiation. Tests are performed up to 36 inch drops, at which point the hydrostatic pressure developed by the impact is sufficient to burst the diaphragm even with non-explosive materials (e.g.-water).

b. Solids

The fuels, which are solids at ambient temperature, were tested utilizing the standard HRC Drop Weight Apparatus for solids. In this test a 2 kg. weight is dropped from various preset heights onto a carefully designed cup or holder containing the sample. A loud report, flame or other signs of combustion are all taken as signs of a positive test. Data is reported as that height which yields a 50% probability of initiation. Tests are performed up to 48", the maximum drop height available on this apparatus.

Task 4 - Detonation Velocity Determination

Detonation velocity experiments are conducted using sample containers fabricated from 8" long Schedule 80 stainless steel 2" internal diameter. The bottom of the tube is sealed with a thin non-reactive plastic diaphragm. A high-energy donor charge (160 gm. RDX) placed directly below the diaphragm acts as initiator. A cold-rolled steel

plate 4" x 4" x 0.375" placed atop the fixture serves as a witness plate.

Each test fixture is equipped with a constant-current resistance wire circuit for measurement of detonation velocity. As the detonation wave passes up the fixture, its accompanying shock wave crushes a thin-walled aluminum tube onto an enclosed resistance wire, causing a drop in voltage in the constant current circuit. This voltage drop, directly proportional to the wire length consumed, is recorded on an oscilloscope. The propagation velocity, obtained from the voltage-time record, is generally an unambiguous method for determining whether a detonation has occurred. The condition of the witness plate after the experiment is used as a supplementary indication of the nature of the reaction. The experiment is performed in duplicate.

This procedure is based on techniques developed at NOL (White Oak, Maryland), and at the U.S. Bureau of Mines (Bruceton) and at RVO-TNO (Riswijk, The Netherlands).

Task 5 - Card Gap Test

The Card Gap Test technique is based on procedures developed at NOL (White Oak, Maryland) and at a wide variety of military installations, aerospace contractor facilities and private organizations. The test fixture is essentially identical to that described in Task 4 above, except that the velocity instrumentation is optional, the witness plate is separated from the test material by a 0.0625 air gap, and polyethylene spacer "cards" 0.010 inch thick are inserted between the donor charge and the acceptor to attenuate the shock. The criterion for a positive result is the punching of a hole in the witness plate. Experiments are performed with varying numbers of spacer cards until the number of cards reducing probability of positive results to 50% is discovered. The higher the card gap value (number of cards for 50% probability), the more sensitive the explosive. This was not conducted for samples in which the results of Task 4 indicated no detonation.

Task 6 - Long-Term Thermal Stability Study

A 50 gram sample charge was placed in a glass cup in a SS bomb equipped for continuous pressure and temperature monitoring (280 cc net vol). The vessel was placed in an oil bath and brought to 100°C (or appropriate lower temperature), and the system monitored for a period of 48 hours for temperature and/or pressure excursions. The absolute values of temperature and/or pressure excursions are not highly reliable, as the primary purpose of the procedure is to discover the existence of such excursions

rather than the actual magnitude. However, data as to magnitude of excursions is of sufficient reliability to make an assessment of the degree of hazard posed by the reactions discovered.

For LGP 1845, NOS 365, and 13 M HAN, an additional trial was conducted at 75°C, due to their failure in the 100°C trials. The amount of NOS 365 used was decreased to 10 gm due to the violence of the reaction observed in the 100°C trial (50 gram sample).

Task 7 - Flash Point Determination

Flash points were examined in the Cleveland Open Cup tester. In this method, a sample of the test material is heated gradually in an open container. At specified temperature intervals a small test flame is passed across the opening. The lowest temperature at which the application of the test flame causes the vapors above the surface of the liquid to ignite is taken as the flash point.

The specific procedure for this experiment is designated ASTM 92-72.

Task 8 - Autoignition Temperature (Setchkin)

The object of this procedure is to determine the lowest temperature at which fuel vapors will spontaneously ignite in air. Experience shows that autoignition temperature is dependent on apparatus geometry and volume, and to some extent on sample charge volume. The most representative laboratory procedure generally is that of Setchkin (Nat. Bur. Stds.).

The experimental apparatus consists of a one liter spherical flask in a temperature-controlled bath or oven. A sample charge volume of 0.05 cc. is injected into the flask at a preselected temperature and the time-to-ignition is recorded. (Ignition is detected by the appearance of a flash in the flask.). The temperature is raised or lowered, as appropriate, and the procedure is repeated. Time is plotted as a function of temperature. The temperature at which the time becomes "infinite" is the tentative auto-ignition temperature.

Additional trials are conducted at the tentative auto-ignition temperature to determine whether different sample charging volumes will produce lower ignition temperatures.

If a different sample charging volume does produce ignition at the tentative autoignition temperature, the procedure is repeated until the true Setchkin autoignition temperature is determined to $\pm 5^{\circ}\text{C}$.

Task 9 - Deflagration Potential Determination

The object of this procedure is to determine whether a condensed phase sample can be ignited at a high-temperature site and can then sustain a propagating subsonic reaction to completion.

The sample (ranging from 10 to 50 grams) is placed in a glass cup inside a heavy-walled stainless steel vessel equipped with high-speed pressure and temperature recording devices. Two ignition sources are introduced into the sample chamber. The first is a tightly coiled nichrome fusing wire just below the sample surface; the second is an electrically activated pyrotechnic igniter (squib) directed at the sample surface.

In the initial experiment at ambient conditions, the fusing coil is activated first. If no reaction is observed, the squib is fired. If both attempts fail to ignite the sample, a second experiment is performed at 100°C under an applied nitrogen pressure of 250 psig (to simulate inertial effects in large samples). A minimum of two experiments is performed. Pressure-time records are obtained for all propagating reactions.

This procedure is similar to burning-rate studies (e.g.-Crawford Bomb Studies) except that applied pressures are much lower and linear regression rate measurements are not made.

Task 10 - Thermal Stability Scan

This procedure was used to further examine the response of the LGP candidate materials to rapid exposure to elevated temperatures, being essentially an amplification of Task 2 (JANAF Thermal Stability), but with lower confinement and glass enclosure to prevent catalysis by metal walls.

A 10 gram charge of sample was placed in an all-glass enclosure in the Thermal Stability Bomb (ref. Task 6). The enclosure was equipped with a glass thermocouple well in the side, so as to monitor sample temperature without metal contact. The glass cup containing the sample was covered with an inverted glass beaker to prevent contamination by refluxing from the instrumented bomb head. The vessel was placed in an oil bath which was heated so as to produce a sample temperature rise of 2°C/minute. Pressure and sample temperature were monitored continuously throughout the experiment.

This task was limited to LGP 1845 and NOS 365.

Task 11 - Bonfire Exposure (Small Lots)

This procedure was used to further evaluate the response of LGP candidates to rapid exposure to elevated temperatures. This procedure was significantly less rigorous than that of Task 2 (JANAF Thermal Stability) or Task 10 (Thermal Stability Scan), but was more directly comparable to the specified procedure of TB 700-2 for Unconfined Burning.

For each material examined, one 4 oz. glass bottle and one 4 oz. Naglene bottle, each equipped with a plastic screw cap, were charged with 140 gm. of sample. Each bottle was then placed on a grate 8" from the ground. Kerosene soaked wood 1" x 2" x 12" was stacked teepee-style around the support pedestal and ignited. The results were observed and recorded.

This task was limited to LGP 1845 and NOS 365.

Experimental Results

Task 1 - Trauzl Tests

The results of this test series are presented in Table 1. Briefly, they indicate that none of the samples tested sustain a detonation when initiated by a Number 8 blasting cap. The 11 M and 13 M HAN, the propellant mixtures and TEAN evidence substantial pressure development when so initiated, however.

Task 2 - JANAF Thermal Stability

The results of this test series are presented in Table 2. It can be seen that all samples, except TMAN and TEAN, exhibited considerable exothermic activity in the 100-200°C area; IPAN reacted strongly above 200°C; all samples except TMAN and 2.8 M HAN caused disc rupture. LGP 1845 exhibited the most severe behavior, in one trial exhibiting an exotherm at 135°C and bursting the disc, and in the other, apparently going to a detonation at 167°C. TMAN while showing no major exotherm, exhibited minor exothermic activity several times during the scan.

It appears that the oxidizer-fuel mixtures (LGP 1776, LGP 1845, and NOS 365), are less stable under thermal exposure than either the oxidizers or fuels alone.

Task 3 - Impact Tests

The results of this test series are presented in Table 3. All nine materials are relatively insensitive to impact, exhibiting high (>30 in) values for the 50% positive drop height.

Task 4 - Detonation Velocity

The results of this test series are presented in Table 4. The data indicates that the three oxidizers do not propagate a detonation while the three mixtures appear to sustain a "low velocity detonation" as evidenced by the plate damage and tube fragments.

Task 5 - Card Gap Tests

The results of this test series are presented in Table 5. Only the three propellants were tested since the results of task 4 were negative for the oxidizers. The results indicate that all three mixtures exhibit card gap values below 70 cards and that, of the three, LGP 1845 appears to be most sensitive to initiation.

Task 6 - Long-Term Thermal Stability

The results of this test series are presented in Table 6. Briefly, they indicate that 2.8 M HAN, 11 M HAN, TEAN, IPAN, TMAN, and LGP 1776 are capable of withstanding incubation at 100°C for 48 hours without undergoing a thermal explosion. Both 13 M HAN and LGP 1845 exhibit rapid exothermic decompositions, at 28.5 and 18.35 hours respectively;

LGP 1845 produces enough pressure to cause the rupture of a 2000 psi burst disc. NOS 365 exhibits a much more severe reaction, sustaining a detonation (or a reaction closely approximating a detonation) after 6.25 hours.

In additional trials at 75°C with LGP 1845 and 13 M HAN, no reaction was detected in 48 hours of incubation. A ten-gram quantity of NOS 365 reacted sharply in 9.5 hours, however, rupturing the 2000 psig safety disc.

In the light of information from other sources, it was decided to perform experiments at 75°C and 100°C with NOS 365, and at 100°C with LGP 1845, in which the sample was completely enclosed in glass in the bomb so as to prevent metal catalysis (see Task 10 Description). These three experiments all continued for at least 48 hours with no evidence of thermal explosion observed for any sample.

Task 7 - Cleveland Open Cup Flash Points

The results of this test series are presented in Table 7 (the Cleveland Open Cup method was substituted for the Tag Closed Cup, due to the nature of the materials examined). The data indicates that none of the nine samples has a flash point below 75°C.

Task 8 - Autoignition Temperatures

The results of this test series are presented in Table 8. Briefly, it can be seen that the three oxidizers would not autoignite up to 500°C, while the other six samples ignited at temperatures in the 205-410°C area.

Task 9 - Deflagration Potential Tests

The results of this test series are presented in Table 9. As indicated, none of the materials studied in this program deflagrated on exposure to fusing wire or squib initiators at ambient conditions. The fuels were also stable to these initiators at 100°C, 150 psig. Because of the nature of the oxidizers and propellants, a maximum temp. of 70°C was used in the second trial. Only 2.8 M HAN was stable to the initiators at these conditions; both 11 M HAN and 13 M HAN were stable to the wire, but ignited with the squib; NOS 365 and LGP 1776 ignited with the wire; and LGP 1845 spontaneously decomposed before either initiator was fired. This latter phenomenon may have been related to the exposure of the sample to metals of the initiators.

Task 10 - Thermal Stability Scans

The results of these tests are presented in Table 10. LGP 1845 commenced runaway exothermic decomposition at about 145°C; within about 5 minutes the temperature reached 175°C where catastrophic decomposition occurred. Between 157° and 175°C, pressure rose to about 80 psig; at 175°C the trace disappeared as pressure rose sharply to over 2000 psig. NOS 365 appeared to self-heat at about 135°C; over about 13 minutes the temperature attained about 147°C, at which point pressure rose sharply (trace disappeared) from 0 to over 2000 psig.

Task 11 - Bonfire Exposures

The results of these tests were all essentially identical. With both LGP 1845 and NOS 365 the Nalgene containers melted and the contents fell into the fire. In neither case was any significant contribution to the fire observed. In the glass bottle, LGP 1845 expelled white vapors through the cap after about 15 minutes; the gases burned briefly. Container remained in place until fire went out. In the case of NOS 365, the plastic cap caught fire; subsequently the glass broke in place on the grate, dumping the contents. In neither case was any explosion or flare burning noted.

Discussion

The following indicates the relationship the procedures used in this program bear to the protocol of TB 700-2, based on the nature of the information generated. It is appropriate to note that both protocols are meant to classify the materials per se, as opposed to containers or devices charged with the materials. Depending on the nature of packaging contemplated, additional experiments with packaged samples may be desirable or necessary.

<u>TB 700-2 Designation</u>	<u>Proposed Procedures</u>
3-8. Detonation Test	Trauzl Block Test
3-9. Ignition and Unconfined Burning Test	Flash Point Test Autoignition (Setchkin) Deflagration Potential JANAF Thermal Stability Test Thermal Stability Scan Bonfire Exposure
3-10. Thermal Stability Test	Long-Term Thermal Stability
3-11. Impact Sensitivity Test	Impact Sensitivity Test
3-12. Card Gap Test	Detonation Velocity Det. Card Gap Test

For convenience in analysis of the results of this program, the data is summarized in Table 11. In Table 12, the significance of the data in terms of classification criteria of TB 700-2 is indicated.

For purposes of concordance between the protocol used and TB 700-2, the following principles were observed:

1) The detonation (lead cylinder) test of TB 700-2 is totally unconfined. The Trauzl Block provides some significant confinement to product gases, and materials that do not detonate do yield positive expansion values in Trauzl Block tests when decomposition can produce significant product gas. Accordingly, materials yielding expansion values below 8 cc/gm in the Trauzl Block are considered "negative" relative to TB 700-2 Detonation Test.

2) The ignition and unconfined burning test of TB 700-2 evaluates both sensitivity to ignition and violence of burning. Materials exhibiting flash points of over 75°C and autoignition temperatures of over 100°C are considered unlikely to ignite under conditions incident to transportation and storage. Materials that do not deflagrate readily are also considered unlikely to ignite under such conditions. The JANAF Thermal Stability Test provides significant confinement, but reflects the stability of materials under relatively brief high heat exposure, and, in this context, is used as a measure of the response of a confined material (as a liquid in transportation must be) to relatively brief fire exposure. Materials that do not detonate in the JANAF Thermal Stability Test are considered to be unlikely to detonate under brief fire exposure. Materials that react strongly in the JANAF Thermal Stability Test are considered to require further evaluation by Thermal Stability Scan and Bonfire Exposure. The Thermal Stability Scan reduces the confinement of the JANAF Thermal Stability Test and eliminates any possible metal catalysis. Liquids that react sharply and/or massively in the Thermal Stability Scan are considered to pose serious packaging problems in transportation, but the degree of confinement is still substantial, and recourse is had to the Bonfire Exposure. In the Bonfire Exposure, the liquids are examined under conditions of minimum practical confinement for transportation. Materials that react violently in the Bonfire Exposure are considered to fail the Ignition and Unconfined Burning Test for purposes of TB 700-2 classification.

3) The 48 hour thermal stability tests are considered to be essentially equivalent in both protocols. A preliminary experiment is performed at 100°C (to obtain a measure of margin of safety) in the protocol used in this program. Failure to survive the 48 hour exposure at 100°C is not considered definitive for this test; rather, a second experiment at 75°C is performed. Failure to survive 48 hours at 75°C is considered failure to meet the basic criterion of TB 700-2 Thermal Stability Test, and would result in a recommendation of, "Forbidden".

4) The distinguishing criterion for DOT Restricted classification according to TB 700-2 is 4" drop height with an 8 pound weight. The impact apparatus used in this program has a 4.4 pound weight. Materials exhibiting impact values of greater than 10 inches are considered to meet the maximum sensitivity criteria of TB 700-2 Impact Test, and are considered to be unrestricted on the basis of this criterion.

5) Card Gap Tests are required by TB 700-2 when a detonation is obtained in any of the TB 700-2 tests previously mentioned. For the purpose of increasing the rigor of the protocol actually used, Detonation Velocity Tests were performed on all oxidizers and propellant mixtures in the present protocol. The key criterion for distinguishing between Class 2 and Class 7 in TB 700-2 is the value of 70 cards. Accordingly, materials exhibiting card gap values below 70 cards by the plate puncture criterion of TB 700-2 are considered Class 2 for the purpose of Card Gap Test results.

Results of Detonation Velocity and Card Gap Tests merit special discussion. The Detonation Velocity Tests on the oxidizer solutions (ref. Table 4) clearly indicate no detonation by the plate puncture criterion; the velocity reading for 13 M HAN is rather high, but the physical evidence still indicates no detonation.

Results of the Detonation Velocity Tests for all three propellants include substantial velocities, significant fragmentation of all the tubes and severe bowing or breaking of the witness plates. Although the plates were not "holed" the violence of the response relative to that for the oxidizers is considered significantly greater and adequate to warrant the "Low Velocity Detonation" designation. Accordingly, it was decided by mutual agreement of ARRADCOM and HRC personnel that Card Gap trials were warranted on the propellants. The prime object of the Card Gap trials was chosen to be LGP 1845, which displayed the most severe response in Detonation Velocity trials.

The initial trials in the Card Gap series, with LGP 1776 and NOS 365 at 8 cards, both produced less physical damage than in the Detonation Velocity trials. As these had been marginal in the Detonation Velocity trials (0 cards), no further work was considered appropriate with these propellants.

Card Gap trials with LGP 1845 at 8, 24 and 50 cards produced significantly greater damage than experienced in the Detonation Velocity trials (0 cards). At 70 cards, the damage appeared to abate slightly, although the plate was still broken.

At the request of ARRADCOM representatives, two trials with LGP 1845 diluted with 5% (b.w.) water were performed, with ambiguous results (one clearly negative, one apparently positive).

At this point, it was decided by mutual agreement of ARRADCOM and HRC personnel to perform two trials with 6 inch square witness plates (as specified in TB 700-2) at 70 cards, using as-received LGP 1845. The objective was to meet the specific TB 700-2 criterion for mechanical witness as precisely as possible. Both trials resulted in negative indications, and the program was terminated.

The propellants obviously have significant energy that can be liberated under conditions of high incident hydrodynamic shock. All of the propellants, however, will meet the criterion of TB 700-2 for Military Class 2 as far as Card Gap value is concerned (below 70 cards).

Classification was then recommended on the following basis (ref. Table 11 for summary of data application):

TMAN	No classification under TB 700-2.
TEAN	No classification under TB 700-2.
IPAN	No classification under TB 700-2.
2.8 M HAN	No classification under TB 700-2. Oxidizer appears appropriate for DOT purposes.
11 M HAN	No classification under TB 700-2. Oxidizer appears appropriate for DOT purposes.
13 M HAN	No classification under TB 700-2. Oxidizer appears appropriate for DOT purposes

- LGP 1776 Military Class 2 (DOT Explosives Class B), as this material reacts quite sharply at about 145°C under brief exposure and can propagate a detonation or near-detonation under shock.
- LGP 1845 Military Class 2 (DOT Explosives Class B), as this material can react violently under brief exposure in the 135°-167°C region under strong confinement and can propagate a detonation or near-detonation under shock.
- NOS 365 Military Class 2 (DOT Explosives Class B) as this material can react very sharply after very brief exposure to 145°C under strong confinement and can propagate a detonation or near-detonation under shock.

NOTE: The recommended classifications for the LGP 1845 and NOS 365 are for lowest possible confinement, where fire exposure will definitely result in very rapid failure of confining vessel and venting of products. Any shipping container conformation should be subjected to fire exposure tests before use, as degree of confinement may well alter the recommended classification.

CONCLUSIONS

No classification appears warranted for the fuels or oxidizers under TB 700-2 criteria. The DOT "oxidizing material" classification appears appropriate for the oxidizers.

Specification of appropriate DOT Classification for fuels is not possible on the basis of this program, but may be performed by persons aware of the compositions of these materials.

LGP 1776 appears properly classified as Military Class 2.

LGP 1845 appears properly classified as Military Class 2 when packaged under minimum possible confinement designed to vent products quickly and easily under fire exposure.

NOS 365 appears properly classified as Military Class 2 when packaged under minimum possible confinement designed to vent products quickly and easily under fire exposure.

NOTE: Packaging of LGP 1845 or NOS 365 in containers permitting contact of the contents with metals should be prohibited. See also note at end of preceding section of this report.

Table 1. Trauzl test results

Test No.	Sample	V (cc/g)		Overall
		1 gm loading	2 gm loading	
1	2.8 M HAN	1.0	1.0	1.0
2	11 M HAN	4.4	3.0	3.7
3	13 M HAN	5.0	4.0	4.5
4	LGP 1776	6.5	3.0	4.7
5	LGP 1845	6.0	4.2	5.1
6	NOS 365	4.0	3.2	3.6
7	TEAN	3.0	1.5	2.7
8	IPAN	0.5	1.0	0.7
9	TMAN	0.6	1.2	0.9
10	Special ^a	-	0	0
11	Special ^b	-	0	0

^aMixture consisting of 1 gm NOS 365 and 1 gm Dow Corning High Vacuum Grease (Silicone Lubricant), tested 30 minutes after mixing.

^bMixture consisting of 1 gm NOS 365 and 1 gm Dow Corning High Vacuum Grease (Silicone Lubricant), tested 24 hours after mixing.

Table 2. JANAF thermal stability test results

Test No.	Sample	Temperature of major exotherm onset (°C)	Remarks
1	2.8 M HAN	202	Sharp and rapid exotherm
2	11 M HAN	165	Sharp and rapid exotherm, burst disc
3	13 M HAN	148	Sharp and rapid exotherm, burst disc
4	LGP 1776	145	Very sharp and very rapid exotherm, burst disc
5	LGP 1845	135*	Very sharp and very rapid exotherm, burst disc
6	NOS 365	105	Very sharp and very rapid exotherm, burst disc
7	TMAN	-	No major exotherm observed, showed sporadic, weak exotherms
8	TEAN	195	Gradual, smooth exotherm, burst disc
9	IPAN	185, 220	Two sharp exotherms observed, burst disc

* In the replicate trial, LGP 1845 remained stable to 167°C, then underwent an extremely rapid and energetic reaction, resembling a detonation.

Table 3. Impact test results

Test No.	Sample	Drop Heights, (in.)		
		<u>0% Positive</u>	<u>50% Positive</u>	<u>100% Positive</u>
1	2.8 M HAN	34	35	36
2	11 M HAN	31	33	35
3	13 M HAN	32	33	34
4	LGP 1776	31	32	34
5	LGP 1845	28	30	31
6	NOS 365	29	30	32
7	TMAN	48	> 48	> 48
8	TEAN	48	> 48	> 48
9	IPAN	48	> 48	> 48

Table 4. Detonation velocity test results

<u>Material</u>	<u>Run</u>	<u>D.V.</u>	<u>Remarks</u>	<u>Conclusion</u> [*]
2.8 M HAN	1	1830 m/s	Broad strips recovered. Plate OK	No
	2	1870	Broad strips recovered. Plate OK	No
11 M HAN	1	2210	Strips recovered. Plate OK	No
	2	2150	Strips recovered. Plate OK	No
13 M HAN	1	2700	Strips recovered. Plate OK	No
	2	(Lost)	Strips recovered. Plate OK	No
LGP 1776	1	2350	Moderate fragmentation. Plate broken	LVD
	2	2490	Moderate fragmentation. Plate bowed	LVD
LGP 1845	1	2560	High fragmentation. Plate broken	LVD
	2	2350	High fragmentation. Plate broken	LVD
NOS 365	1	2630	Moderate fragmentation. Plate bowed	LVD
	2	3050	Moderate fragmentation. Plate bowed	LVD

*The conclusion, "No", represents a judgement of the result based on the plate damage criterion corresponding to TB700-2. "LVD" indicates a "Low Velocity Detonation".

Results indicate that the HAN materials do not sustain a detonation for purposes of hazard classification, at the 0 card level. The LGP materials produced significant fragmentation and plate breakage; for the present purpose, these were considered as marginally positive at the 0 card level.

Table 5. Card gap test results

<u>Test No.</u>	<u>Sample</u>	<u>No. Cards</u>	<u>D.V. (m/sec)</u>	<u>Remarks</u>
1	NOS 365	8	2490	Mod. fragmentation, plate bowed
2	LGP 1776	8	2420	Mod. fragmentation, plate bowed
3	LGP 1845	8	2700	High fragmentation, plate shattered
4	LGP 1845	24	(Lost)	High fragmentation, plate shattered
5	LGP 1845	50	2491	High fragmentation, plate shattered
6	LGP 1845	70	2837	Mod. fragmentation, plate broken
7	LGP 1845 (5% H ₂ O)	70	2353	Strips recovered, plate OK
8	LGP 1845 (5% H ₂ O)	70	(Lost)	High fragmentation, plate shattered
9	LGP 1845	70	2422	Mod. fragmentation, plate bowed*
10	LGP 1845	70	2630	Mod. fragmentation, plate bowed*

*The test was conducted using standard 6" x 6" witness plate as mandated by TB700-2, all other trials used 4" x 4" witness plate as originally proposed.

Table 6. Long-term thermal stability test results

Test No.	Sample	Test temp (°C)	Mass of sample (gm)	Observations
1	2.8 M HAN	100	50	No reaction in 48 hours
2	11 M HAN	100	50	No reaction in 48 hours
3	13 M HAN	100	50	Rapid self-htg. began 28.5 hrs. Max. Press. = 1950
4	LGP 1776	100	50	No reaction in 48 hours
5	NOS 365	100	50	Severe reaction (possibly detonation) at 6.5 hours bolts on bomb burst, major damage to facility
6	LGP 1845	100	50	Sudden decomp. 18.35 hrs.; burst 2000 psig disc.
7	IPAN	100	50	No reaction in 48 hours
8	TEAN	100	50	No reaction in 48 hours
9	TMAN	100	50	No reaction in 48 hours
10	LGP 1845	75	50	No reaction in 48 hours
11	NOS 365	75	10	Sudden decomp. 9.5 hrs.; burst 2000 psig disc.
12	13 M HAN	75	50	No reaction in 48 hours.
13	NOS 365	75	10	All-glass; no reaction in 48 hours.
14	NOS 365	100	10	All-glass; no reaction in 48 hours.
15	LGP 1845	100	10	All-glass; no reaction in 48 hours.

Table 7. Cleveland open-cup flash point determination results

Test No.	Sample	Results
1	2.8 M HAN	No flash to 87°C (when boiling began)
2	11 M HAN	No flash to 87°C (when boiling began)
3	13 M HAN	No flash to 87°C (when boiling began)
4	IPAN	No flash to 100°C (sample was a liquid at this temperature)
5	TEAN	No flash to 100°C (Sample was a liquid at this temperature)
6	TMAN	No flash to 100°C (Sample remained solid at this temperature)
7	LGP 1776	No flash to 75°C*
8	LGP 1845	No flash to 75°C*
9	NOS 365	No flash to 75°C*

* Due to the reactive nature of these materials, it was deemed inadvisable to increase the test temperature beyond 75°C.

Table 8. Results of AIT determination tests

<u>Test No.</u>	<u>Sample</u>	<u>Autoignition Temperature (°C)</u>
1	2.8 M HAN	>500 (decomposed with white smoke)
2	11 M HAN	>500 (decomposed with white smoke)
3	13 M HAN	>500 (decomposed with white smoke)
4	NOS 365	285
5	LGP 1776	272
6	LGP 1845	310
7	IPAN	255
8	TMAN	205
9	TEAN	410

Table 9. Results of deflagration potential tests

Sample	Amount (gm)	Normal conditions ^a		Special conditions ^b	
		wire	squib	wire	squib
2.8 M HAN	10	no ignition	no ignition	no ignition	no ignition
11 M HAN	10	no ignition	no ignition	no ignition	ignited
13 M HAN	10	no ignition	no ignition	no ignition	ignited
LGP 1776	10	no ignition	no ignition	ignited	-
LGP 1845	10	no ignition	no ignition	see footnote c	
NOS 365	10	no ignition	no ignition	ignited	-
IPAN	10	no ignition	no ignition	no ignition	no ignition
TMAN	10	no ignition	no ignition	no ignition	no ignition
TEAN	10	no ignition	no ignition	no ignition	no ignition

^a Ambient temperature, atmospheric pressure

^b Temp = 100°C (for TMAN, TEAN, IPAN), 70°C (all other samples), 250 psig pressure (all samples)

^c Sample spontaneously ignited almost immediately after pressurization

Table 10. Thermal stability scan test results

Normalized ^a Time	Temperature	
	LGP 1845	NOS 365
0 min.	20°C	20°C
10	28	32
20	40	45
30	52	62
40	65	75
50	80	90
60	95	102
70	106	113
80	117	123
90	127	132
110	143	138 ^c
120	158 ^b	

^a Normalized at 20°C departure point = 0 minutes.
Both samples 10 gm in nominal 280 cc SS vessel;
sample in all-glass enclosure.

^b At 119.5 min., P = 0 psig; at 120 min, P = 80 psig;
at 120.5 min., P > 2000 psig.

^c At 109.7 min., T = 147°C, P = 0 psig; at 109.8
min., T > 200°C, P > 2000 psig.

Table 11. Summary of test data

Test	TMAN	TEAN	IPAN	2.8 M HAN	11 M HAN	13 M HAN	LGP 1776	LGP 2845	NOS 365
Trauzl (cc/g)	0.9	2.7	0.7	1.0	3.7	4.5	4.7	5.1	3.6
Flash Point	None	None	None	None	None	None	None	None	None
A.I.T. (°C)	205	410	255	> 500	> 500	> 500	272	310	285
Deflagration Amb. T.P. Elev. T.P.	No No	No No	No No	No No	No squib	No squib	No Wire	No Auto	No Wire
JANAF Th. St. (°C)	No	195	185	202 sharp	165 sharp	148 sharp	145 v. sharp	135 v. sharp	105 v. sharp
Th. St. Scan (°C)	X	X	X	X	X	X	X	170 v. sharp	147 v. sharp
Bonfire Exp.	X	X	X	X	X	X	X	No Exp.	No Exp.
Long-term ^a Th. St. 100°C 75°C	48 X	48 X	48 X	48 X	48 X	28.5 ^b X	48 X	48 ^c 48	48 ^c 48 ^c
Impact (in)	> 48	> 48	> 48	35	33	33	32	30	30
Detonation	X	X	X	No	No	No	LVD	LVD	LVD
Card Gap	X	X	X	X	X	X	<70	<70	<70

X = test considered unnecessary

^a Parameter is survival time in hours.

^b Open glass liner; material may have received metal contaminants by reflux action.

^c Closed glass liners; in open glass liners, LGP reacted violently in 18.35 hours., NOS 365 in 6.5 hours at 100°C, 9.5 hours at 75°C; materials may have reacted under metal catalysis.

Table 12. Application of data for classification in terms of TB 700-2

Test	TMAN	TEAN	IPAN	2.8 M HAN	11 M HAN	13 M HAN	LGP 1776	LGP 1845	NOS 365
Trauzl Block	U	U	U	U	U	U	U	U	U
Flash Point	U	U	U	U	U	U	U	U	U
A.I.T.	U	U	U	U	U	U	U	U	U
Deflagration	U	U	U	U	U	U	U	U	U
JANAF Th.St.	U	U	U	U	U	U	2	7 ^a	7 ^a
Th.Stab.Scan	X	X	X	X	X	X	X	7 ^b	7 ^b
Bonfire Exp.	X	X	X	X	X	X	X	U ^c	U ^c
Long-term Th.St.	U	U	U	U	U	U	U	U	U
Impact	U	U	U	U	U	U	U	U	U
Detonation	X	X	X	U	U	U	2	2	2
Card Gap	X	X	X	X	X	X	2	2	2

X Test considered unnecessary
U Unrestricted

2 Military Class 2, DOT Explosives B
7 Military Class 7, DOT Explosives A

^aHigh confinement in stainless steel.

^bGlass container, product gases not vented due to SS external container.

^cGlass or plastic container designed to vent product gases at very low pressures.

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