

24th EXPLOSIVE SAFETY BOARD SEMINAR  
HAZARD CLASSIFICATION OF LIQUID PROPELLANTS

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INTRODUCTION

The lack of a formal protocol in the DoD safety community to evaluate liquid propellants prompted the establishment of a Hazard Classification of liquid propellants program. The DoD safety manual TB-700-2 has been used exclusively for the hazard classification of solid propellants and explosives in storage and transportation. A recent revision of this manual now addresses energetic liquids on a case by case basis. An examination of the UN document and NATO AOP-7 revealed deficiencies in making a final assesment for a Hazard Classification of energetic liquids. The Un document only addressed explosives in transportation; while the NATO AOP-7 provides no criteria for making a final judgement to a Hazard Classification. The tests, procedures, and criteria for making a final classification can be found in the TB-700-2. For this reason, the TB 700-2 was selected as the role model for developing the Hazard Classification of energetic liquids.

Since the tests and test procedures found in the TB-700-2 were designed to evaluate solid materials, it became apparent that modifications to these tests and test procedures would have to be made. The liquid propellants LP 1845 and LP 1846 are extremely sensitive to transition metals and nitric acid. Thus, containment of these liquids for a test evaluation would have to be made in a container compatible with these liquid.

This paper will address the events leading up to the selection of the TB 700-2 as the role model for the Hazard Classification of liquid propellants, establishment of the interim- classification tests, test procedures, modifications to the test procedures, and criteria for classifying liquid propellants. The recommended tests designed for a final classification will be contingent upon evaluating the liquid propellants in approved DoD packaging.

# Report Documentation Page

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## DISCUSSION

The UN document was initially examined for potential tests, test procedures, and criteria that might be beneficial in the establishment of a Hazard Classification of the liquid propellants. This initial examination was made based upon the Department of Transportation's acceptance of this document as their guideline for transporting explosives. As illustrated in Figures 1 and 2, the document determines the division of a Hazard Classification from a series of questions and answers. As seen in Figures 1 and 2, a material is considered for a Class I the answer is yes to the question "is the substance manufactured with the view of producing a practical explosion" Test series 1 is performed if the answer is no. Following this test series, if the answer is yes to the next question "is it an explosive substance?", then test series 2 is performed. Should the substance be considered as a Class 1 material, then test series 3 is conducted. When the results of test series 3 demonstrate that the material is thermally stable, test series 4 is conducted. The material is provisionally accepted into a Class 1 if packaged substance is considered not too hazardous for transportation.

The question now asked is "can the substance be considered for a Division 1.5?" If the answer is yes, then the test series 5 is performed. Should the results of this test indicate that the substance is an insensitive substance, then it is considered a Division 1.5 material. However, if the answer to the question above is no, then the packaged substance is subject to test series 6. Depending upon the series of questions asked depicted in Figure 2 and the answers given based upon test results will determine how the material will be characterized. Test series 1 through 6 can be found in Figures 3-8.

The NATO AOP-7 outlines the characteristic qualifications for liquid propellants in Figure 9. Note that in Figure 10, the qualification guidelines follow those of the TB 700-2. Specific physical property tests call for density, melting point, boiling point, coefficient of thermal expansion, vapor pressure, and flammability/detonability. The required NATO AOP-7 qualification tests are given in Figure 11. Optional qualification tests are cited in Figure 11a. Unfortunately, these tests, spelled out for the NATO AOP-7, provide no criteria for assessing or designating a Hazard Classification.

In the TB 700-2, mandatory tests, shown in Figure 12, are required for an interim hazard classification for solid propellants and explosives. Alternate tests that may be required, depending on the application, are depicted in Figure 13. Criteria to establish the interim classification for a Class A or B explosive material are given in Figure 14. No criteria are given for an interim classification for solids as a Class C, explosive. To obtain a final classification, the single package, stack test, and external fire tests are required. As

shown in Figure 15, additional tests, such as the bullet impact, fragment, package drop, and oblique drop tests, may be required before a final Hazard Classification can be rendered. The decision to conduct these tests will be determined by the appropriate safety group. The bullet impact and fragment tests are part of the requirements for an insensitive munition.

The criteria for arriving at a final Hazard Classification for a material are shown in Figure 16. Note that criteria for a Class C material are now given. Include in this criteria should have been that the material does not detonate under atmospheric conditions nor are fragments produced. Schemes for a large scale test analyses are cited in Figure 17 and 18. The single package, stack and external fire tests are the only required mandatory tests for a final Hazard Classification of a material.

Before initiating a test program, a methodology was presented to the safety community for approval. As depicted in Figures 19, 20, and 21, this methodology received the approval of the tri-services and DoD Explosive Safety Board. Note that for liquids, a Class C designation has been recommended. This differs from the TB 700-2 in that only a Class A or B is given as a result of the card gap test. When 70 or more cards are required to attenuate a detonation in the card gap, a Class A explosive is assigned to the material. If the test results are 70 cards or 0, the material is a Class B explosive. It is recommended that the material be considered a Class C material when 0 cards are used in the card gap test.

Utilizing the tests cited in the TB 700-2 manual, modifications had to be made to evaluate a liquid. As shown in Figures 22, 23, and 24, the liquid propellants were housed in compatible polyethylene sample test containers for testing. This precaution had to be taken since the liquid propellants LP 1845 and LP 1846 are decomposed by transition metals. Contact of these materials with compatible stainless steel containment had to be assured in the impact and critical diameter tests.

All the mandatory and auxiliary tests were conducted for LP 1845 and LP 1846. The results of these tests are given in Tables 1-10. The test description of each test is presented below.

## TEST DESCRIPTION

### THERMAL

Decontaminate a two(2) inch diameter x 2-1/2 inch high x 0.5 mil thick polyethylene bottle filled with deionized water by placing in an oven at 70C for 24 hours. Weigh the dried bottle. Fill the decontaminated bottle with liquid propellant, weigh the bottle and liquid propellant and place in a constant temperature, explosion proof oven. Raise the temperature to 75C and maintain this temperature for a period of 48 hours. Remove and weigh the bottle and liquid propellant. Record the weight change and any reactions that may have occurred over this period of exposure.

## CARD GAP

A typical card gap tester is shown in Figure 25. The test apparatus consists of a one piece 1.875 inch O.D. x 5.5 inch long mild steel tube. The ignition source consists of two pentolite pellets that weigh approximately 60 grams and a J-2 blasting cap. A 6 inch x 6 inch x 0.375 inch mild steel witness plate is used to determine if a detonation has occurred. A detonation is indicated when a clean hole is cut into the witness plate. Cellulose acetate (or equivalent) cards 2 inches x 0.01 inches thick are used to attenuate a detonation. The greater the number of cards, the more sensitive the material. Four small pieces of plastic material cut into (0.0625 x 0.5 x 0.5 inches) pieces are used as shims to support the tube and maintain a 0.0625 inch air gap between the test sample and the witness plate.

A 0.5 mil thick polyethylene sleeve is placed inside the mild steel tube to prevent contamination of the liquid propellant. The liquid propellant is placed inside the tube and the first test is conducted. In the first test, the cellulose cards are omitted. Should no detonation occur in the first test, the test is repeated two more times. If a detonation occurs, then a test series given in Figure 26 will be followed with the cellulose cards placed between the tube containing the liquid propellant and the pentolite booster.

The first test performed in Figure 26 is with 8 cards. If a detonation occurs, then the number of cards is doubled (add 8 cards) for the next test. If a detonation occurs in this test, the number of cards is doubled again (add 16 cards). Continue doubling the number of cards until no detonation occurs. When no detonation occurs, then the number of cards is reduced by one half the preceding increment. As an example, if the test is run at 32 cards but not at 64, then the next test will be run at 48 cards. If a detonation occurs at this reduced number of cards, the number of cards will be increased by one half the preceding increment of 56 cards. This test procedure is continued to a point where no detonation is obtained. A 50% probability that a detonation has occurred is a measure of charge sensitivity at where a 50% probability that a reaction has occurred at a given attenuation gap length.

## IMPACT

The standard JANNAF drop weight tester was used to establish the impact sensitivity of liquid propellants. A typical test sample holder for the apparatus is illustrated in Figure 27. The liquid propellant test sample is enclosed in a cavity formed by a steel cup, elastomeric "O" ring and a steel diaphragm. A 4.4 pound (2 kilogram) weight is dropped onto a piston from a height of 48 inches. If a positive result occurs, the weight is dropped from a height one half the original height. This adjustment of the

drop weight to one half the distance is continued until no positive reaction occurs. A positive result has occurred when the steel diaphragm is punctured with an accompanied loud report, severe deformation of the diaphragm or evidence that the sample is consumed. Data are reported at the height which yields 50% probability of initiation.

#### DETONATION

A lead cylinder, 1-1/2 inch diameter x 4 inches high, is placed upon a 12 inch square x 1/2 inch thick SAE 1010 mild steel plate. Fill a decontaminated polyethylene bottle (2.5 inches high x 2 inches in diameter) with a liquid propellant sample. A no. 8 blasting cap with the following requirements is placed perpendicular and in contact with the liquid surface:

1. A cap containing 0.4-0.45 grams of PETN base charge pressed into an aluminum shell having a bottom thickness not to exceed 0.03 inches.
2. A specific gravity not less than 1.4 grams/cubic centimeter.
3. Primed with standard weight of primer in accordance with the manufacturer's specifications.

In the center of a wooden block, drill a hole and position the blasting cap. Ignite the blasting cap and examine the lead cylinder for and deformation. Any deformation of the lead block that is 1/8 inch or more will be considered evidence that a detonation has occurred. Conduct a minimum of five tests more or until a detonation has occurred.

#### IGNITION AND UNCONFINED BURNING

Kerosene soaked sawdust is placed in a 12 inch x 12 inch x 4 inch stainless steel container with a 1/16 inch wall thickness. The sawdust is evenly filled to a level of 1/4 inch. A 2 inch diameter decontaminated polyethylene bottle is filled with a 2-1/2 inch height of liquid propellant. The polyethylene filled with test sample is placed in the center of the kerosene soaked sawdust and ignited with an electric match-head igniter. This test is repeated twice.

Four liquid propellant filled decontaminated polyethylene bottles are placed in the center of a container filled with kerosene soaked sawdust. The bottles are placed in a row with each bottle in contact with the next bottle. The sawdust is ignited at one end with an electric match-head igniter. The test is repeated two more times.

#### ADIABATIC COMPRESSION

The schematic of the U-tube compression ignition test set-up is illustrated in Figure 28. The following test parameters are

required:

U-tube radius	1.0 inch
Sample volume	3.0 cubic centimeters
U-tube height	9.0 inches
Ullage space	6.0 inches
Tubing material	304 stainless steel
Tubing diameter	0.25 inch O.D. x 0.035 thick
Pressure valve orifice	0.187 inches

The U-tube is closed at one end with a cap. A 3 ml. quantity of test fluid is placed in the curvature of the U-tube. The open end of the U-tube is connected to the discharge valve. A reservoir is pre-pressurized with nitrogen to 2000 psi. The test is conducted by a suddenly pressurizing the U-tube. The pressure surge forces the liquid in the curvature to violently compress the ullage space containing the liquid vapors into the closed end. This rapid rate of pressurization is sufficient to provide adiabatic compression. Rupture of the U-tube is an indication that an explosion or detonation has occurred. The test is repeated with nitromethane as a control and the test results are compared.

#### CRITICAL DIAMETER

A schematic of the critical diameter test setup is shown in Figure 29. Tests are conducted using different size diameter cylinders with L/D ratios of 2:1. The liquid propellant tests were conducted using 2,3,4 and 5 inch diameter cylinders. Each cylinder was welded to a 316 stainless steel 1/4 inch thick witness plate. This assembly was passivated with nitric acid to remove any potential contamination. An explosive C-4 charge is placed on top of the open end of the container filled with the liquid. A 0.5 ml thick polyethylene sheet separates the charge and liquid. As the size of the container increases from 2 to 4 inches, the explosive charge was increased proportionally to ensure that the same energy per unit area was maintained. The 2 inch tests were conducted with 160 grams of C-4, the 3 inch tests used 360 grams, the 4 inch tests used 640 and the five inch tests required 1000 grams for each test. These C-4 charge weights were chosen to produce a fixed energy input of  $3.11 \times 10^5$  Joules/sq in. Detonation velocity probes were inserted into the cylinders to determine the shock wave velocity as it travels through the liquid. The explosive charge is ignited and any reaction is recorded on motion picture film.

#### FLASH POINT

The standard procedure for the ASTM-92-72 Cleveland Open Cup Flash Point Test Method was followed to evaluate the liquid propellants. The standard brass cup used in this procedure was replaced by a Pyrex cup that reduced the standard volume from

70 ml. to approximately 50 ml. The cup was filled with liquid propellant and placed in the test apparatus. The fluid filled cup was heated to 25C. A gas-fired flame was passed over the liquid. If no flash occurred, the temperature was raised in increments of 10C to a maximum of 75C. The lowest temperature where the pilot flame caused the vapors above the liquid to ignite was taken as the flash point.

#### MINIMUM PRESSURE FOR VAPOR PHASE IGNITION

A schematic for the pressure test vessel for vapor phase ignition is depicted in Figure 30. Water is used to calibrate the vessel. The vessel is evacuated and 2 ml. of distilled water is injected into the vessel. The temperature is slowly raised at a rate of 5C per minute. At the 5C intervals, pressure and temperature are recorded. A sample of n-propyl nitrate is used as a standard for comparison. A fuse wire, 3 inches in length, is installed in the vessel such that 1 inch is unsupported. The vessel is evacuated and 2 ml. of n-propyl nitrate is injected into the vessel. The temperature of the liquid is raised to a temperature of 160C (use caution). Reduce the pressure to 2.2 atmospheres (29 psia). Ignite the vapor with the fuse wire and record the voltage, current, and pressure. Repeat the test two more times.

The apparatus is cleaned with distilled water and 3 ml test sample of liquid propellant is injected into the vessel. The temperature is slowly raised at 5C per minute until 100C is reached and then an attempt at ignition is made. Record voltage, pressure and current at this temperature. If no ignition occurs, discontinue the test restart test with procedure cited above except raise the upper limit of the temperature 5 C and attempt ignition. If no ignition occurs, repeat the test until ignition or a reaction occurs. At each of these ignition points, record the voltage, current and pressure.

#### ELECTROSTATICS

Electrostatic energy stored in a charge capacitor is discharged to the sample material to determine whether the electrostatic discharge will cause a sample to decompose, flash, burn, etc. The liquid sample is placed in a stainless steel 316 sample holder or equivalent material compatible with the liquid propellant that will permit the discharge to pass through the sample. The capacitor has a 50000 volt potential. A discharge needle is lowered above the liquid until a spark is drawn through the liquid sample (20 mg.). The standard test interval ranges from 0.0001 microfarads (uf) and 0.00125 Joules at 5 kV to 1 uf and 12.5J at 5 kV. The test is initiated at 1 uf and 12.5 J level. If a negative result occurs, testing is at this level until 20 negatives are reported. If the result is positive, such as a spark, flash, burn, odor or noise other than the instrument noise, a lower discharge level is selected until



20 or more energetic results occur. The test voltage of 5 kV or less at ambient temperatures between 18.3C and 32.2C is used with a relative humidity not to exceed 40%.

## DISCUSSION OF RESULTS

### THERMAL

The thermal test (Table 1) is the test used by the Department of Transportation as a basis for "forbidden" materials for transportation. Had a detonation occurred with the LP materials, the materials could not be shipped. Both materials indicated minimal loss in weight with no visible signs of a reaction having taken place.

### CARD GAP

The card gap test results found in Table 2 indicated that zero (0) cards were used. This reflects the insensitivity of the liquid propellants, LP 1845 and LP 1846. It is felt that the length of the sample holder tube, 5.5 inches, may not provide enough residence time for the shock wave to cause a reaction. It is suggested that the tube length be increased to 16 inches. Another concern is the diameter of this sample tube. The diameter can be below the critical diameter and, as such, will not support the transmission of detonation wave.

### IMPACT

Table 3 lists the impact test data for LP 1845 and LP 1846. Since LP 1846 has approximately 3% more water than LP 1845, the difference of only 1 inch between the two was to be expected, and LP 1845 more sensitive. Nitromethane, classified by the Department of Transportation as a flammable liquid is more sensitive than the liquid propellants.

### DETONATION

Detonation test data are given in Table 4. As seen, no detonations occurred in LP 1845 or LP 1846. The Department of Defense Safety community considers this test as a mandatory critical test. A detonation by a material under this test requirement would immediately characterize the material as a Class A Division 1.1.

### IGNITION AND UNCONFINED BURNING

The test results for ignition and unconfined burning are shown in Table 5. The transfer of thermal heat from sawdust soaked in kerosene to liquid propellant samples produced to reaction. This test simulates a fire that could develop during the storage of propellants. The question resolved is whether a detonation can occur of a fire hazard.

## ADIABATIC COMPRESSION

Adiabatic compression test results are depicted in Table 6. When a pressurization rate of 260,000 psi/sec was applied to LP 1845 and LP 1846, no detonations or explosions occurred. It is critical that the same test configuration be used for each test; otherwise, erroneous test data can be obtained. It has been found that a slight change, such as replacing the end cap with a pressure transducer cap, will have a pronounced effect upon the test results. The standard end cap which has a conical interior produced a detonation when tested with nitromethane. When a flat interior transducer cap replaced the standard cap, no detonation occurred. Both tests were conducted at the same pressurization rate. It is theorized that there is a heat transfer problem associated with the conical interior cap. A bubble can be trapped in the conical space which can afford a hot spot for a reaction to occur.

## CRITICAL DIAMETER

Critical diameter test data for LP 1845 and LP 1846 can be found in Table 7. The detonation probes indicated that no detonations occurred with LP 1845 in the 3 inch diameter test cylinders; but, a detonation was detected in the 4 inch diameter cylinder. LP 1846 proved to be less sensitive. Here no detonation was detected in the 4 inch diameter cylinder; however, a detonation wave was seen in the 5 inch diameter cylinder. This differential in the critical diameter can be attributed to approximately 3% more water in the basic LP 1846 stoichiometric formulation. The recommended 3:1 charge height to diameter was not followed from the standard test because the deviation in the planar wave across the diameter from the test charge used in this test would be negligible.

## FLASH POINT

Table 8 records the flash point test data results for LP 1845 and LP 1846. LP 1845 and LP 1846 are stoichiometric formulations containing 16.8% and 20.0% water, respectively. The vapor above both these compounds is essentially water. Therefore, one would expect that there would be no flash point for both these materials, and this was the case for both materials.

## MINIMUM PRESSURE FOR VAPOR PHASE IGNITION

LP 1845 and LP 1846 minimum pressure for vapor phase ignition data can be found in Table 9. As in the case for flash point, the vapor above the liquid phase is water. In this test, the LP materials began to decompose when the temperature reached 120C. Thus, there is no minimum pressure for vapor phase

gnition as the material does not ignite under these conditions.

## ELECTROSTATICS

The electrostatic test results for both liquid propellants evaluated can be seen in Table 10. Both materials proved negative to electrostatic ignition. Hydroxylammonium nitrate and triethanolammonium nitrate are nitrated salts that are completely ionized in the water portion of the basic formulation. Therefore, electrostatic charge build-up is readily dissipated through these formulations and can not materialize into a hazardous discharge.

## SUMMARY

Based upon the results of the small scale interim classification tests, the safety community assigned a Hazard classification of Class B Explosive, Division 1.3C. The assignment of a compatibility Group C poses a problem for the logistics chain. The safety community has characterized these materials as oxidizers. This assignment is incorrect since the basic formula contains an oxidizer and a fuel component. As a Group C category, liquid propellants can not be stored with solid materials. However, they can be stored with other oxidizers. To place this under a Group J category which is designated for liquid propellants and gels would require that the materials be stored separately from any other material. One can see the dilemma. A program has been recommended to resolve this problem.

The problems associated with the HAN-based liquid propellants sensitivity to decompose in contact with transition metals and nitric acid can be resolved. Strict manufacturing practices and storage containers compatible with the liquid propellants can eliminate or greatly reduce the threat of contaminating the liquid propellant. The surveillance program, that is in progress, will greatly enhance the design of the containers for long term storage. Any pressure generated from the HAN-based propellant's decomposition can be relieved through proper venting techniques and avoid any safety hazards while in storage.

The results of various tests have shown that the HAN-based liquid propellants are very insensitive to initiation. Destruction of armored vehicles has been the result of stored ammunition detonating when hit by a shaped charge. The need for insensitive munitions is critical for the preservation of the vehicle and more importantly the personnel. Successful gun firings of the HAN-based propellant in the 155mm regenerative gun scheduled for 1991 will usher in a new era for tactical weapons systems. The Hazard Classification program has paved the way for this attainment. This concludes the paper presentation.

UNITED NATIONS HAZARDOUS SUBSTANCE ACCEPTANCE PROCEDURE

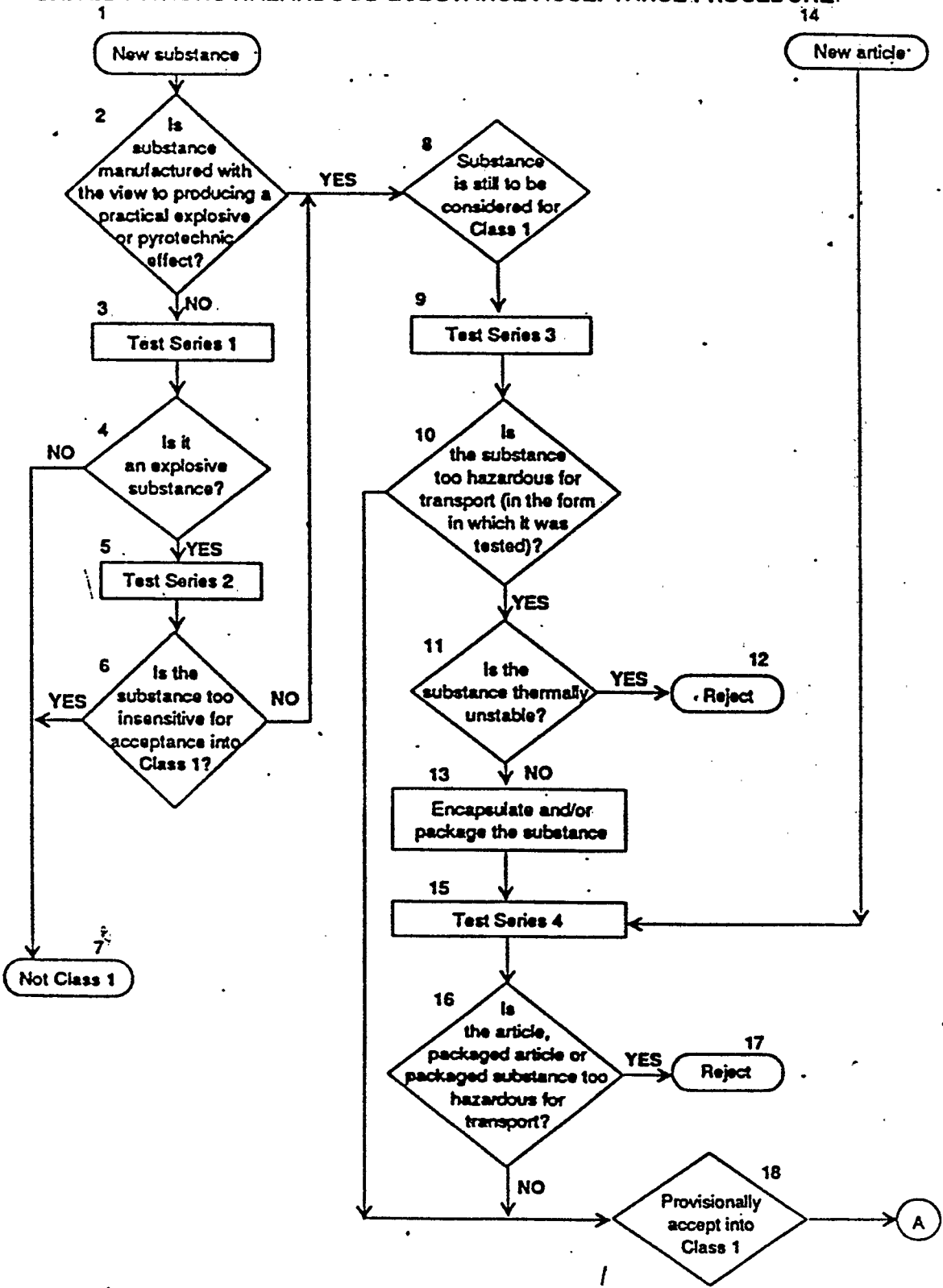


FIGURE 1

UNITED NATIONS PROCEDURE FOR ASSIGNMENT OF HAZARD DIVISION

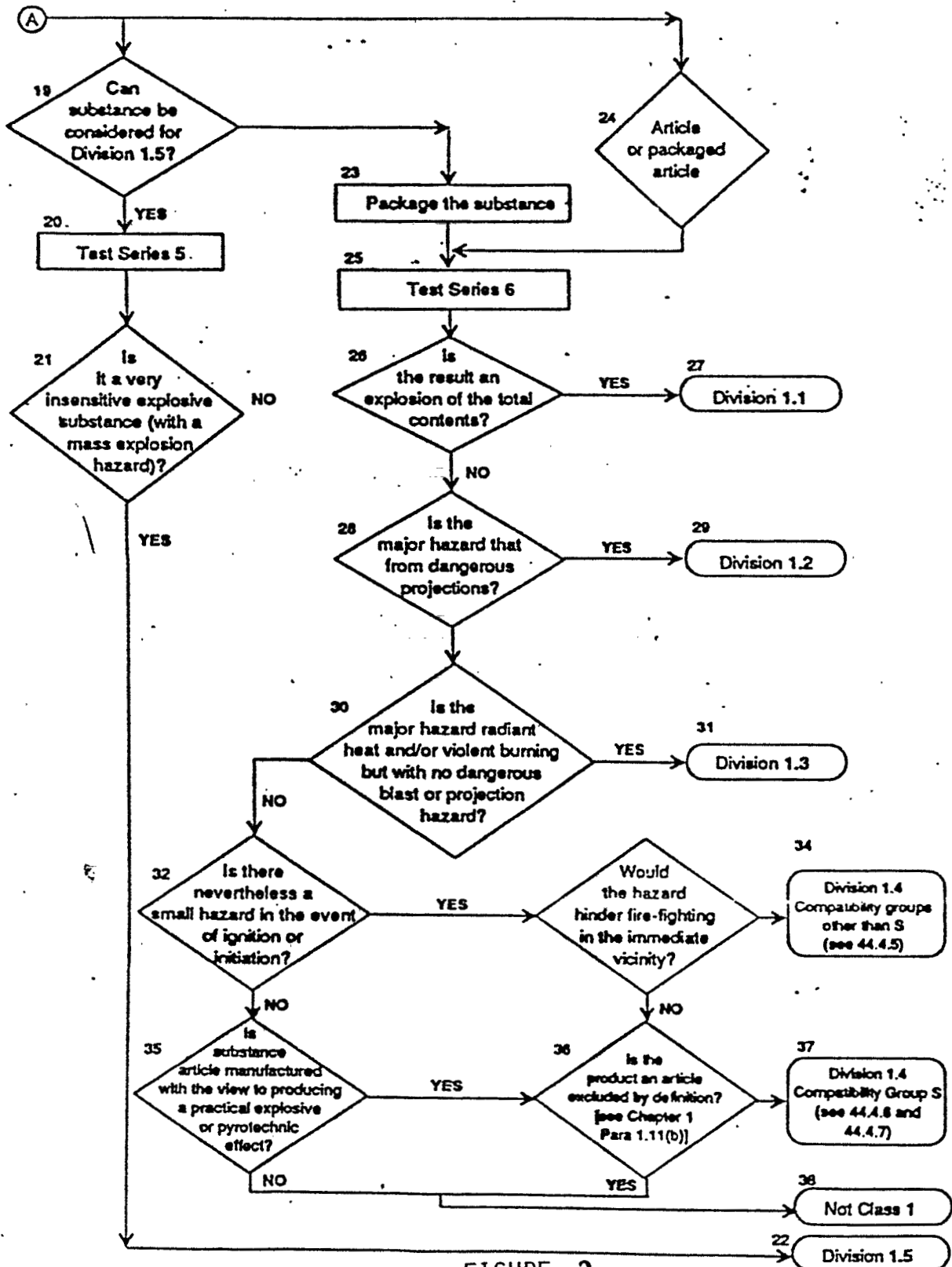


FIGURE 2

FIGURE 3

## UN DOCUMENT TEST SERIES 1

- **TYPE 1 (a) TESTS: SHOCK TESTS WITH A DEFINED BOOSTER UNDER CONFINEMENT**
  - TEST 1 (a) (i) BAM 50/60 STEEL TUBE TEST
  - TEST 1 (a) (ii) TNO 50/70 STEEL TUBE TEST
  - TEST 1 (a) (iii) CARD GAP TEST FOR SOLIDS
  - TEST 1 (a) (iv) CARD GAP TEST FOR LIQUIDS
  
- **TYPE 1 (b) TESTS: COMBUSTION OR THERMAL TESTS**
  - TEST 1 (b) (i) KOENEN TEST
  - TEST 1 (b) (ii) INTERNAL IGNITION
  - TEST 1 (b) (iii) SCB TEST

FIGURE 4  
UN DOCUMENT  
TEST SERIES 2

- \* TYPE 2 (a) TESTS: SHOCK TESTS WITH DEFINED BOOSTER UNDER CONFINEMENT
  - \* TEST 2 (a) (i) BAM 50/50 STEEL TUBE TEST
  - \* TEST 2 (a) (ii) TNO 50/70 STEEL TUBE TEST
  - \* TEST 2 (a) (iii) GAP TEST FOR SOLIDS
  - \* TEST 2 (a) (iv) GAP TEST FOR LIQUIDS
  
- \* TYPE 2 (b) TESTS: COMBUSTION OR THERMAL
  - \* TEST 2 (b) KOENEN TEST
  - \* TEST 2 (b) (i) INTERNAL IGNITION
  - \* TEST 2 (b) (ii) TIME/PRESSURE TEST
  - \* TEST 2 (b) (iii) SCB TEST

# UN DOCUMENT TEST SERIES 3

- (A) SENSITIVITY TO IMPACT
  - \* BUREAU OF EXPLOSIVE MACHINE
  - \* BAM FALLHAMMER
  - \* ROTTER TEST
  - \* 30 kg FALLHAMMER
  - \* MODIFIED TYPE 12 IMPACT TOOL
- (B) SENSITIVITY TO FRICTION
  - \* BAM FRICTION APPARATUS
  - \* ROTARY FRICTION
  - \* ABL FRICTION
- (C) THERMAL STABILITY
  - \* THERMAL STABILITY TEST AT 75C
- (D) RESPONSE TO FLAME
  - \* SMALL SCALE BURNING TEST (USA)
  - \* SMALL SCALE BURNING TEST (FRENCH)



FIGURE 6

# UN DOCUMENT TEST SERIES 4

- \* TYPE 4 (a) TEST: THERMAL STABILITY OF PACKAGED OR UNPACKAGED SUBSTANCES
  - \* TEST 4 (a) THERMAL STABILITY TEST FOR ARTICLES AND PACKAGES
- \* TYPE 4 (b) TESTS: EFFECT OF DROPPING THE EXPLOSIVE FROM A HEIGHT OF A FEW METERS
  - \* TEST 4 (b)(i) STEEL TUBE DROP TEST FOR LIQUIDS
  - \* TEST 4 (b)(ii) TWELVE METER DROP TEST FOR ARTICLES AND SOLID SUBSTANCES

FIGURE 7  
UN DOCUMENT  
TEST SERIES 5

- \* TEST 5(a): SHOCK TESTS-IGNITION BY STD. DETONATOR
  - \* TEST 5(a) CAP SENSITIVITY TEST
- \* TEST 5(b): THERMAL TESTS
  - \* TEST 5(b)(i) : DDT TEST- IGNITION BY HOT WIRE
  - \* TEST 5(b)(ii): DDT TEST- 5 GRAM IGNITER
- \* TEST 5(c): SUBJECT TO LARGE FIRE
  - \* EXTERNAL FIRE TEST
  - \* TEST 5(c): IGNITION BY INCENDIARY SPARK
    - \* PRINCESS INCENDIARY SPARK TEST
- \*\* NOTE: HAZARD DIVISION 1.5 MUST PASS ALL TESTS

# UN DOCUMENT TEST SERIES 6

- \* TEST TYPE 6(a): TEST ON SINGLE PACKAGE
  - \* WHETHER INITIATION OR IGNITION CAUSES BURNING OR EXPLOSION
  - \* SURROUNDINGS ENDANGERED BY THESE EFFECTS
- \* TEST TYPE 6(b): TESTS ON STACK OF PACKAGES
  - \* WHETHER BURNING OR EXPLOSION IS PROPAGATED FROM ONE PACKAGE TO ANOTHER
- \* TEST TYPE 6(c): EXTENAL FIRE TEST
  - \* HOW PACKAGES IN STACK BEHAVE TO EXTERNAL FIRE
  - \* SURROUNDINGS ENDANGERED BY BLAST WAVES, HEAT RADIATION AND FRAGMENTS

FIGURE 9

# NATO CHARACTERISTIC QUALIFICATION FOR LIQUID PROPELLANTS

1. GENERAL CHARACTERISTICS
  - 1.1 COMPOSITION
  - 1.2 TYPE\ROLE
  - 1.3 RELATED APPLICATIONS &  
COMPOSITIONS
  - 1.4 FABRICATIONS
  - 1.5 PHYSICAL PROPERTIES
2. CHEMICAL CHARACTERISTICS
  - 2.1 STABILITY
  - 2.2 COMPATIBILITY
  - 2.3 TOXICITY
3. PROPELLANT CHARACTERISTICS
  - 3.1 BURNING CHARACTERISTICS
  - 3.2 IMPULSE\IMPETUS
  - 3.3 HEAT OF COMBUSTION

# QUALIFICATION (NATO) FOR LIQUID PROPELLANTS

- \* QUALIFICATIONS GUIDLINES
  - \* DOD TB 700-2
  - \* NAVSEAINST 8020.8
  - \* TO 11 A-1-47
  - \* DLAR 8220
- \* QUALIFICATIONS
  - \* PHYSICAL PROPERTIES
    - \* DENSITY
    - \* MELTING POINT
    - \* BOILING POINT
    - \* COEFFICIENT OF THERMAL EXPANSION
    - \* VAPOR PRESSURE
    - \* FLAMMABILITY\DETONABILITY
  - \* CHEMICAL PROPERTIES
    - \* COMPATIBILITY
    - \* TOXICITY

FIGURE 11

# NATO QUALIFICATION TESTS FOR HAZARD CLASSIFICATION OF LP

- \* UNCONFINED BURNING ( BONFIRE )
- \* IMPACT
- \* CARD GAP
- \* MINIMUM PRESSURE FOR VAPOR  
PHASE IGNITION
- \* FLASH POINT
- \* ADIABATIC COMPRESSION
- \* DETONATION VELOCITY

FIGURE 11A

# NATO . OPTIONAL QUALIFICATION TESTS FOR LIQUID PROPELLANTS

- \* ATTACK BY FRAGMENT
- \* HIGH VELOCITY IMPACT
- \* DROP TEST PACKAGE
- \* OBLIQUE IMPACT
- \* CRITICAL CONDITIONS FOR  
SELF HEATING

FIGURE 12

# INTERIM HAZARD CLASSIFICATION TESTS FOR LIQUID PROPELLANTS

- \* THERMAL STABILITY
- \* IMPACT
- \* CARD GAP
- \* DETONATION TEST
- \* IGNITION AND UNCONFINED  
BURNING

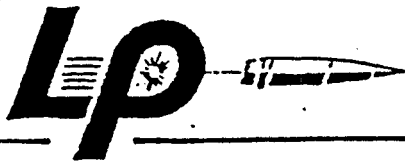
MANDATORY



FIGURE 13

# HAZARD CLASSIFICATION TESTS

- \* JANNAF THERMAL STABILITY
- \* ADIABATIC COMPRESSION
- \* CRITICAL DIAMETER
- \* FLASH POINT
- \* MINIMUM PRESSURE FOR VAPOR PHASE IGNITION
- \* ELECTROSTATICS



## **EVALUATION CRITERIA**

### **SMALL SCALE TESTS (INTERIM)**

#### **DOT "FORBIDDEN"**

- Thermal Stability: detonation, burning, marked decomposition

#### **DOT CLASS A (DOD 1.1) - if one or more occurs**

- Detonation Test: 1/8 inch or more deformation of lead cylinder
- Card Gap Test: detonation sensitivity of 70 or more cards
- Impact sensitivity: explosion at 4 inches, but not more than 10 inches
- Ignition and Unconfined Burning: detonation

#### **DOT CLASS B (DOD 1.3) - if ALL occur**

- Ignition and Unconfined Burning: deflagration
- Thermal Stability: no result
- Detonation test: no detonation AND Card Gap: detonation sensitivity less than 70 cards OR no reaction at zero cards
- Impact Sensitivity: no explosion at 10 inches or less

# HAZARD CLASSIFICATION LIQUID PROPELLANT END ITEM TESTS

- \* SINGLE PACKAGE TEST
- \* STACK TEST
- \* EXTERNAL FIRE
- \* BULLET IMPACT (50MM)
- \* PACKAGE DROP TEST
- \* OBLIQUE PACKAGE DROP TEST
- \* FRAGMENT TEST

FULL SCALE TESTS

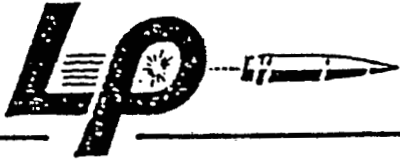


FIGURE 16  
**LIQUID PROPELLANT GUN DEMONSTRATION PROGRAM**

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## **EVALUATION CRITERIA**

### **LARGE SCALE, PACKAGE TESTS (FINAL)**

#### **DOT CLASS A (DOD 1.1)**

- Packages mass detonate
- Bulk materials with Card Gap of 70 or more cards

#### **DOT CLASS A (DOD 1.2)**

- Packages do not mass detonate
- Package tests produce hazardous fragments

#### **DOT CLASS B (DOD 1.3)**

- Packages do not mass detonate
- Bulk materials with Card Gap of less than 70 cards
- Radiant heat flux  $> 0.3$  cal/sq cm-sec beyond 100 feet

#### **DOT CLASS C (DOD 1.4)**

- Hazardous fragment and firebrand density no more than one/600 sq ft beyond 100 feet
- Radiant heat flux no more than  $0.3$  cal/sq cm-sec beyond 100 feet

FIGURE 17

# HAZARD CLASSIFICATION PACKAGE TEST ANALYSES

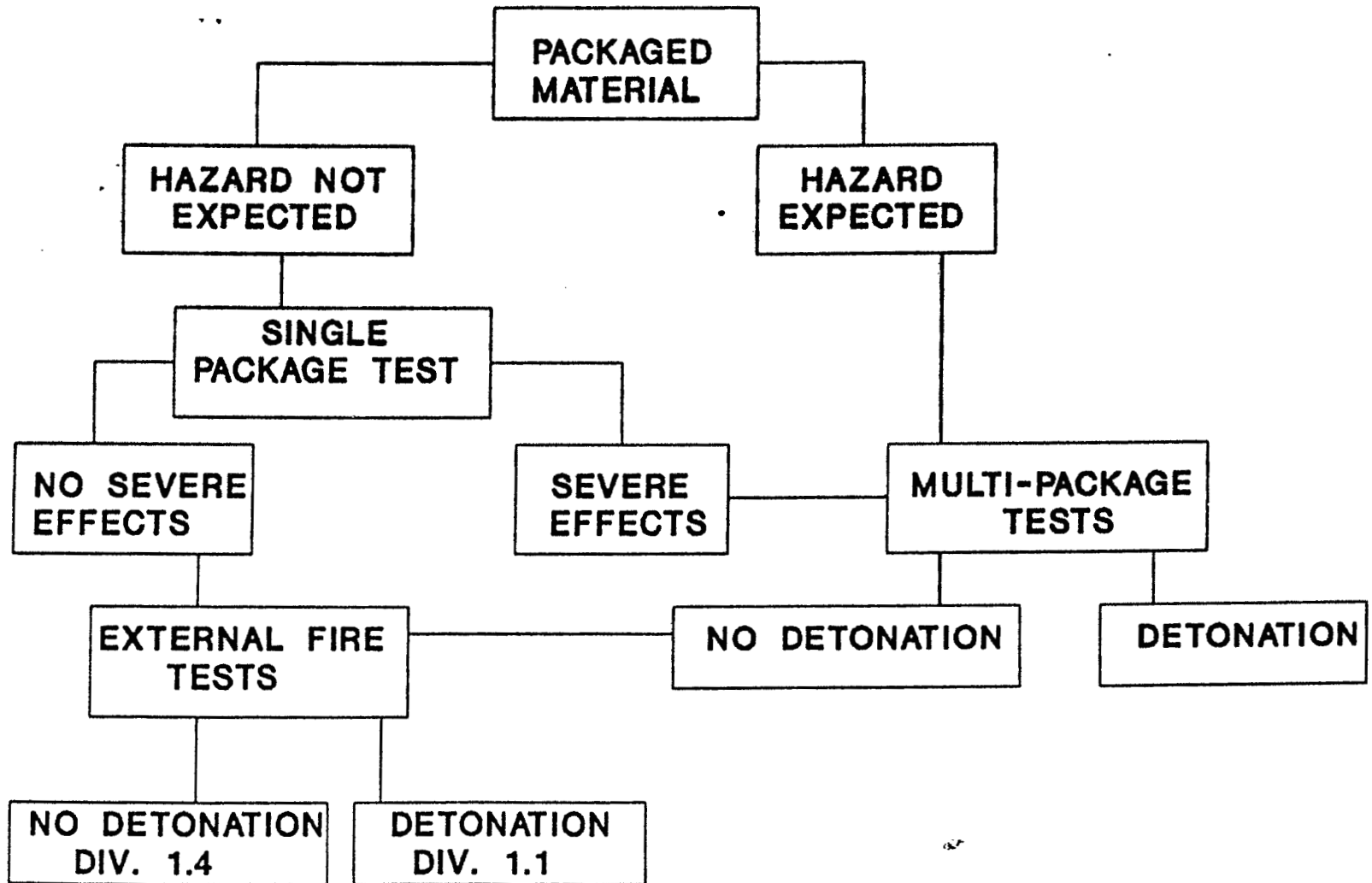


FIGURE 18

# HAZARD CLASSIFICATION PACKAGED MATERIAL ANALYSES

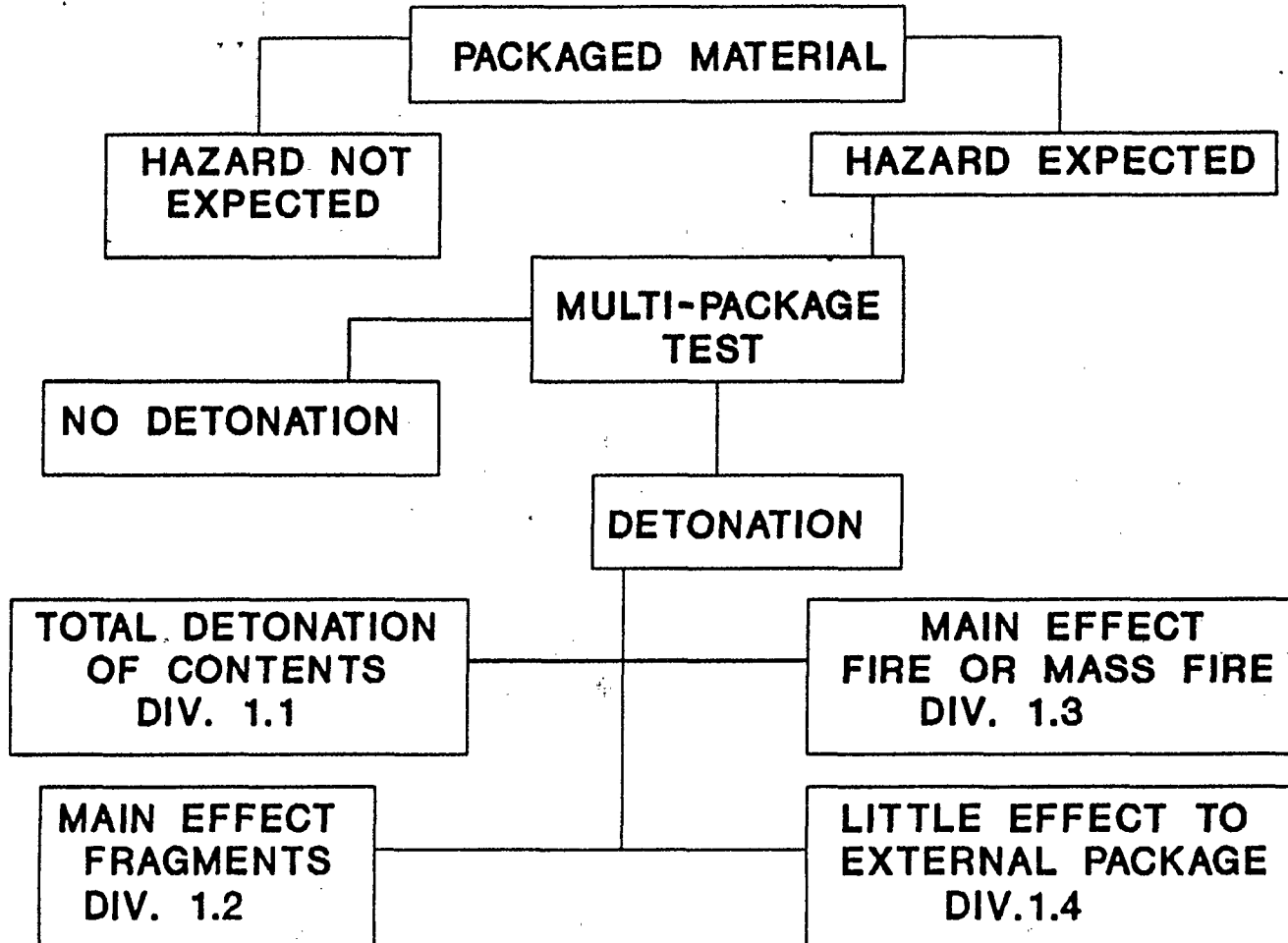


FIGURE 19

# Hazard Classification Methodology

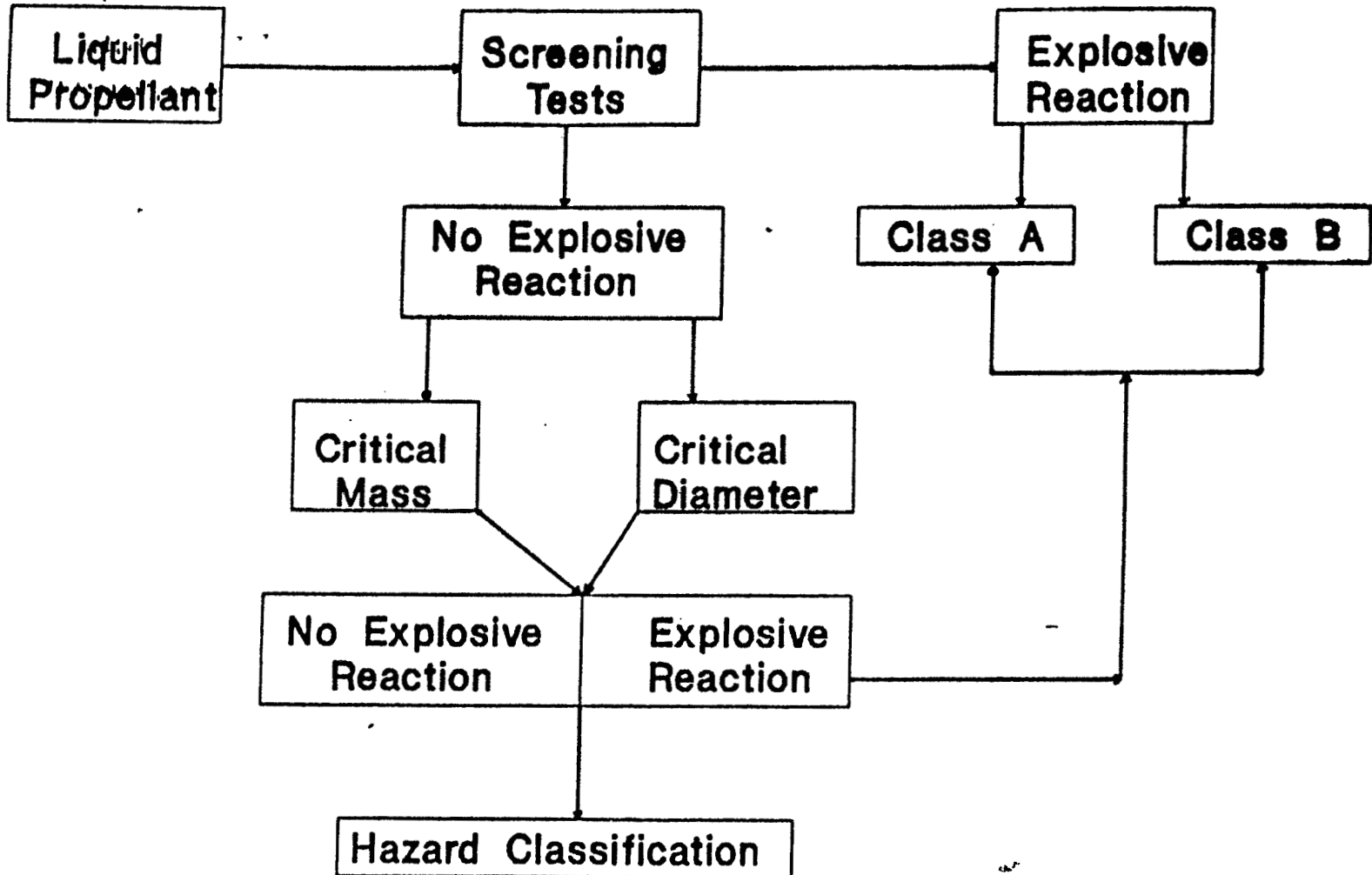
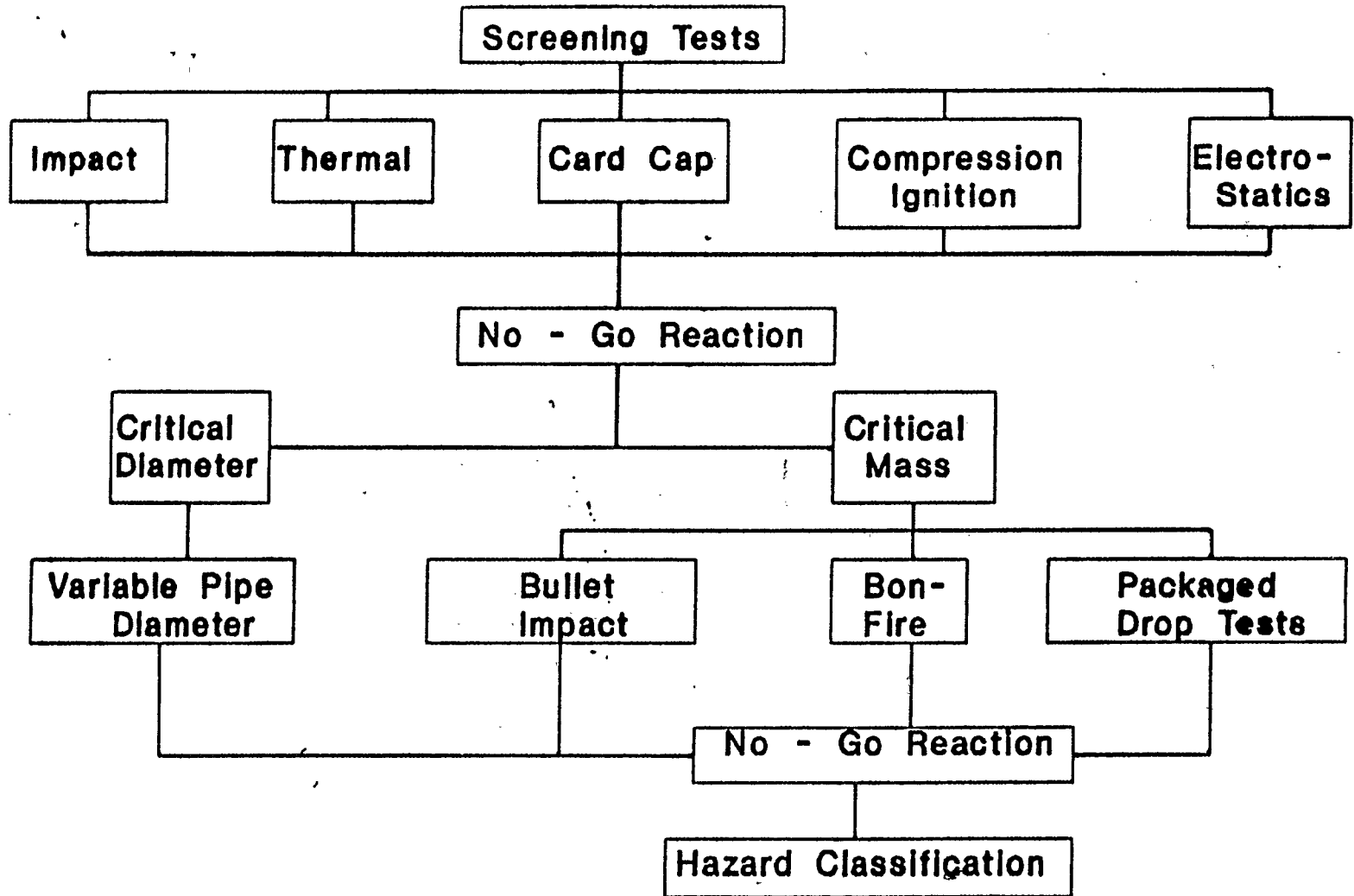


FIGURE 20

# No - Go Reaction Methodology

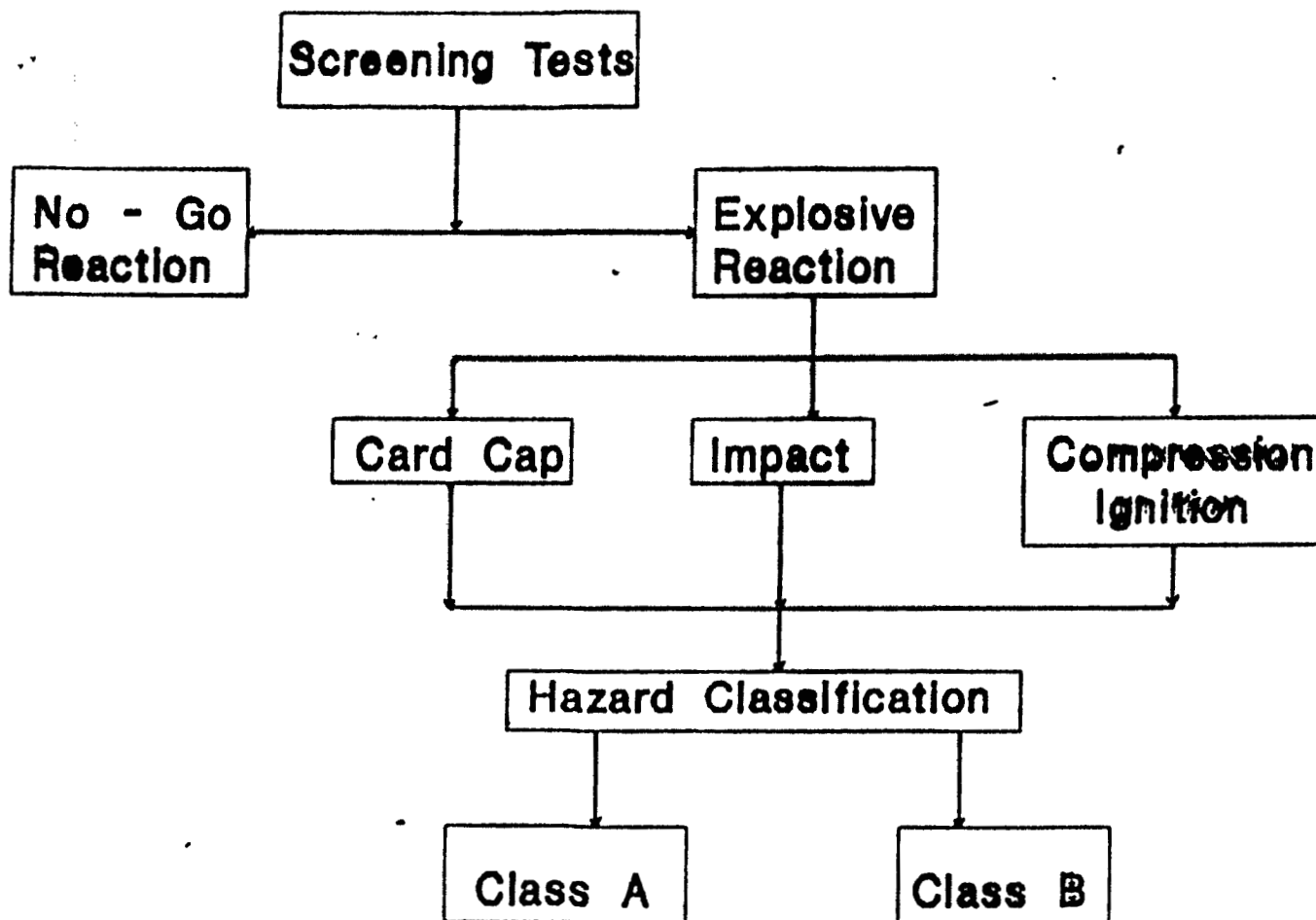


1691



FIGURE 21

# Explosive Reaction Methodology



1092

FIGURE 22

**TB 700-2 SOLID VS. LIQUID PROPELLANT TEST COMPARISON**

**Solid Propellant**

**Liquid Propellant**

**THERMAL STABILITY TEST REQUIREMENTS**

Constant temperature oven

Constant temperature oven

75°C for 48 hours

75°C for 48 hours

*2-in. propellant cube*

*8 cu in. of LGP in a 2-in. diameter polyethylene bottle*

**CARD GAP TEST REQUIREMENTS**

Tubing 1-7/8 in. diameter, 5.5 in. long

Tubing 1-7/8 in. diameter, 5.5 in. long

Two pentolite pellets

Two pentolite pellets

Engineers blasting cap

Engineers blasting cap

Steel plate 6×6×3/8 in.

Steel plate 6×6×3/8 in.

*Propellant sample machined or cast 1-7/8 in. diameter, 5.5 in. long*

*Propellant in a 1.5-mil polyethylene liner 1-7/8 in. diameter, 5.5 in. long*

**TB 700-2 SOLID VS. LIQUID PROPELLANT TEST COMPARISON**

**Solid Propellant**

**Liquid Propellant**

**DETONATION TEST REQUIREMENTS**

1.5-in. diameter lead cylinder

1.5-in. diameter lead cylinder

*No. 8 blasting cap*

*No. 8 blasting cap encased in 1.5-mil polyethylene film*

*2-in. propellant cube*

*8 cu in. of LGP in a 2-in. diameter polyethylene bottle*

1094

**IGNITION AND UNCONFINED BURNING TEST REQUIREMENTS**

Kerosene-soaked sawdust bed

Kerosene-soaked sawdust bed

Electric match igniter

Electric match igniter

*2-in. propellant cube*

*8 cu in. of LGP in a 2-in. diameter polyethylene bottle*

FIGURE 24

**TB 700-2 SOLID VS. LIQUID PROPELLANT TEST COMPARISON**

**Solid Propellant**

**Liquid Propellant**

**IMPACT SENSITIVITY TEST REQUIREMENTS**

Bureau of Explosives tester

Bureau of Explosives tester

*Sample placed in cup assembly*

*Sample placed in stainless steel cup assembly modified to accept liquids*

Ten tests at 3-3/4 in. drop height

Ten tests at 3-3/4 in. drop height

Ten tests at 10 in. drop height

Ten tests at 10 in. drop height

**CRITICAL DIAMETER TEST REQUIREMENTS**

*Velocity probes*

*Velocity probes*

*Witness plate*

*Stainless steel witness plate welded to tubing*

Pentolite explosive charge, 51 grams

C-4 explosive charge, 311 kJoules/in<sup>2</sup>

E-99 detonator

E-99 detonator

*Tubing, 1/8 to 1 in. OD*

*Stainless steel tubing 2, 3, 4, and 5 in. OD with wall thickness of 0.0625 in. Welded assembly must be passivated with nitric acid prior to test*

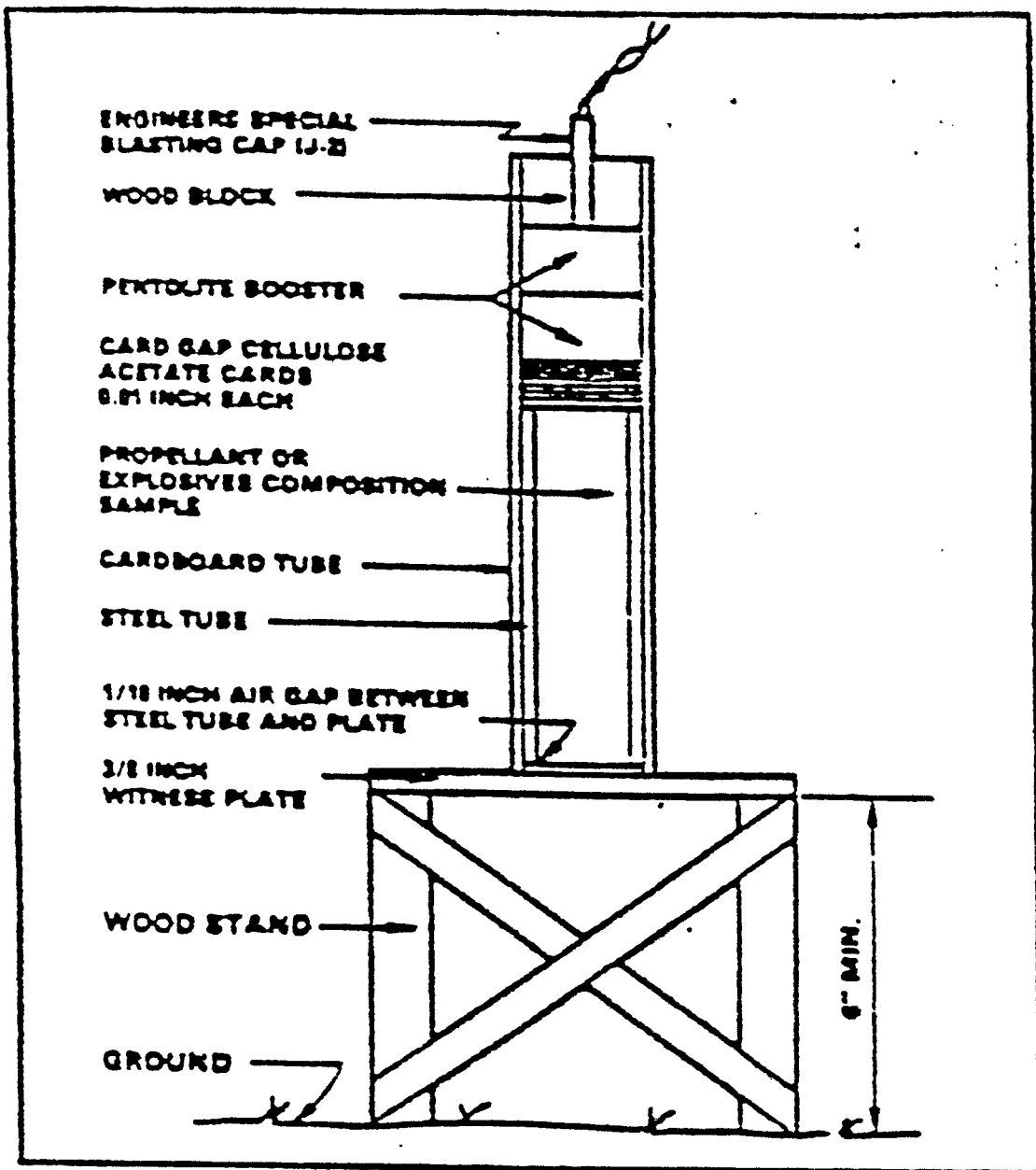
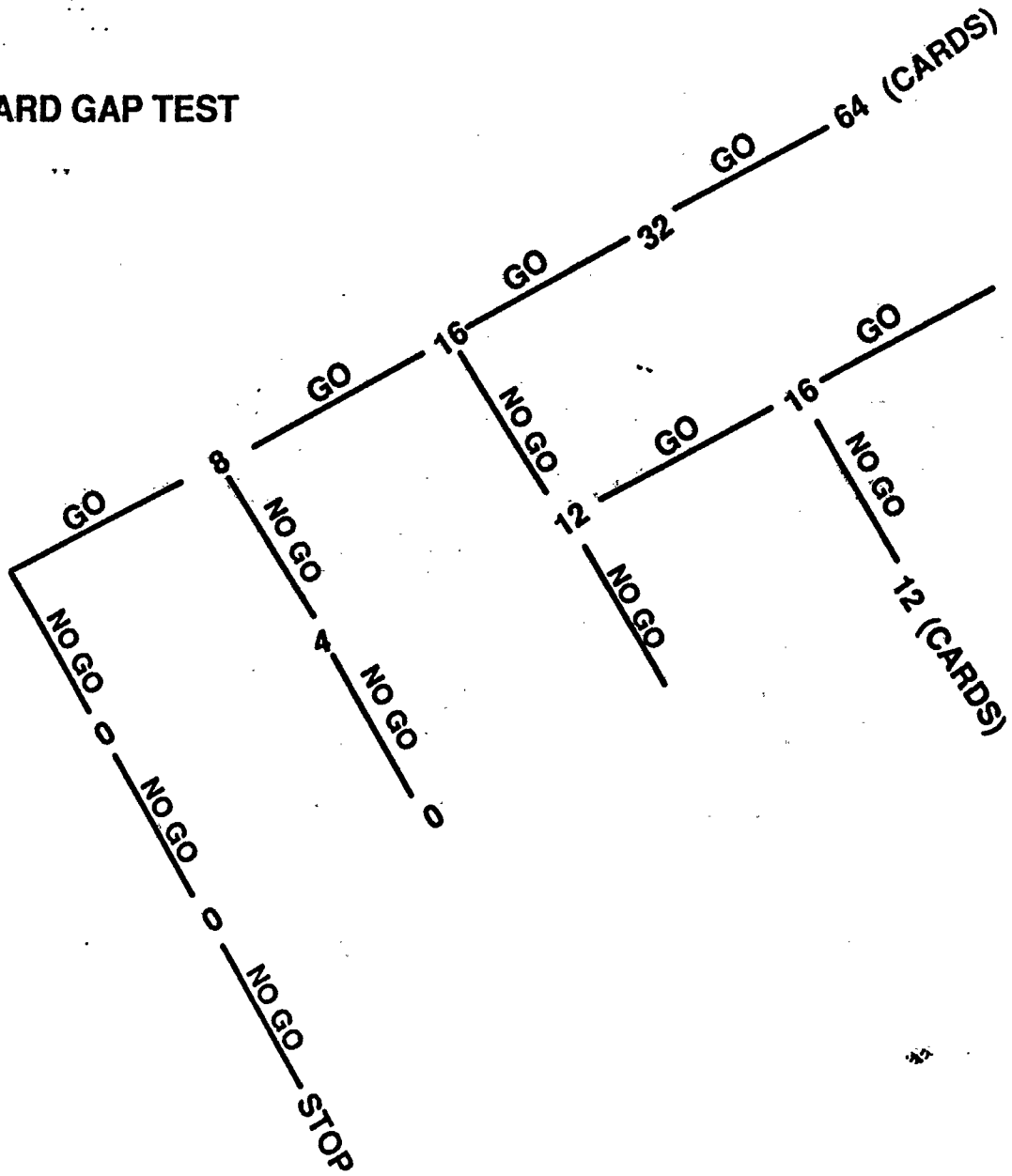


Figure 25. Card Cap Test Configuration

FIGURE 26

### CARD GAP TEST



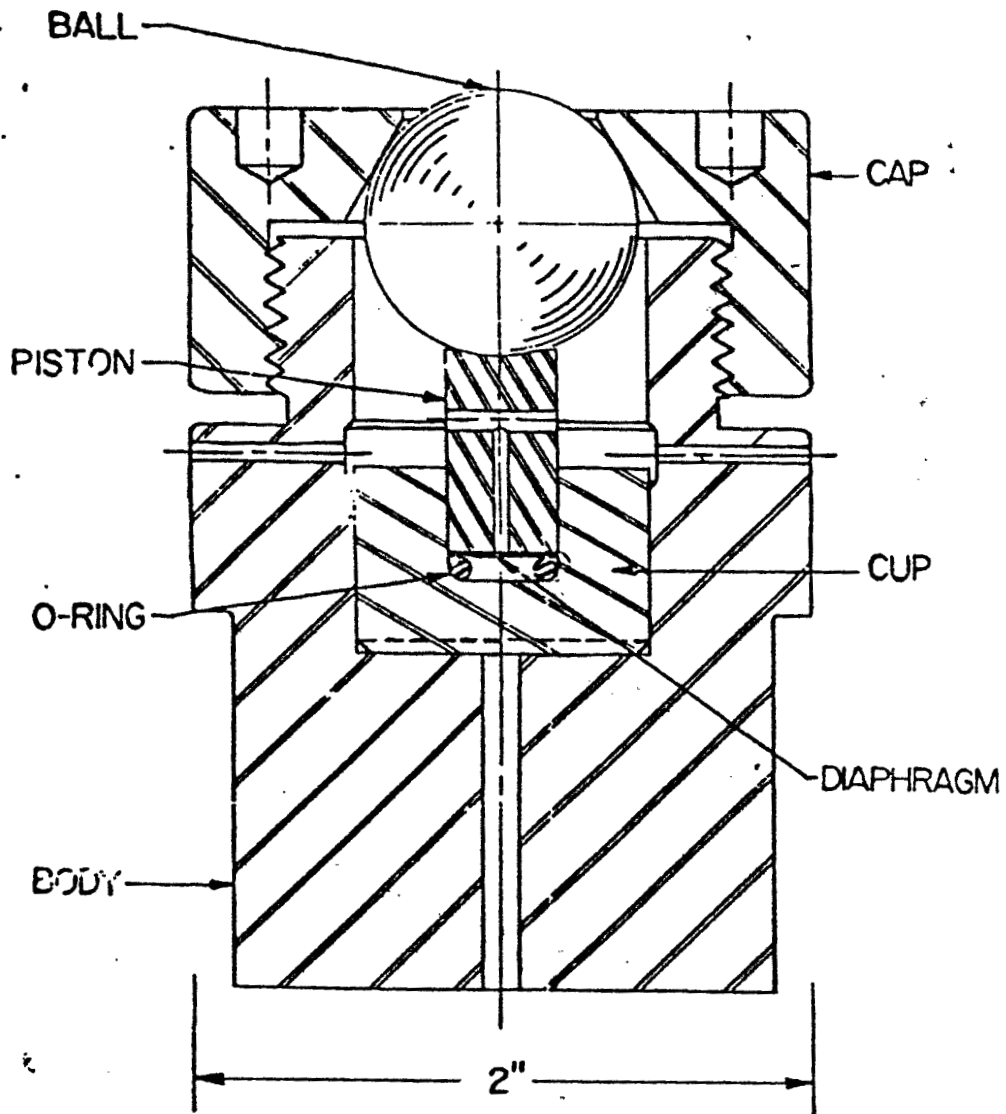


FIGURE 27 - SAMPLE CUP ASSEMBLY

1099

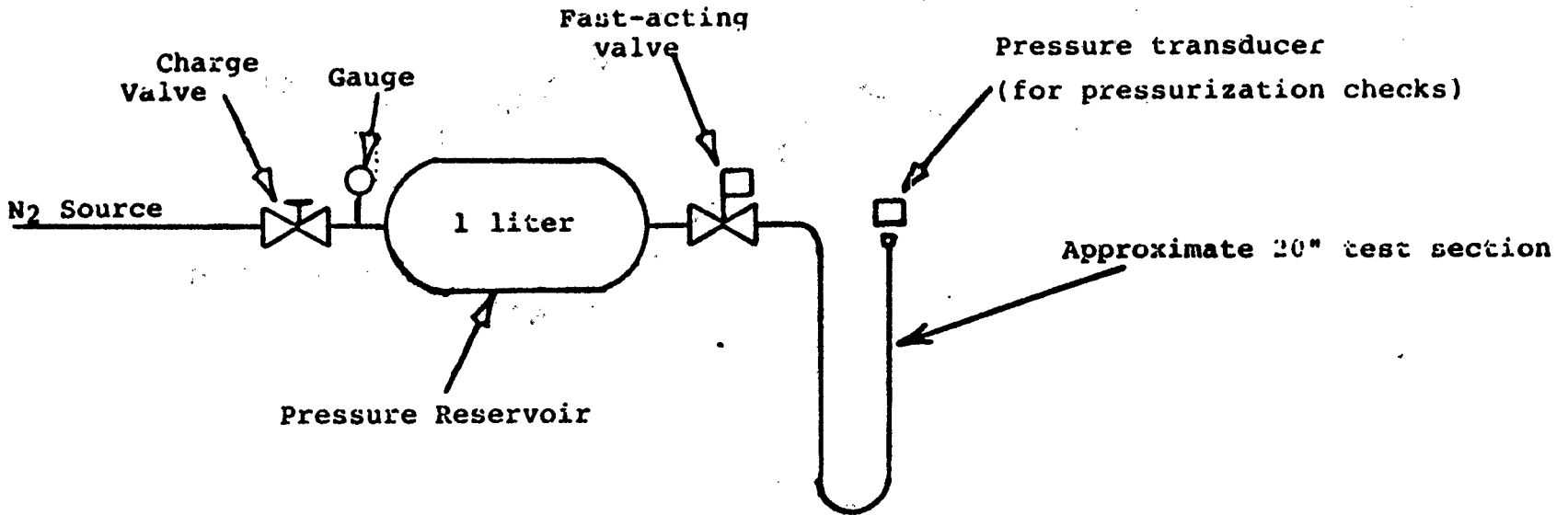


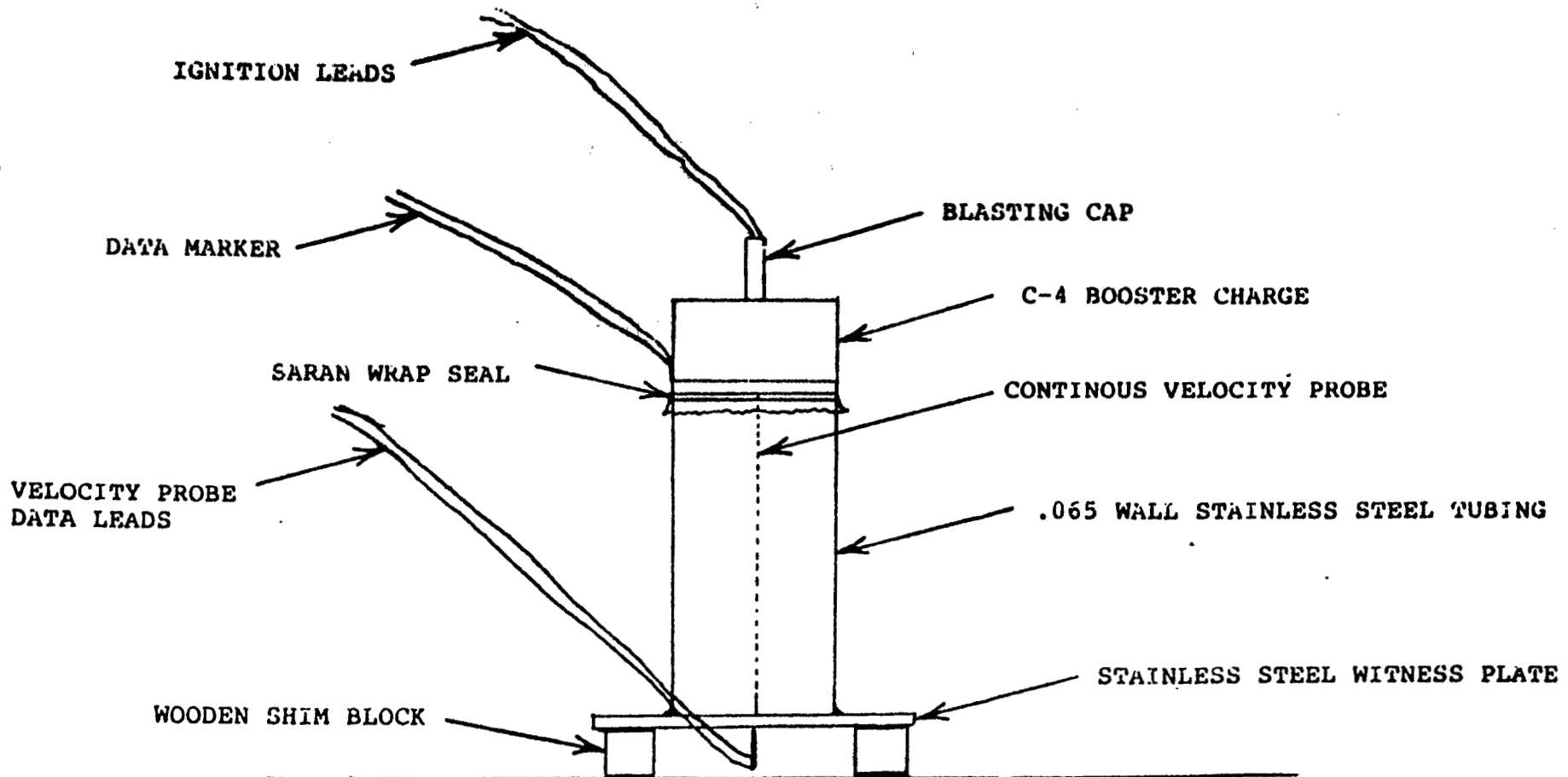
FIGURE 28



FIGURE 29

CRITICAL DIAMETER TEST SET-UP

1100



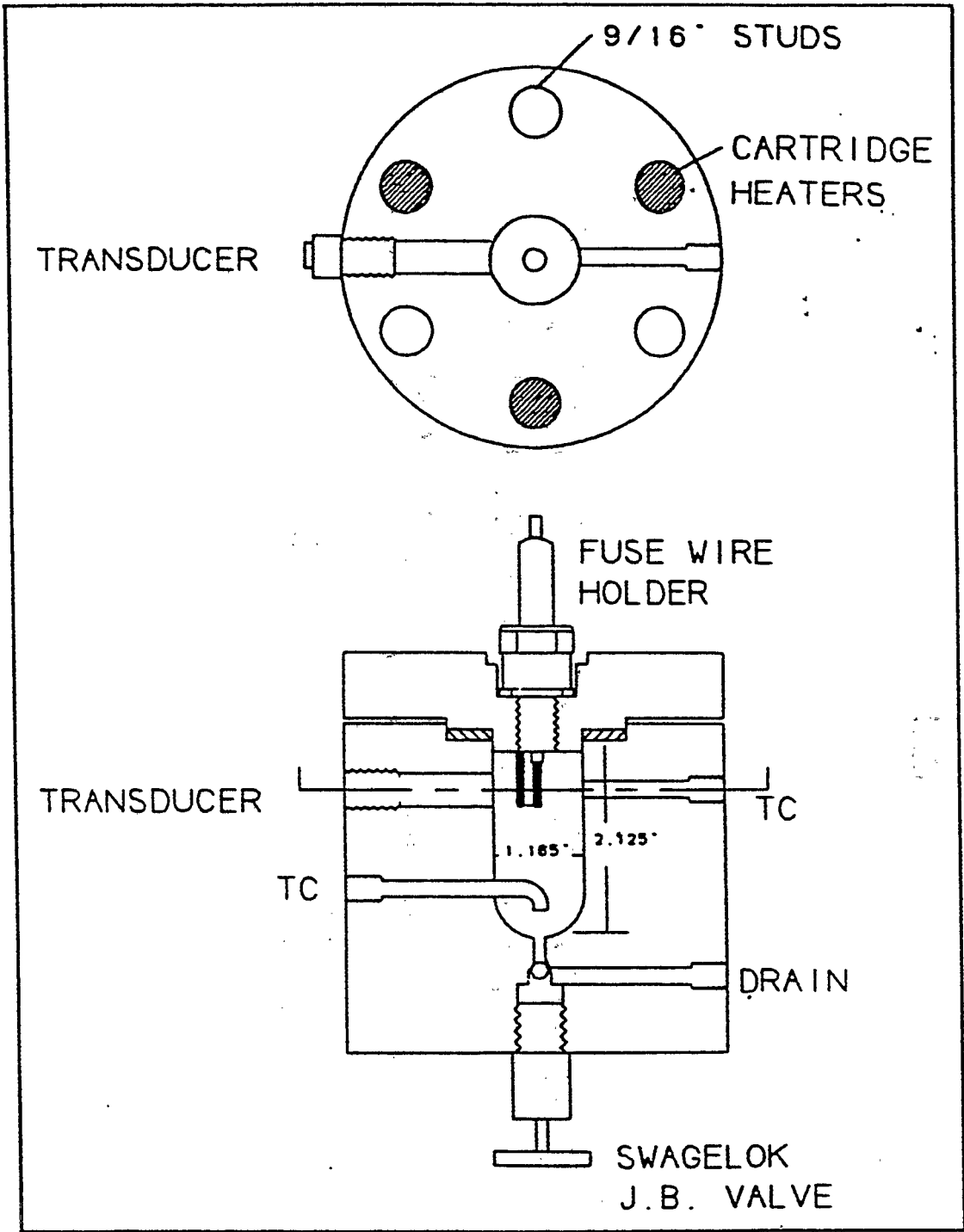


Figure 30 Pressure Test Vessel

TABLE 1

**THERMAL STABILITY TEST RESULTS  
48 HOURS AT 75°C IN VENTED OVEN**

<b>SAMPLE ID</b>	<b>REACTION</b>
<b>LP 1845</b>	<b>NONE</b>
<b>LP 1846</b>	<b>NONE</b>

TABLE 2

**CARD GAP DATA FOR  
LP 1845 AND LP 1846**

<b>SAMPLE IDENTIFICATION</b>	<b>NUMBER OF CARDS</b>	<b>VISUAL OBSERVATION</b>
1. LP 1845	0	WITNESS PLATE DEFORMED NO HOLES IN PLATE
2. LP 1846	0	WITNESS PLATE DEFORMED NO HOLES IN PLATE

TABLE 3

**IMPACT TEST DATA FOR LP 1845 AND LP 1846**

SAMPLE ID	DROP HEIGHT (IN.)		
	0%	50%	100%
LP 1845	28	30	31
LP 1846	29	30.5	33

1104

TABLE 4

## DETONATION TEST RESULTS

**SAMPLE ID**

**DETONATION REACTION**

**LP 1845**

**NONE**

**LP 1846**

**NONE**

TABLE 5

## IGNITION AND UNCONFINED BURNING TEST RESULTS

SAMPLE ID	DETONATION REACTION
LP 1845	NONE
LP 1846	NONE

TABLE 6

## ABIABATIC COMPRESSION TEST RESULTS

SAMPLE ID	REACTION
CONTROL (WATER)	NONE
LP 1845	NONE
LP 1846	NONE
NITROMETHANE	DETONATION

1107



TABLE 7

## CRITICAL DIAMETER TEST DATA

SAMPLE IDENTIFICATION	BAFFLES*	DETONATION REACTION
1. LP 1845	NO	NO REACTION AT 3 INCHES REACTION AT 4 INCHES
2. LP 1846	NO	NO REACTION AT 4 INCHES REACTION AT 5 INCHES
3. LP 1845	YES	NO REACTION AT 3 INCHES REACTION AT 4 INCHES
4. LP 1846	YES	NO REACTION AT 4 INCHES REACTION AT 5 INCHES

\* WHIFFLE BALL TYPE POLYETHYLENE SPHERES OCCUPYING APPROXIMATELY 12% OF THE CANISTER VOLUME

TABLE 8

**FLASH POINT**  
**75°C (PROPANE FLAME)**

**SAMPLE ID**

**REACTION**

**LP 1845**

**NO REACTION**

**LP 1846**

**NO REACTION**

TABLE 9

## MINIMUM PRESSURE FOR VAPOR PHASE IGNITION

SAMPLE ID	REACTION
WATER (CONTROL)	NONE
LP 1845	NONE (MATERIAL DECOMPOSED)
LP 1846	NONE (MATERIAL DECOMPOSED)

TABLE 10

**ELECTROSTATIC TEST RESULTS**  
**1 MICROFARAD AND 12.5 JOULES AT 5 KV**

<b>SAMPLE ID</b>	<b>REACTION</b>
<b>LP 1845</b>	<b>NO REACTION</b>
<b>LP 1846</b>	<b>NO REACTION</b>

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