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DEVELOPMENT OF HAZARDS CLASSIFICATION DATA ON PROPELLANTS AND EXPLOSIVES

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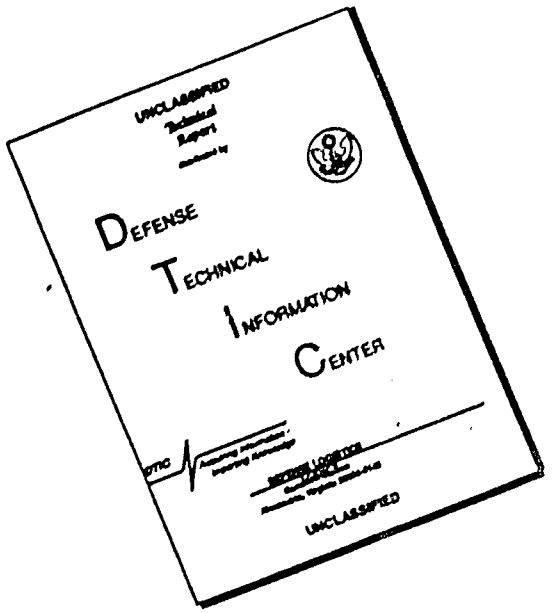
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3) Review and critique of current hazards classification procedures and quantity distance requirements

4) Preliminary development of a hazards classification procedure for in-process materials, and

5) Experimental evaluation of selected small-scale tests for application in the hazards classification of in-process materials.

It was found that the most common accident causes are friction, impact, electrostatic discharge, and thermal heating. Test methods are suggested for evaluating the sensitivity of in-process materials to these stimuli.

A preliminary outline is presented of a new explosive classification procedure that considers other hazards (viz. fragments, thermal effects, glass breakage) in addition to blast.

FOREWORD

This investigation dealt with the development of a procedure for the hazards classification of in-process explosive and propellant materials.

D. R. Morita was the IITRI project engineer during the initial, analytical phase of the study and was the primary contributor to the first part of this report. D. Kalkbrenner, aided by J. Mavec, was responsible for the experimental portion of this work. In addition to the authors, other IITRI personnel who contributed to this program were R. Pape, A. Goldsmith, J. Daley and M. Amor.

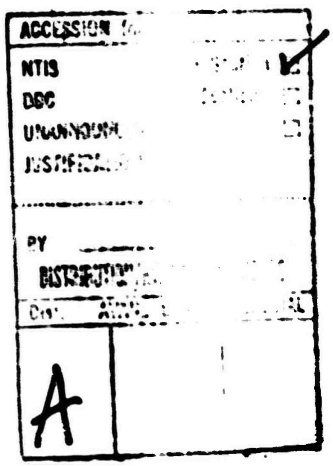


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SUMMARY

There are many deficiencies in the current hazards classification schemes. Some of these are related to the classification procedures, some to the implementation of the procedures, and some to the final usage of the assigned classifications (quantity-distance). The most significant of these deficiencies are the following:

1) The DOD hazard classification system nominally offers seven classes to which a material can be assigned. However, most materials of the type found in a manufacturing process are assigned to either Class 2 or Class 7. These two classes do not cover the range of hazards associated with in-process materials.

2) Class 2 is supposed to include materials which are only a fire hazard. In actuality, however, Class 2 materials can experience low to medium velocity detonations and explosions as well as fires. The hazards associated with such detonations and explosions (fragments, blast overpressure) are not covered in any of the quantity-distance requirements associated with Class 2. Even the ability to protect against fire hazards is somewhat marginal since asymmetric burning is not considered.

3) Class 7 is supposed to cover mass detonable materials. The assignment to Class 7 however, is based only on tests of small quantities of material. Therefore it is possible, in some cases where there are large critical diameters or heights, that mass detonable materials are put into Class 2 erroneously. The quantity-distance requirements for Class 7 are inadequate. They provide only for minimizing structural damage and do not consider human casualties which could occur due to impulse, fragments or glass breakage.

4) The Class 7 distances were based on the distances specified in the old American Table of Distances. This table was prepared from data on 117 accidents from around the turn of the century. Most of the materials involved in the accidents, especially at the

higher quantity levels, had TNT equivalencies less than one. A prime example is black powder with a TNT equivalency of about 30 percent. Thus, the pressures that are experienced at a given distance were generally less than would occur with today's materials, which may have TNT equivalencies in excess of one. In addition, it was apparent that the decision not to include fragment and glass breakage hazards was made at this time, as none of the data on these hazards were utilized.

During this program, the most probable causes of an accident were identified in an accident analysis. The causes varied with the process operation and material type. However, friction, impact, electrostatic discharge and heating were the most commonly identified causative stimuli.

A comparison was made of the ignition sensitivities of materials involved in accidents with those of other materials not involved in accidents. Statistically significant differences in sensitivity for the two groups of materials were identified for friction, impact, and electrostatic discharge stimuli. These differences formed the basis for deriving sensitivity criteria for the different stimuli. No differences were found for ignition due to heating, impingement or adiabatic compression.

A preliminary hazards classification procedure was developed. It combines material properties with sensitivity and effects testing. A conversion to the UN hazards classes was made, and the concept of a threat equivalency is introduced.

Tests which could be applicable in hazards classification procedures for in-process materials were surveyed, and the most promising candidates were selected for experimental evaluation. Four representative in-process propellant and explosive materials were chosen for the program, and eight detailed evaluations were completed. These were:

- Transition to detonation
- Impact sensitivity
- Friction sensitivity
- Dusting propensity
- Dust explosibility
- Electrical properties measurement
- Electrostatic discharge, and
- Thermal sensitivity

INTRODUCTION

The United States Army Materiel Command is assuming authority and control over all government-owned ammunition plants. As such, the U. S. Army has the responsibility for ensuring the safety of the plants. One vital aspect of the safety assurance program is the specification of proper separation distances within each plant. Thus quantity-distance requirements and hazards classification are important. Unfortunately, the current hazards classification document, TB700-2, (Ref 1) specifically excludes all in-process materials. Therefore this study was aimed at the development of a hazards classification procedure for in-process materials.

There is an intimate relationship between hazards classification and quantity-distance (QD) requirements. The term "quantity-distance" is used to designate the relationship between quantities of explosive materials and the distance between them, or the distance between an explosive and a vulnerable installation such as an inhabited building, a traffic route, aircraft, etc. Minimum separation distances are prescribed to reduce risks to acceptable levels.

Hazards classification is the assignment of a material or end item (in this case only in-process materials) to a particular hazard class which best describes the threat presented by the material. This requires the use of a hazards classification procedure which provides the guidelines and criteria on which the choice of the hazards class is based. The assigned hazards class of the material is then used as the basis for selecting the proper quantity-distance relationship. Thus, if the hazards classification procedure erroneously assigns a material to the wrong class, either safety is compromised or excessive safety requirements are imposed. Both possibilities are expensive.

This study dealt with the hazards classification problem in the following ways:

- By considering the shortcomings of existing classification procedures
- By considering how the threats imposed by in-process materials relate to the quantity-distance requirements
- By using actual accident experience as criteria for classifying the materials, and

- By combining in a logical manner sensitivity and effects testing.

As a result, a comprehensive hazards classification procedure was developed for in-process materials. It should be emphasized that this procedure is preliminary in nature and is subject to future modifications.

There is a large body of data related to sensitivity which has been compiled at the various ammunition plants as a result of in-house testing or from hazards analyses of the plants. It is expected that much more data will be collected in the future. The hazards classification procedure presented in this report has been prepared in such a manner that existing data can be used. This will eliminate duplication of effort and minimize the cost of classifying materials.

A SURVEY AND REVIEW OF CURRENT HAZARDS CLASSIFICATION PROCEDURE

Hazards classification, in the narrowest sense, is the assignment of a hazards class to a material or end item. This, of course, requires the use of a hazards classification procedure which provides the guidelines and criteria on which the choice of the hazards class is based.

Hazards classification, however, is much broader than what the hazard classes or classification procedures alone would suggest. This is due to the fact that the hazards classification impacts upon other areas, notably the quantity-distance requirements.

There are three hazards classification schemes which either are or soon will be in use in the United States. The most commonly known is that represented by TB700-2 (Ref 1). This document presents a formalized set of procedures for determining the hazard classification of explosives, solid propellants, and end items containing either or both of these materials. Either specifically or by implication, it does not include in-process materials or operations, hazards due to electrostatic influence or liquid explosives. A preliminary draft version of an explosives hazard classification procedure, currently under discussion at DoD presents a formalized set of procedures for determining the hazard classification of explosives, solid propellants and end items. It differs from TB700-2 in that NATO-UN hazard classifications are assigned. It specifically or implicitly does not include the same materials as TB700-2.

The in-process materials to be classified as a result of this program include solid and liquid explosives, slurries and solid propellants. Therefore, it was useful to review all three documents -- TB700-2 (Ref 1), CPIA/194 (Ref 2), UN-NATO (Ref 3).

Hazard Classes

For the Department of Defense, the hazard classes are defined in DoD 4145.26 (Ref 4), described in AMC 385-100 (Ref 5) and are assigned on the basis of TB700-2 (Ref 1). In all, there are eight DoD hazard classes. These are listed in Table 1. The hazard classification of liquid propellants are defined in CPIA/194 (Ref 2.) In this scheme, there are four hazard groups. These groups are listed in Table 2.

Table 1

DoD hazard classes and typical items assigned to each class

Class	Type of hazard and examples
1	High fire hazard with no blast and virtually no fragmentation (Ref 4) -small arms ammo, squibs and safety fuse (Ref 5)
2	Vigorous fires, firebrands, explosions (Ref 4) - military pyrotechnics, bulk solid propellant, CBR items (Ref 4)
3	Fragments, toxicity or blast (Ref 4) - rocket igniters, artillery and cannon primers, primer detonators (Ref 5)
4	Fragments, toxicity or blast (Ref 4) -illuminating cartridges, bounding type AP mines (Ref 5)
5	Fragments, toxicity or blast (Ref 4) - explosive D loaded projectiles, chemical munitions with explosive bursters, (Ref 5)
6	Fragments, toxicity or blast (Ref 4) - TNT loaded projectiles, Amatol or Ammonal loaded projectiles (Ref 5)
7	Mass detonation (Ref 4) - bombs, detonators, warheads, explosives (Ref 4)
8	CB agents (Ref 4) - Groups A and B chemical ammunition (Ref 5)

Table 2

CPIA/194 hazard groups for liquid propellants (Ref 2)

Group	Type of hazard
I	Fire hazard potential - alcohol, anhydrous ammonia, hydrocarbon fuels.
II	Flare type fire if fuel is present - fluorine, halogen fluorides, LOX.
III	Container rupture or explosion -boranes, methane, ethylene oxide, LH_2.
IV	Mass detonation - nitromethane, n-propylnitrate/ethyl nitrate.

NATO and the United Nations have arrived at a system for the classification of explosives (Ref 6). There are eight classes of dangerous goods in which explosives and propellants belong to Class 1. Within Class 1, there are four subdivisions which are used for quantity-distance purposes. These are given in Table 3.

Table 3

NATO-UN hazards classes

Class division	Hazard description
1.1	Mass detonating
1.2	Non-mass detonating -- fragment producing
1.3	Mass fire
1.4	Moderate fire -- no blast

The NATO-UN classification scheme is important as the United States has adopted the scheme and began its implementation in January 1977.

There appears to be some correspondence between the various hazard classification schemes. Table 4 shows the correlation between DoD, NATO-UN (Ref 6), and CPIA classification schemes.

Table 4

Correlation between DoD, NATO-UN and CPIA hazard classification schemes

DoD Class	NATO-UN Class Division	CPIA Group
1	1.4	I
2	1.3	II
3	1.2	III
4	1.2	III
5	1.2	III
6	1.2	III
7	1.1	IV

The best correlation is between the NATO-UN and CPIA classification schemes.

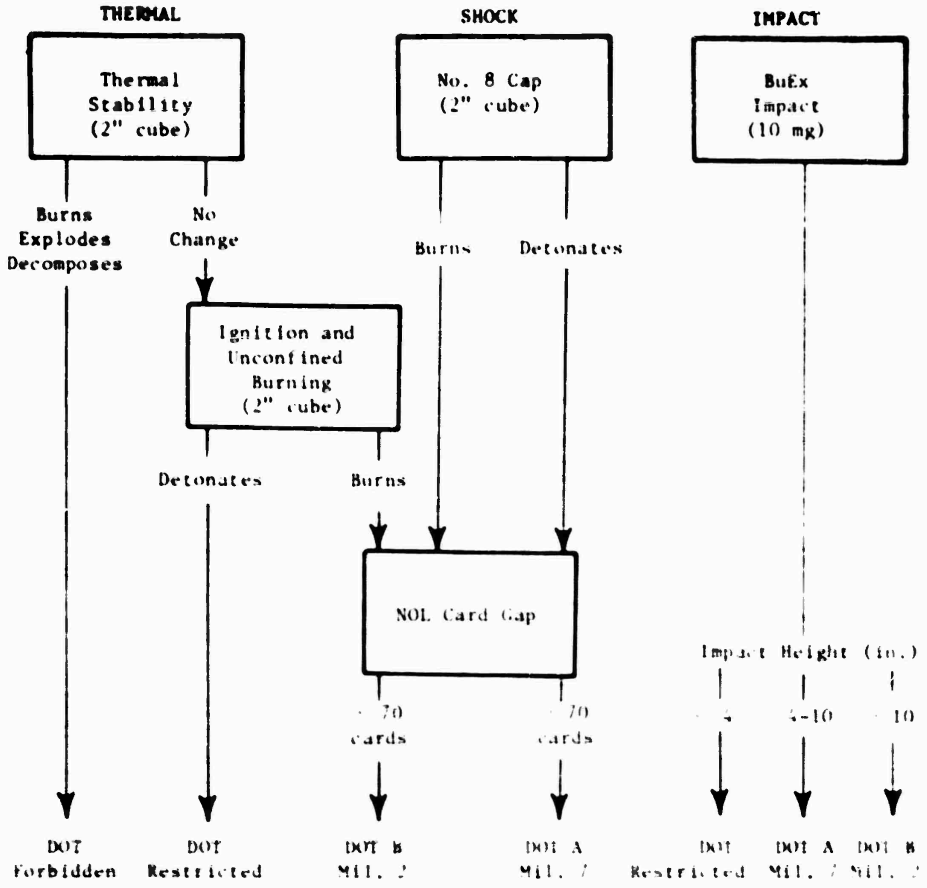
Hazard Classification Procedure

The current guide to DoD hazard classification is TB700-2 which sets forth procedures for the assignment of the quantity-distance class. The guide consists of five chapters. Chapters 1 and 2 consist of introductory general information. Chapters 3 to 5 give minimum test criteria for:

- bulk explosive and solid propellant compositions,
- ammunition and explosives items, and
- quantities of large ordnance containing solid propellants for establishing quantity-distance criteria.

Of these, only Chapter 3 contains a section on "Interpretation of Results" which enables conversion of test results to a hazards class. B. Brown (Ref 7) shows how the hazards class is assigned. The pertinent diagram from Ref 7 is reproduced here as Fig 1.

Fig 1 shows that bulk explosive and solid propellant compositions can be assigned only to Military Class 2 or Class 7. Under certain circumstances, no class is assigned. Thus, as is pointed out by Settles (Ref 8), Class 7 consists of materials which exhibit high velocity detonation under the test conditions, and Class 2 consists of materials which can exhibit medium or low velocity detonation, explosion, or fire.



NOTE THAT:

Transportation and handling (impact) safety is based on results of testing 10 mg samples only

fragment or firebrand data are obtained

Fig 1 Current bulk composition test interpretation scheme (1967 revision of TB700-2, Ref 7)

CPIA/194 provides hazard group designations for selected liquid propellants. However, it does not indicate the basis for assigning a given propellant to a given group. It also states that the classification of prepackaged items of liquid propellant containing both fuel and oxidizer may be accomplished using the procedures of TB700-2. This is impossible as either the tests in TB700-2 are not capable of dealing with liquids (Chapter 3) or are specifically for solid propellants (Chapter 5).

The preliminary DoD draft document (Ref 3) which includes the NATO-UN hazard classes utilizes tests for classifying materials similar to those given in TB700-2, Chapter 4. The data to be collected in the tests however, are much more extensive than that collected in TB700-2. In addition, detailed criteria are used to put each material into its proper class. These criteria are summarized in Table 5.

Table 5

Criteria for assignment of articles and materials to the proper NATO-UN hazard division

Class Division	Criteria
1.1	1. The articles or materials mass detonate 2. Propellants have a TNT equivalency greater than 10 percent.
1.2	1. The package or materials do not mass detonate 2. Fragments are produced.
1.3	1. The radiant heat flux is greater than 1.3×10^4 J/m^2/s beyond a 30 m radius. 2. The TNT equivalency is 10 percent or less.
1.4	1. The hazardous fragment and firebrand density is no more than one per 56 m^2 beyond a 30 m radius 2. The radiant heat flux is no more than 1.3×10^4 J/m^2/s beyond a 30 m radius.

The hazard classification procedures of TB700-2 have been criticized by many people intimately involved with classification and hazards such as Brown (Ref 7), Settles (Ref 8), Voeglein (Ref 9), Masten (Ref 10), and Pratt (Ref 11). Even supporters of TB700-2 such as Demberg (Ref 12), concede that there are many deficiencies in TB700-2 which should be corrected. The following discussion presents a summary of the problems associated with the scope, procedures and tests of TB700-2.

As was noted earlier, the scope of TB700-2 is rather limited. Many of the comments regarding TB700-2 are related to its limited scope:

- The procedures do not include provisions for classifying in-process materials, pyrotechnics, slurries or liquids.
- The procedures do not provide for classifying any material as Class 1, 3, 4, 5, 6 -- only as either Class 2 or Class 7.
- The procedures do not include provisions for tests relevant to such expected stimuli as electrostatic discharge.
- The tests of Chapter 3 are based on extremely small samples which do not always scale up to actual sizes and quantities.
- The procedures of Chapter 4 do not require the collection of data regarding blast overpressure, impulse, heat flux, firebrands, or fireball diameter. Fragment recovery and mapping is required; however, test procedures are not specified.
- No criteria are provided to allow the tests of Chapters 4 or 5 to be used in classifying the materials or end items.
- The procedures to be used in the tests of Chapters 4 and 5 are not specified in sufficient detail to insure that the methods are standardized and that the results are meaningful.

- The tests specified for determining the DoD transportation class are identical to those required by DOT. However, there are differences in the interpretation of results which often cause the DoD assigned classification to differ from DOT's despite being based on identical test results.

In addition, the following comments relate to specific tests:

- The impact test was originally intended for use with powders that could sift out of boxes during shipment and should not be used to determine the impact sensitivity of anything larger than these powders.
- The impact test is not reproducible and does not scale up to larger sizes.
- The sample sizes for the tests in Chapter 3 are unrealistically small.
- The impact test is impossible to use with granular solids or similar nonhomogeneous materials, where it is difficult to get uniform samples.
- The ignition and unconfined burning tests often show only that a material burns as it was designed to burn (especially pyrotechnics).
- The tests of Chapter 4 should specify how and where the central test item should be primed.
- Many of the tests indicate that the material either did or did not detonate at a high velocity. They do not differentiate between materials that burned or did not react and those which deflagrated, exploded or detonated at a low velocity.

All of these comments are valid criticisms of TB700-2. The primary modifications to TB700-2 that must be made to overcome these deficiencies are:

- Conversion to UN-NATO hazard class designations.

- Specification of more realistic tests to determine the sensitivity and effects of the tested materials.
- Expansion of the procedure to include all forms of explosives and propellants -- especially in-process materials, pyrotechnics and slurries.
- Specification of instrumentation and procedures for the collection of effects data which include fragmentation, blast overpressure and impulse (TNT equivalency), radiant heat, fireball diameter, and firebrands.
- Specification of criteria for use in assigning a material to a hazard classification. The criteria should be in numerical terms whenever possible (e.g., TNT equivalency greater than 10%)

Quantity Distance Requirements

The quantity-distance requirements for DoD Classes 1 to 7 are given in Fig 2. As can be seen in the figure, the quantity distance requirements may seem conflicting as a lower class often has a greater specified distance than a higher class. This is especially noticeable on the left side of the figure when comparing the required distances for Classes 3 to 6 with Class 7. The reason for this paradox is that the hazard of Class 7 materials is a function of mass (or amount of material), while the hazards posed by materials in Classes 1 and 3 to 6 are independent of mass. Figure 2 depicts inhabited building distances. According to DoD 4145.26M (Ref 4), "these distances are the minimum permissable distances between an inhabited building and an ammunition or explosives location. Inhabited building distances are also used for protection of administrative areas, adjacent operating lines and for other exposures within an establishment. Inhabited building distances also shall be provided between ammunition and explosives locations and plant boundaries." It further states that:

1) "Inhabited building distances as set forth in this part protect buildings against 'substantial' structural damage..."

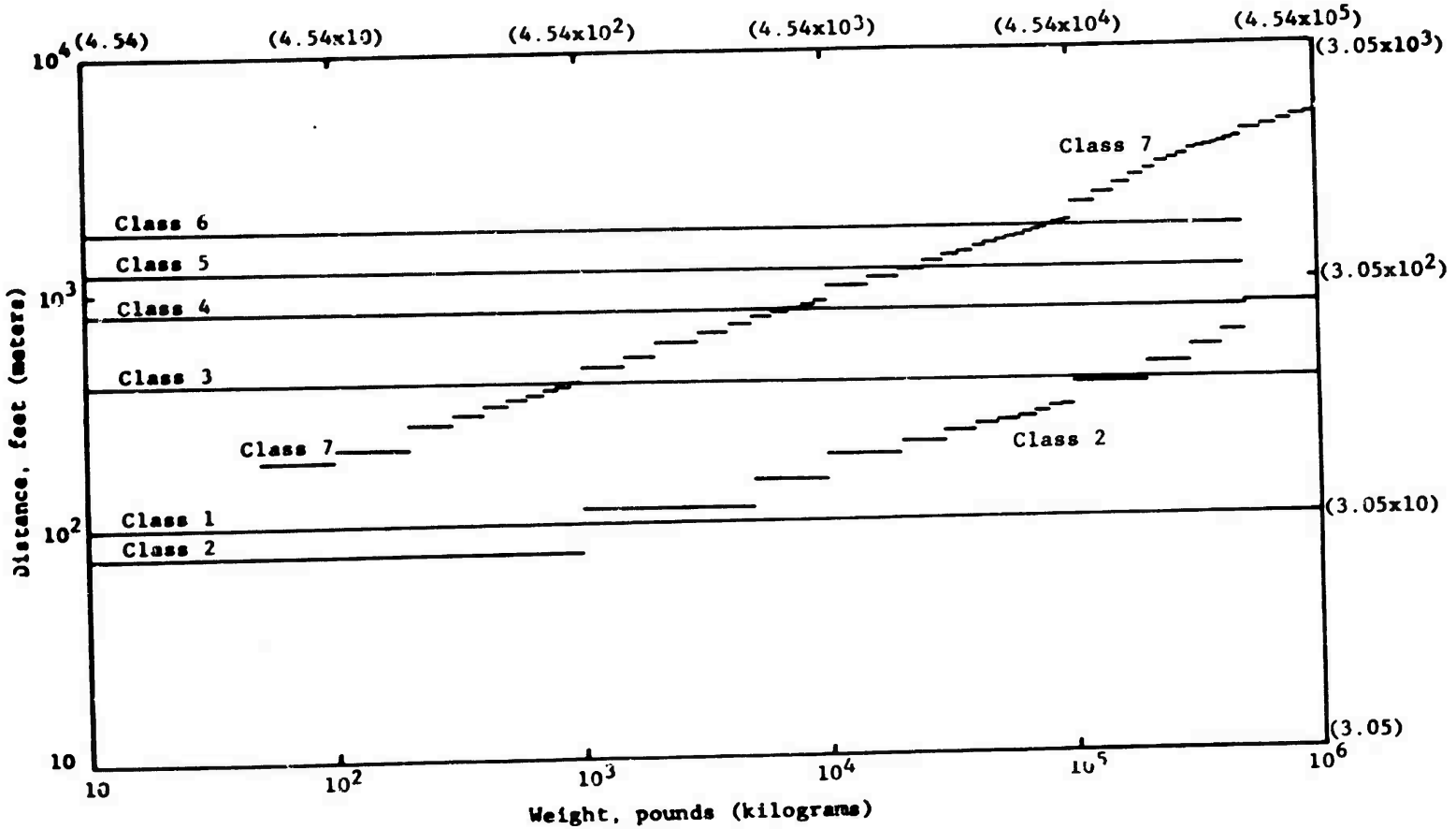


Fig 2 Inhabited building quantity-distance requirements for DoD hazard classes

2) "Inhabited building distances do not provide protection against glass breakage or injury to personnel from glass breakage... The inhabited building distances...for Class 7 are based on damage from blast effects; however, they do provide a high degree of protection except for small quantities where the fragment hazard may be more severe than the blast hazard. Inhabited building distances for ammunition and explosives which are not mass detonating are based on the most severe hazard involved."

Thus, fragments are supposed to be included as a hazard for Classes 3 to 6 but not for Class 7, and a large potential for injury to plant personnel and the public in adjacent areas is tolerated. However, change 1 of AMCR 385-100 (Ref 5) states that although the inhabited building distances are based on blast damage: "For fragment producing Class 7 items,...inhabited building distances will be increased, as necessary, so that the density of hazardous fragments will not exceed one in 600 square feet. A hazardous fragment is one having impact energy of 58 foot-pounds or more. In this connection, as supporting data become available, fragment producing Class 7 items will be grouped additionally into the distance zones used for non-mass-detonating items.

The quantity-distance requirements for liquid propellants, according to CPIA/194 (Ref 2) are shown in Fig 3. Distance requirements for materials in Group IV to protected inhabited buildings are the same as for DoD Class 7, taken from AMCR 385-100 (Ref 5). (These distances, for large quantities, are greater than those given in the later Change 1 of AMCR 385-100).

Distance requirements for materials in Group III are given in CPIA/194 for both unprotected and protected (barricaded) inhabited buildings. Those for unprotected buildings are based on predicted fragment distances from equipment experiencing a vapor phase explosion. Those for protected buildings are based on thermal considerations derived from Bu Mines Report 5705 (Ref 13).

For materials in Groups I and II there is no distinction made between protected and unprotected inhabited buildings. Distances for Group II materials were arbitrarily taken as 3/4 of those for protected inhabited buildings in Group III. Distances for Group I materials were taken similarly as 1/2 those in Group II.

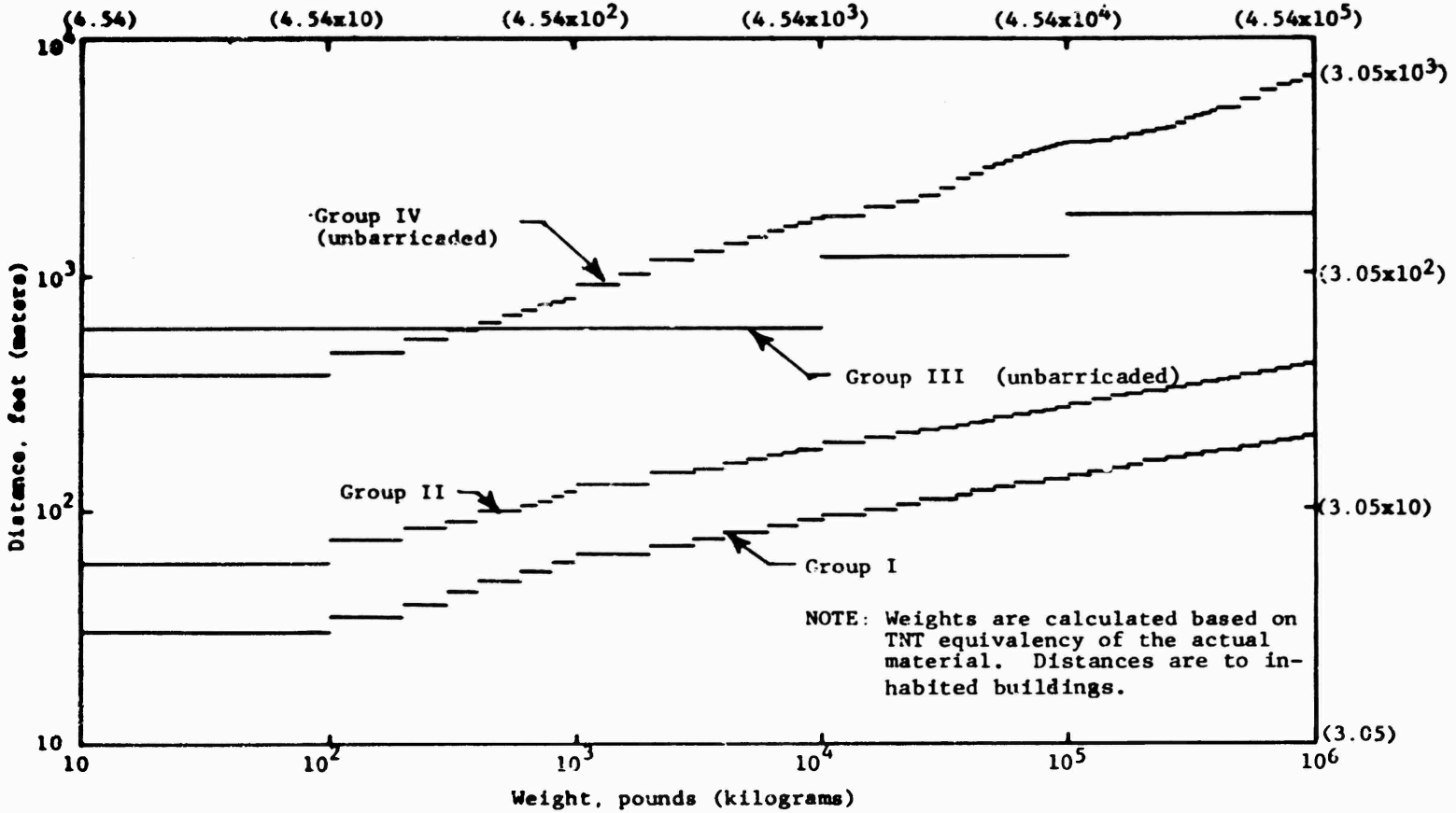


Fig 3 Inhabited building quantity-distance requirements for liquid propellants based on CPIA hazard classifications

There is a crossover between the Group III and Group IV distance requirements. This is probably because Group III considers fragments while Group IV is based solely on blast overpressure.

The quantity-distance requirements for materials classified according to NATO-UN categories are confusing. For materials in UN Classes 1.1 and 1.3, the distances are either those specified in DoD 5154.4S (Ref 14) or those based on the results of the hazard classification tests. For materials in Class 1.2, the distances are those derived from the hazard classification tests. For materials in Class 1.4, the distances are those given in DoD 5154.4S.

Minimum distance criteria for the several NATO-UN classes of materials are given in Table 6. There are a number of shortcomings with the existing quantity-distance requirements which are related to the hazard classification schemes. For the following, we will limit the discussion of quantity-distance (QD) requirements to those of DoD Classes 2 and 7 and CPIA Group IV materials. Classes 2 and 7 were chosen as they are the only classes applicable to in-process material, and as they are the only classes specifically mentioned in TB700-2. Group IV was chosen as its quantity distance requirements were exactly those of Class 7 prior to 1971.

To facilitate the discussion, power curves were fitted to the midpoints of the quantity distance requirements (for inhabited buildings). The resultant equations were:

$$R = 35.0\ W^{0.356} \qquad \text{Class 7} \qquad (1a)$$

$$R = 9.21\ W^{0.313} \qquad \text{Class 2} \qquad (1b)$$

$$R = 107\ W^{0.302} \qquad \text{Group IV} \qquad (1c)$$

Here, R is the required distance in feet to an inhabited building from W pounds of material. The power curves for Class 7 and Group IV are depicted in Fig 4.

The normal relation for a fireball of radius FR feet produced by W pounds of explosive material is

$$FR = 5W^{1/3} \quad \text{(Ref 2,15,16)} \qquad (2)$$

Table 6

Minimum UN-NATO distance criteria

NATO-UN Class	Minimum Distance Criteria
1.1	The distance at which the density of hazardous fragments for personnel in the open exceeds one per 600 sq ft (56 m^2) or a distance of 1250 ft (380 m) whichever is greater.
1.2	The distance at which the density of hazardous fragments for personnel equals one per 600 sq ft (56 m^2) within the first 20 minutes after detonation of the first test item (personnel protection)
1.3	The distance at which the density of one per 600 sq ft (56 m^2) of fragments (58 ft-lb (79J)) and/or firebrands (3.72×10^7J) exist at the conclusion of the test (protection of structures)
1.4	The distance at which a heat flux of 0.3 cal/cm^2/s (1.3×10^4 J/m^2/s) is recorded, or the distance at which firebrand (3.72×10^7J) density equals one per 600 sq ft (56 m^2) whichever is greater.

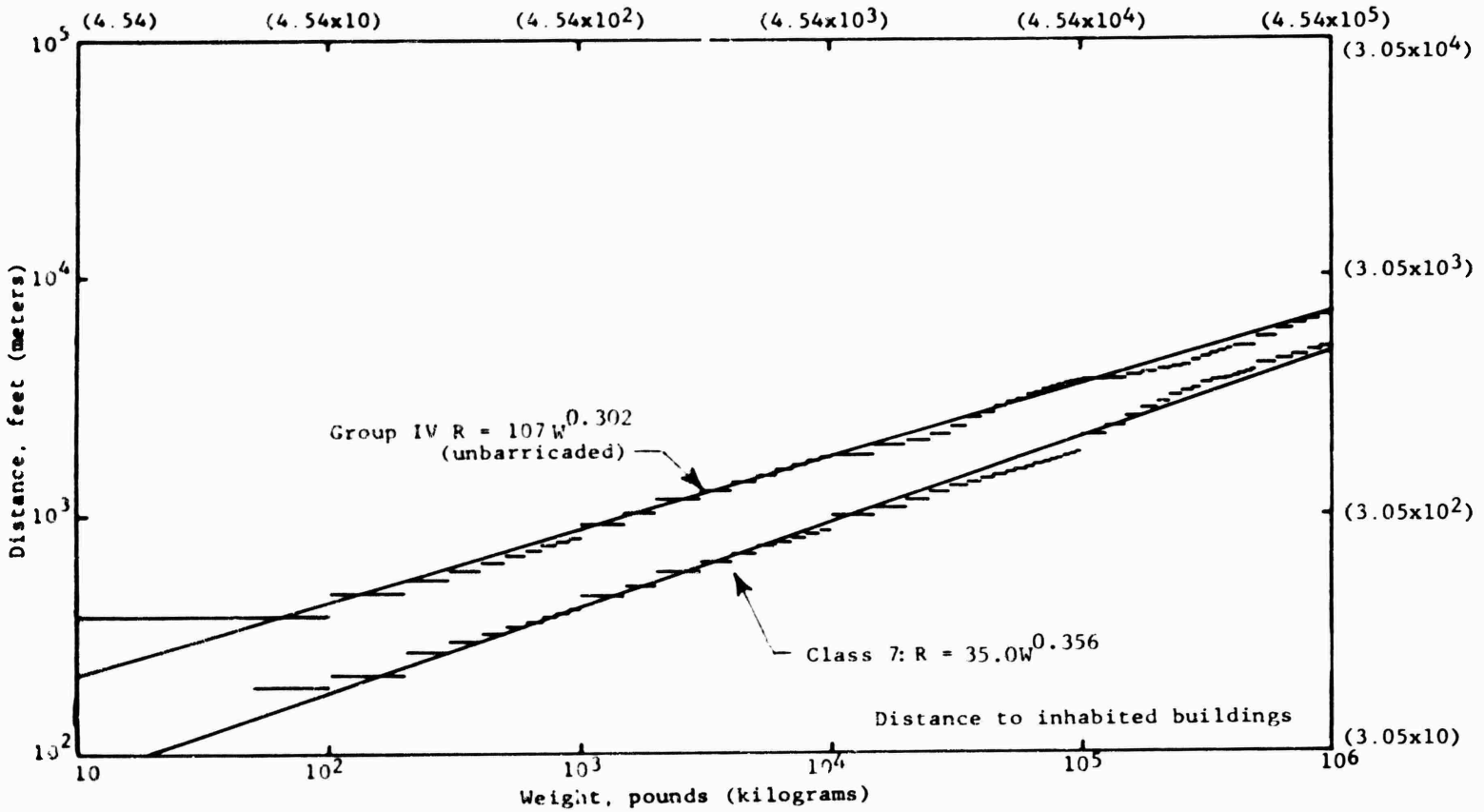


Fig 4 Power curve fit for group IV and class 7 quantity-distance requirements

Comparison of Equations 1a, 1b and 1c with Equation 2 shows that the QD requirements will always place an inhabited building outside of the fireball. However, the effects of winds and other environmental conditions, and the effects of asymmetrical fireballs, are not considered in Equation 2, so that the QD requirements for Class 2 may be only marginally effective. This asymmetry was illustrated in a large scale black powder test where 1450 kg of black powder produced an elliptical fireball with axes of 24 m amd 48 m (Ref 17). Only the shorter 24 m axis was close to the 22 m predicted by Equation 2. It should be noted that the fireball equation for the black powder tests was

$$FR = 5.16\ W^{0.303}$$

which is very close to Equation 2. The radiant heat flux could be in the range fo 1.3×10^5, 2.5×10^5 or 3.8×10^5 J/m^2 or sufficient to produce first, second, or third degree burns (Ref 10). At the least, it could easily exceed the 6.3×10^4 J/m^2 recommended as the maximum exposure to the public (Ref 18).

In terms of fires involving pools of liquid fuels or solid fuels, the quantity-distance requirements may be excessive (Ref 19).

Due to the manner in which Class 2 is defined and the materials it includes, low velocity detonations and explosions as well as fires can be expected (Ref 20). Thus, some blast overpressure can be expected. A power curve was fitted to the overpressure data of Kingery and Pannill (Ref 21). In the range of 0.014 to 1.193 psi (9.6×10 to 1.3×10^4 Pa), this equation is

$$P = 198.765\lambda^{-1.384} \qquad (4)$$

where P is the overpressure in psi and λ is the scaled distance ($\lambda = R/W^{1/3}$, R= distance (ft), W = weight (lb)). If we use E to denote TNT equivalency, Equations 1b and 4 can be combined to arrive at

$$P = 198.765\ \left(\frac{9.213\ W^{0.313}}{(EW)^{0.333}}\right)^{-1.384} \qquad (5)$$

or $$P = 9.196\ E^{0.461}\ W^{0.0277}$$

Assuming that $W^{0.0277} = 1$, we have

$$P = 9.196\ E^{0.461} \qquad (6)$$

If E = 1, the pressure at the Class 2 quantity-distance requirement is 9.20 psi (6.34 x 10^4 Pa). Of course, E will never equal 1 as that implies a high velocity detonation and the material would be Class 7. The pressures at other equivalencies are tabulated in Table 7.

Table 7

Pressures at the class 2 quantity-distance requirement for various TNT equivalencies

Equivalency	Pressure psi (Pa x 10^4)
1.00	9.20 (6.34)
0.90	8.76 (6.04)
0.80	8.30 (5.72)
0.70	7.80 (5.38)
0.60	7.27 (5.01)
0.50	6.68 (4.61)
0.40	6.03 (4.16)
0.30	5.28 (3.64)
0.20	4.38 (3.02)
0.10	3.18 (2.19)
0.05	2.31 (1.59)
0.01	1.10 (0.76)

Thus, to get down to 1.0 psi (7 x 10^3 Pa) at the required distance, a TNT equivalency of less than 1 percent is necessary. This is probably smaller than the TNT equivalencies of many Class 2 materials. As 1 psi (7 x 10^3 Pa) is generally accepted as the criterion for structural damage, a Class 2 material with a TNT equivalency of 1 percent or greater will exceed 1 psi (7 x 10^3 Pa) at the required Class 2 distance. In order to get down to 0.10 psi (7 x 10^2 Pa) to minimize glass breakage, an equivalency of 0.006 percent or less would be required.

The UN-NATO criteria specify that a material with a TNT equivalency of 10 percent or less is in Class 1.3. Thus, according to Tables 7 and 5, a Class 1.3 material would exhibit an overpressure of 3.18 psi (2.19 x 10^4 Pa) at DoD Class 2 distances. However, as the NATO Class 1.3 materials have quantity-distance requirements which are based on actual test results, this problem is minimized.

The root of the quantity-distance requirements for Class 7 materials comes from the old American Table of Distances (Ref 20).

This table was derived from a study of 117 accidental explosions. The criteria used in deriving the quantity-distance relations were the outer limit of structural damage. The equation that was derived was

$$R = 34.75\ W^{1/3} \text{ (Ref 20)} \tag{7}$$

As can be seen, this equation is almost identical with Equation 1a for Class 7 materials. The table does not include the effects of damage due to either fragments or glass breakage.

The accident data used in deriving the American Table of Distances are plotted in Fig 5. Of the 116 accidents shown in the figure, only 4 (3 percent) are above the 0.5 psi (3.5×10^3 Pa) line and 24 (21 percent) are above the 1.0 psi (7×10^3 Pa) line. It should be noted that of the 116 accidents, only 21 were of a material that is currently considered to be high explosive (nitroglycerine). The remainder were accidents involving materials such as black powder, gelatin, and dynamite. Of the 21 accidents involving nitroglycerine, none were of more than 5.44×10^3 kg. As the TNT equivalency of black powder, gelatin, and dynamite is less than one, the accidents underestimate the range at which damage occurrs--especially in terms of today's explosives where TNT equivalencies in excess of one are common.

If we combine Equations 1a and 4, we get

$$P = 198.765\ (34.996\ \frac{W^{0.356}}{W^{0.333}})^{-1.384} \tag{8}$$

or

$$P = 1.448\ W^{-0.0315}$$

assuming a TNT equivalency of one. If we further assume that $W^{-0.0315} \approx 1$ we have

$$P = 1.45 \tag{9}$$

This would show that the Class 7 quantity distance requirements are aimed at attaining a pressure of 1.45 psi (1×10^4 Pa) at the specified distance. Actually, the pressure ranges from 0.93×10^4 Pa at 4.54 kg to 0.65×10^4 Pa at 4.54×10^5 kg. Wilton (Ref 22) claims that at the Class 7 distances, damage of 25 percent should be expected to inhabited buildings and that extensive glass breakage will occur.

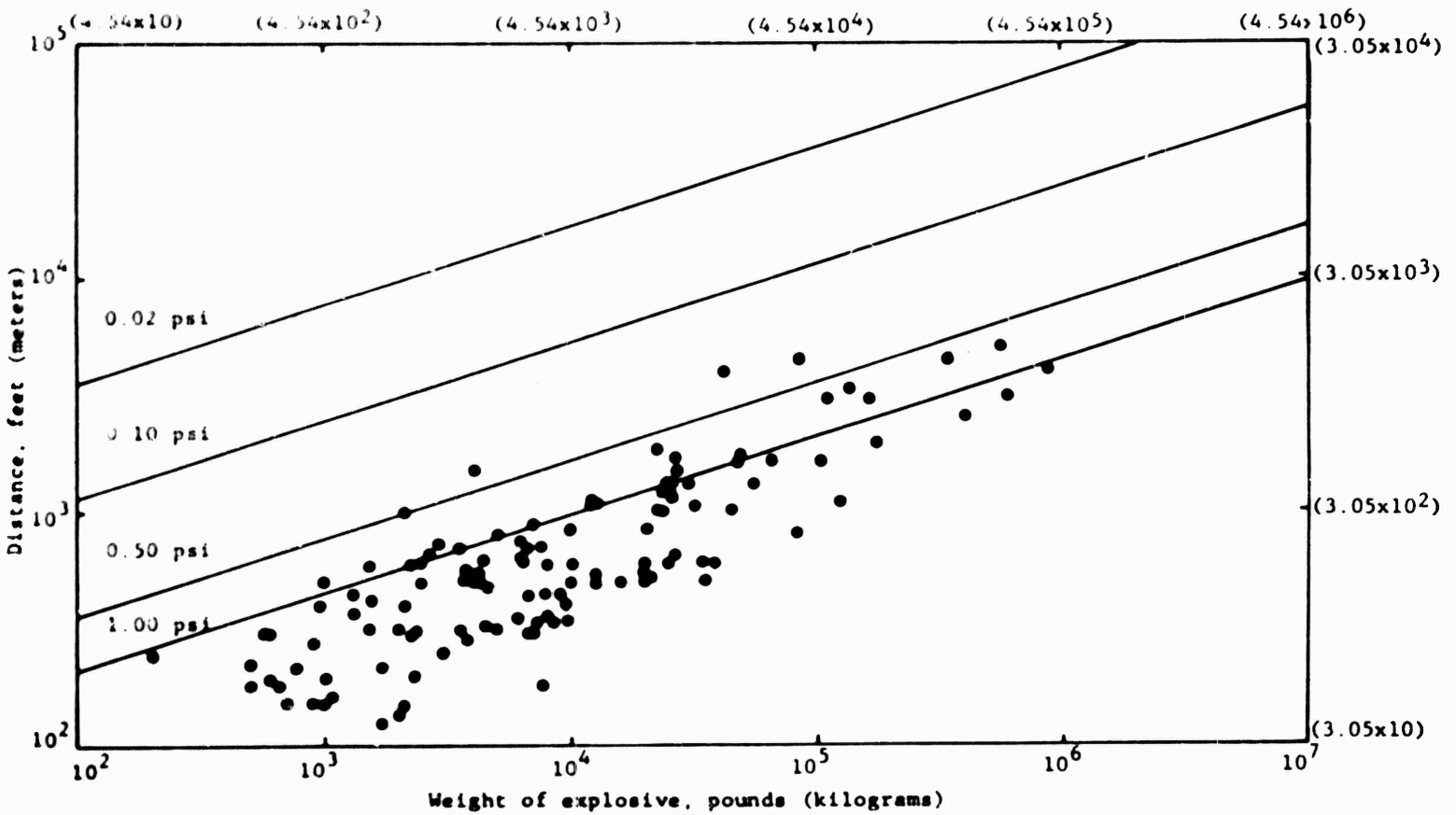


Fig 5 Distance at which structural damage occurred for the accidents on which the American table of distances was based

As was mentioned earlier, fragments are not considered in the Class 7 quantity-distance criteria. Fragmentation, however, should be expected in virtually all accidental explosions. Figure 6 shows the range of fragments for the accidents used to derive the American Table of Distances, and Fig 7 shows the range of fragments from more recent accidents. As can be seen in the figures, many of the accidents resulted in fragments being thrown well beyond the 1.0 psi (7×10^3 Pa) criterion for structural damage. In fact, relative safety from fragments is not attained until the 0.10 psi (7×10^2 Pa) line. However, this cannot be interpreted literally as the distances are for the farthest fragments and not the distances at which the fragment density reaches one per 600 sq ft (56 m^2). The fragment plots and the Class 7 distances are combined in Fig 8.

The breakage of glass has the potential for causing numerous and often severe injuries. Glass breakage is normally expected to occur at pressures down to 0.2 psi (1.4×10^3 Pa). This is shown in Fig 9 which shows the distance at which glass breakage occurred for the accidents included in the American Table of Distances. Glass breakage occurred at pressures under 0.02 psi (1.4×10^2 Pa).

The Group IV quantity-distance requirements were those of Class 7 prior to 1971. These requirements, however, call for a lower pressure than the current Class 7 requirements. Combining Equations 1c and 4, we get:

$$P = 0.308\ W^{0.0432} \qquad (10)$$

The pressure at the required unbarricaded distances ranged from 2.34×10^3 Pa at 4.54 kg to 3.86×10^3 Pa at 4.54×10^5 kg of material. The Group IV quantity-distance schemes include the TNT equivalency of the material.

None of the classification schemes is concerned with impulse, the positive integral of the pressure-time curve. This is probably due to the fact that structural damage correlates better with blast overpressure than with impulse at comparatively long distances. However, this is most unfortunate as impulse can produce hazards, such as knocking down people (the blowdown problem), independently of pressure. A primary example of impulse damage is that caused by a FAE (fuel-air explosive), which has a low blast overpressure but a high impulse. As the armed forces are turning to the concept of a blast enhancement (increased impulse) in new weapon and explosive designs, impulse increasingly will be an important factor.

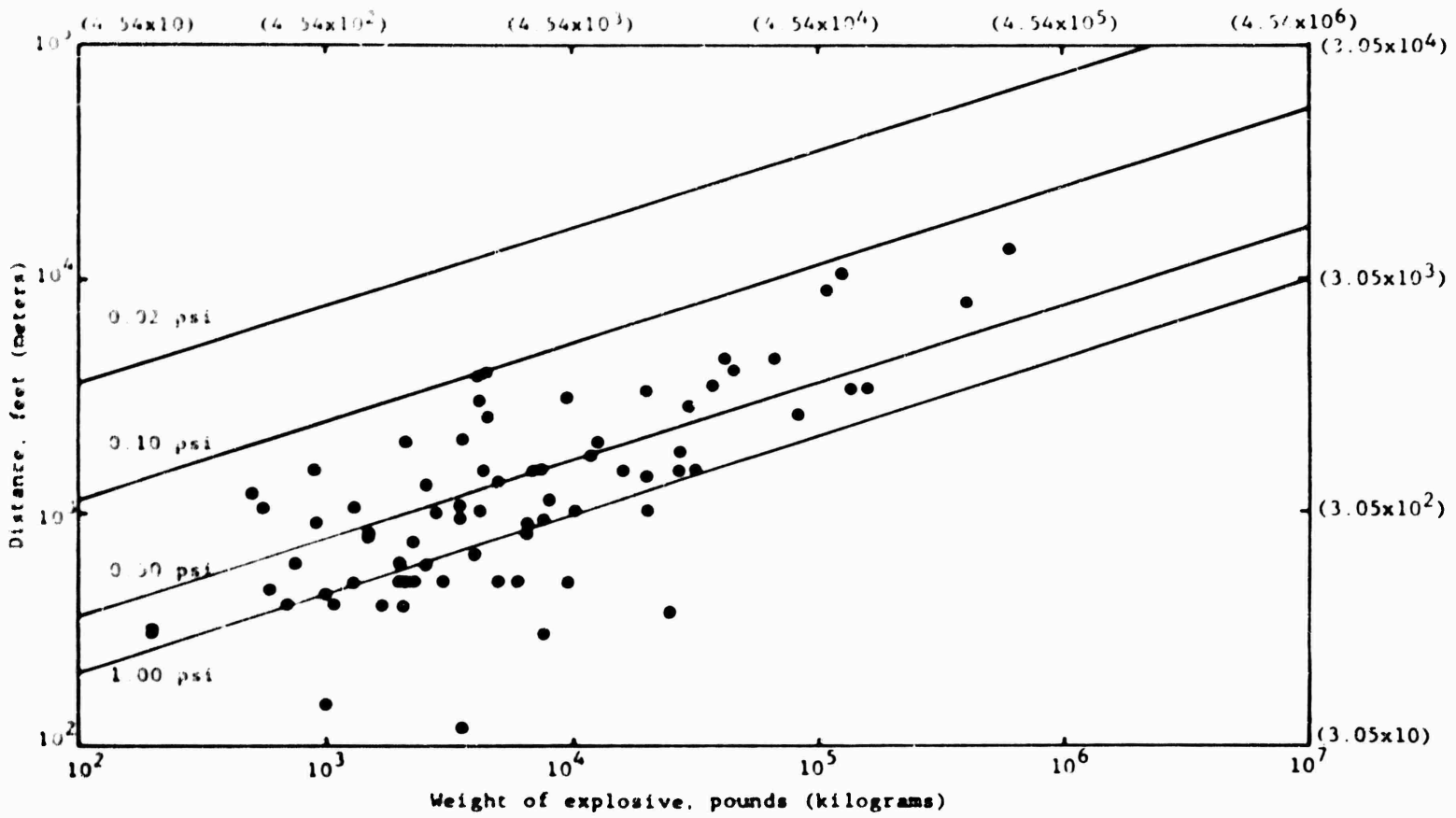


Fig 6 Fragment distance of the accidents which were used as a basis for the American table of distances

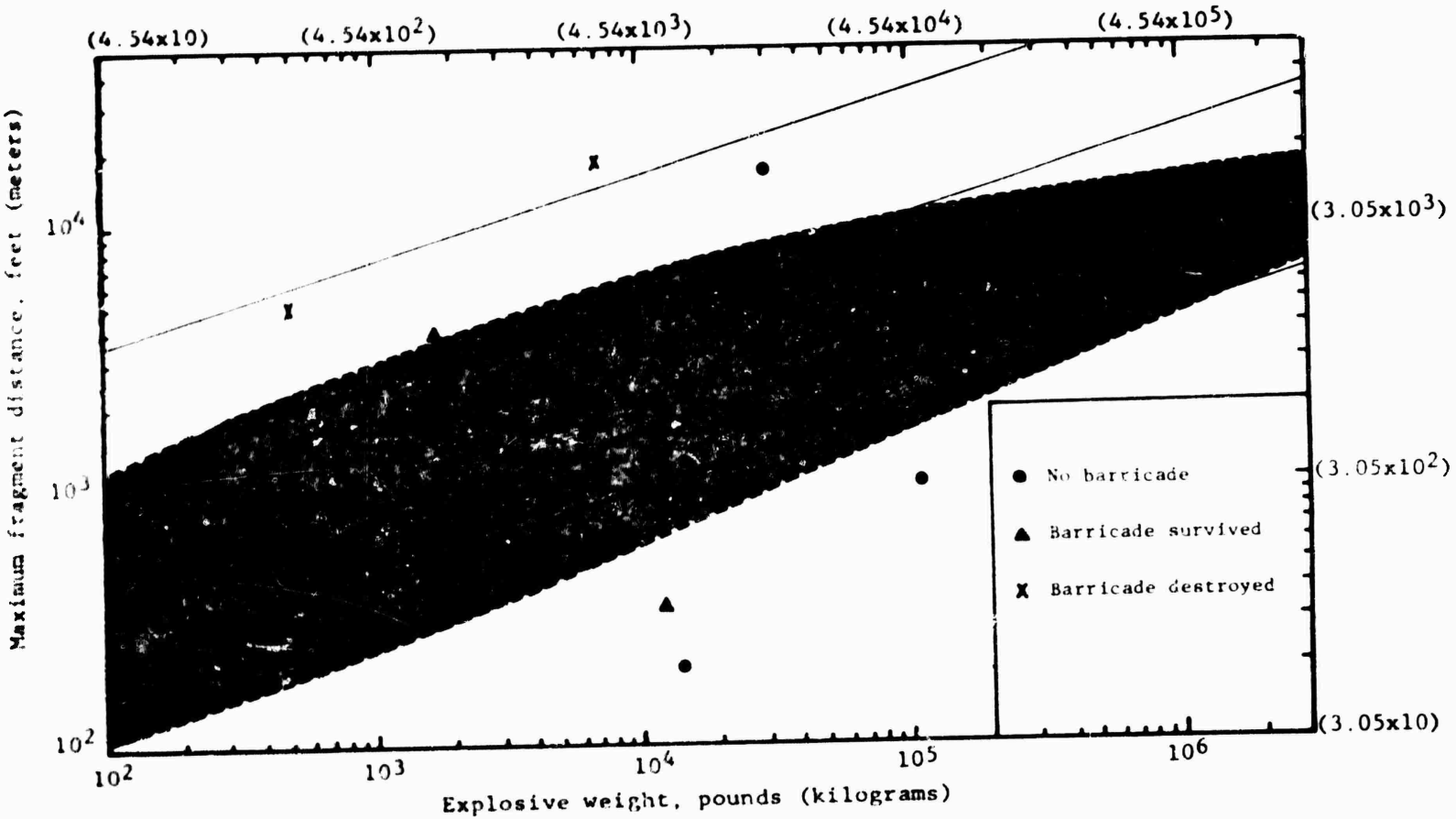


Fig 7 Maximum fragment distances for accidental high explosive surface bursts (Ref 2)

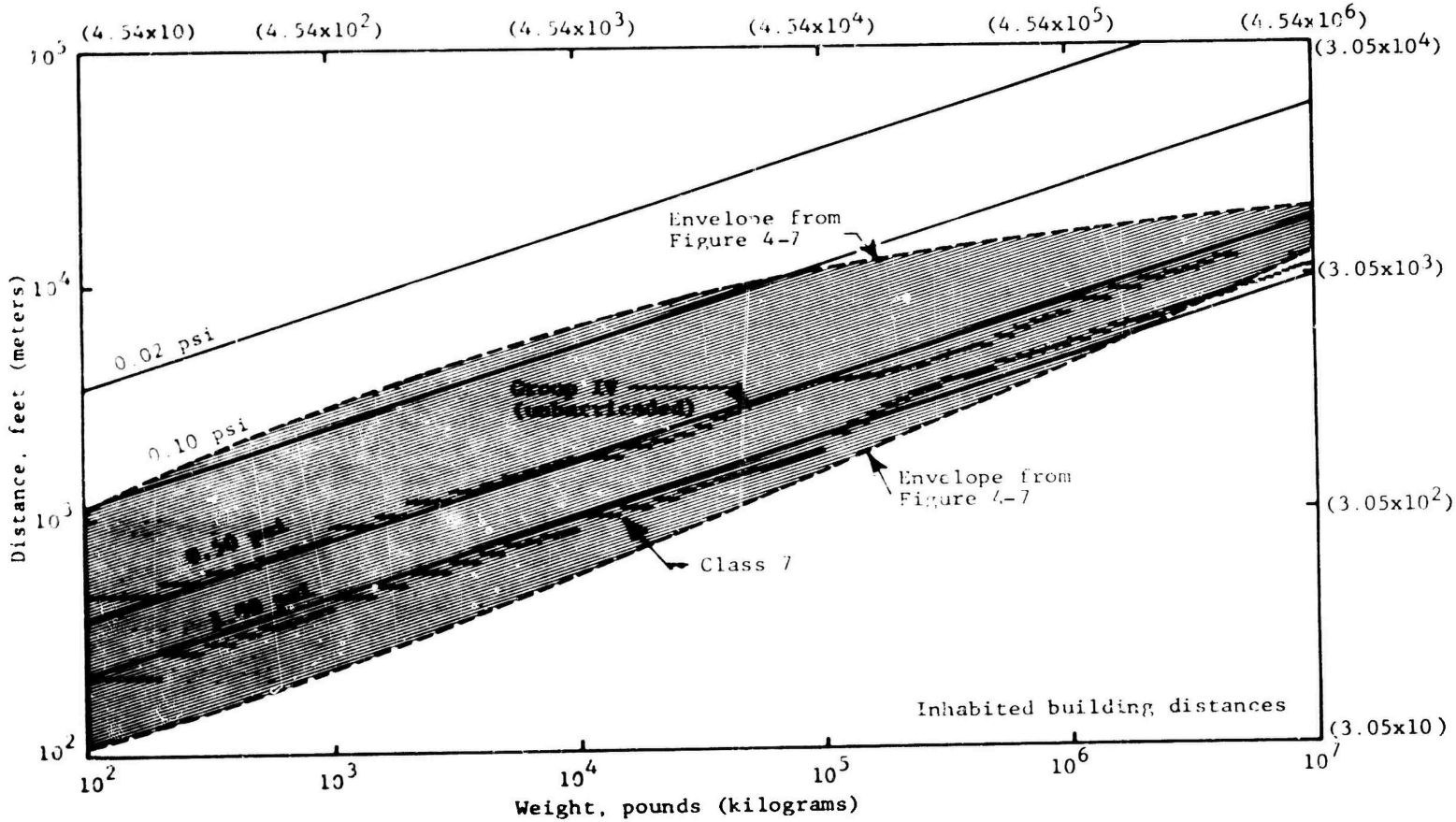


Fig 8 Relationship between class 7 and group IV quantity-distance requirements, free field blast overpressure and maximum fragment distances for accidental high explosive surface bursts

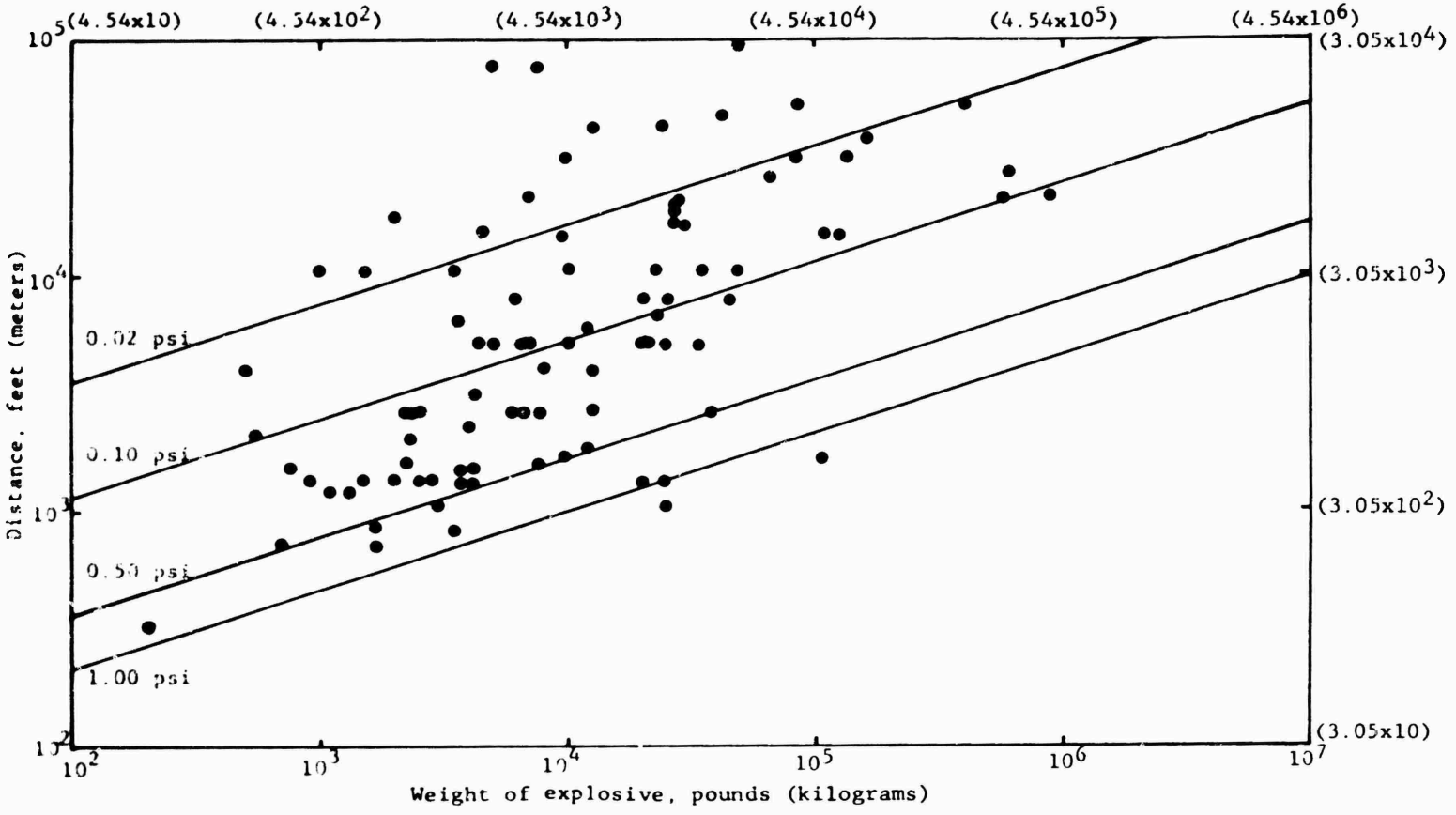


Fig 9 Distance at which glass fragmentation occurred in the accidents used as a basis for the American table of distances

The current DoD classification procedures and the UN-NATO procedures are not directly concerned with TNT equivalency. As the UN-NATO procedures utilize experimental data for setting quantity-distance requirements, the non-use of TNT equivalency is not too important. However, for the DoD classification procedure, where the distances are taken from quantity-distance tables, significant errors can occur. If the actual TNT equivalency is greater than one, the table distance is too short; while if the TNT equivalency is less than one, the table distance is too long. Thus, either the required safety is not provided or more distance than necessary is being required.

All the discussion in this section has been based on the distance to an inhabited building. The distances to public highway/railroads are often less than the inhabited-building distances (e.g., Class 7 material). The main reason given is that as trains, cars and trucks are mobile, the probability that such a vehicle would be endangered is less than the probability that an inhabited building would be endangered. This presumes that the vehicular traffic density is low and always will be low. A secondary justification is that the structural strength of such vehicles is greater than that of a building. This is probably true; however, if the vehicle's glass shatters and the driver loses control, the end effect is the same as if severe structural damage occurred. Breakage of the glass will occur in automobiles as can be seen in Table 8 which was copied from Ref 23. Furthermore, with the large surface area of vehicles, overturning trucks, lane changes, and derailing of railroad rolling stock could occur at modest impulse levels.

Table 8

Automobile window damage during Eskimo II tests (Ref 23)

Ground Range, ft	P_i, psi	Automobile			Windows Damaged	Extent of Window Damage
		Orientation	Number	Description		
730	1.2	Face-On	A1	Renault	None	None
			A2	Pontiac	Windshield Left Rear-Door Right Front-Door Right Rear-Door	Completely broken out Multiple fractures Completely broken out Multiple fractures
		Left Side-On	A3	Dodge* Station Wagon	Windshield Left Rear-Door (Side)**	Multiple fractures Completely broken out
			A4	VW	Left Door	Completely broken out
			A5	Peugeot	Left Front-Door Right Front-Door	Completely broken out Completely broken out
			A6	Chevrolet	Windshield Left Rear-Door	Multiple fractures Completely broken out
			A7	Dodge Fuel Truck	Left Door Left Vent	Multiple fractures Multiple fractures
1130	0.62	Left Side-On	A8	VW Bus	Windshield	Multiple fractures
			A9	Lincoln	Windshield	Multiple fractures
1700	0.41	Left Side-On	A10	Buick	None	None

* An anthropomorphic dummy was secured in the driver's seat of this station wagon by means of a lap seat belt.

** Analysis of the film record from the camera (402 frames per second) viewing this window indicated that the fragments had a mean velocity of about 11 ft/sec.

Note: There was no evidence that any of the automobile windows were broken by bomb fragments or crater ejecta rather than by the airblast itself.

DEVELOPMENT OF CRITERIA FOR EVALUATING THE SENSITIVITY OF IN-PROCESS MATERIALS

Survey of Accident Reports

The Department of Defense Explosives Safety Board (DDESB) acts as a repository for reports on accidents related to the manufacture and use of explosives and propellants. A trip was made to the DDESB to collect relevant accident data. As there was a large number of accident reports on file at the DDESB, only a sample of the accident reports was obtained. The sample was chosen to reflect the types of accidents which were of interest to this program.

The gathered accident data were compiled in tabular form. These tables give the DDESB report number, a description of the material involved in the accident, the estimated quantity of material, the number of injuries and fatalities that resulted from the accident, the component or portion of the process, the type of output, the fragment and glass breakage distances, and the probable cause of the accident as given in the accident report. The accidents were grouped in such a way that each table contains only data on one type of process operation. These tables are given in Appendix A.

Analysis of Accident Causes

The probable causes of the accidents summarized in the tables were generalized to fit into the following categories of stimuli:

- friction
- impact
- adiabatic compression
- electrostatic discharge (ESD)
- heating
- impingement

When several different causes were listed, more than one such category was applicable. Table 9 gives the percentage of each category of stimulus named as a probable cause in accidents within various process areas or operations. For example, 68 percent of the pressing incident reports named friction as one of the probable causes of the initiation. The percentages for a given process operation can total more than 100 percent

Table 9

Probable causative stimuli for accidents occurring in specific process operations

PROCESS OPERATION / PROBABLE CAUSITIVE STIMULI	PRESSING	MIXING	REACTOR	CONVEYING	DRYING	FILLING	SCREENING	MACHINING	PERCENT FOR ALL PROCESS OPERATIONS
Friction	66	86	17	80	50	74	75	95	59
Impact	49	29	8	20	28	35	25	21	32
Adiabatic Compression	35			20				5	11
ESD		12			6	22	50	5	8
Heating	11	6	83	60	61	17		11	26
Impingement				20		8	25		3
PERCENT OF ALL OPERATIONS	27	13	9	4	13	17	3	14	

as more than one stimulus may be named as the probable cause for a single accident. The distribution of accidents by process operation and the frequency by probable stimuli for all process operations are also given.

The probable causes of an accident differ from material to material. This is related both to the material's properties and the types of process operation. Figure 10 presents the data of Table 9 in a more graphical format. Figures 11a and 11b present similar information for secondary explosives and propellants. It is obvious that differences in the probable accident causes (stimuli) exist. For instance, adiabatic compression is more of a problem for propellants in pressing operations than for secondary explosives. This is attributed to the presence of solvents in the propellants being pressed. The data were insufficient, however, to allow the preparation of a similar table for primary explosives.

Data such as those given in Fig 10 are quite useful because they indicate the types of stimuli most likely to cause an accident. As such, they also indicate the areas in which sensitivity testing should be required. It is apparent that friction and impact are the most commonly given causes of an accident, followed closely by heating and ESD. These are the most important stimuli. They are also the primary causes of maintenance accidents which can occur in any process operation. Thus tests to determine the sensitivity of all materials to friction, impact, ESD and heating are imperative. In certain process operations or material conditions, additional sensitivity tests such as for adiabatic compression, impingement, or ESD in dusts may be desirable.

Analysis of Accident Consequences

The DDESB accident reports also were analyzed to determine the correlation between process operations, material types, and accident consequences. Although this is not related to sensitivity, the results are enlightening and the method of analysis is the same as that described in the previous section. The results of the analysis are reviewed here briefly. For this analysis, the accidents were broken into two categories:

- Accidents resulting in an explosion of any type (i.e. detonation, deflagration, fire/explosion, etc.)
- Fires, only

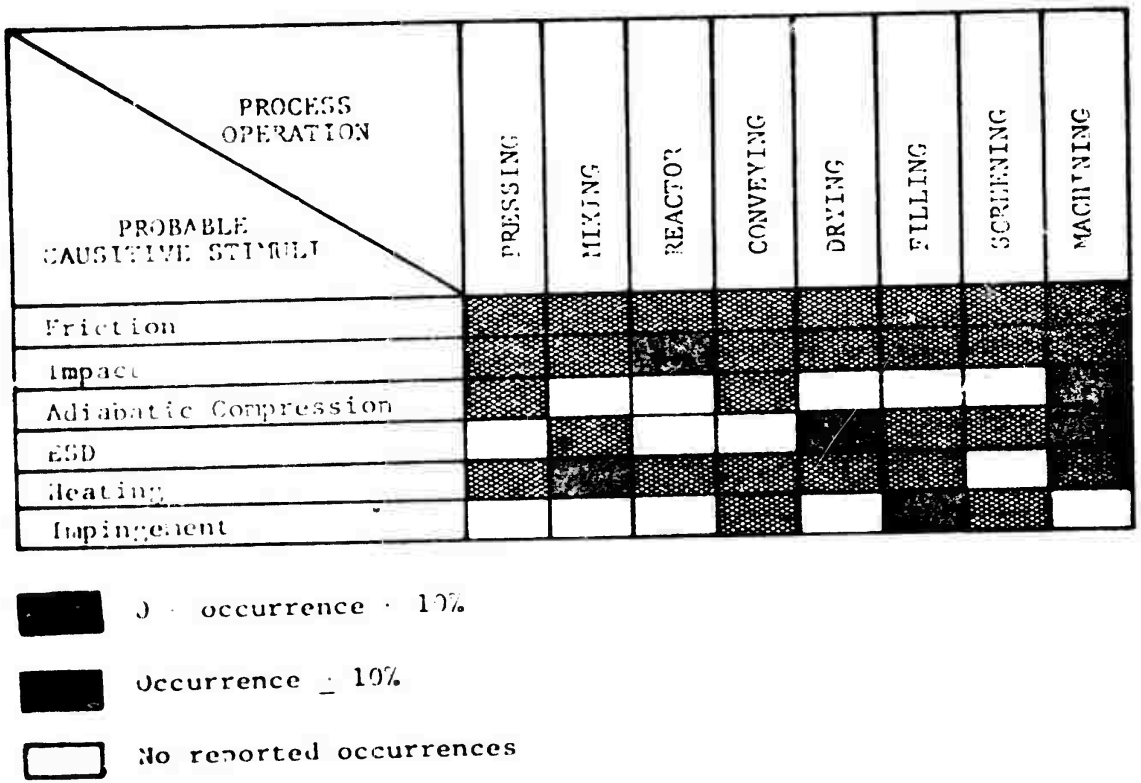
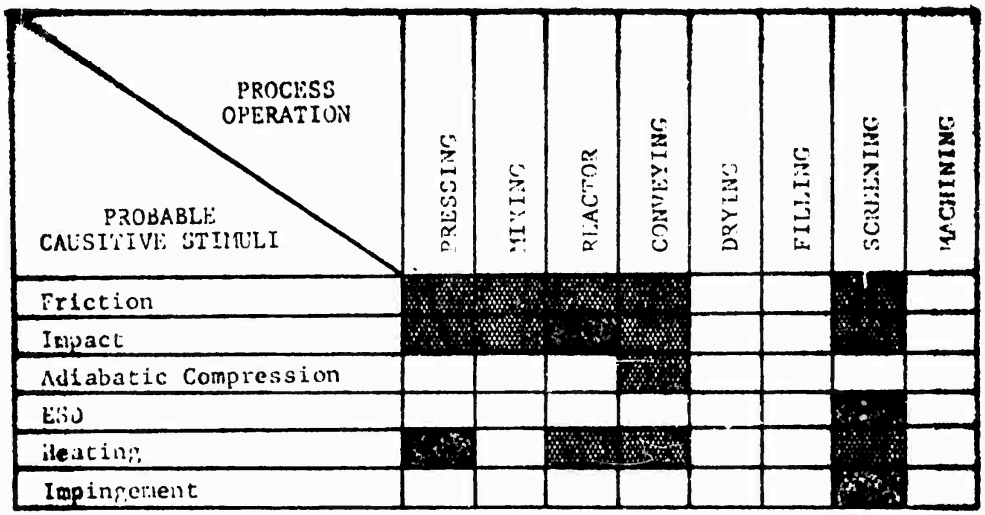


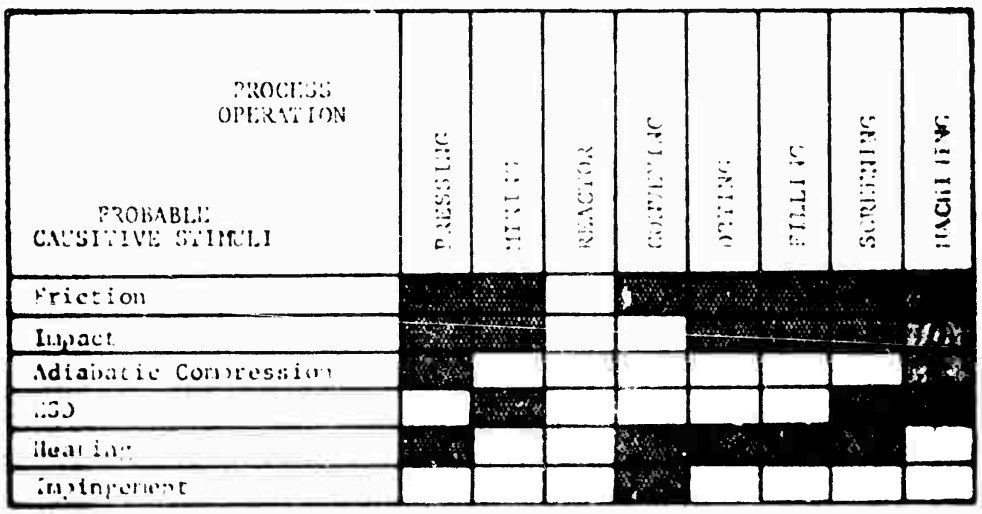
Fig 10 Probable causative stimuli for accidents occurring in specific process operations (all materials)



a. Secondary Explosives

0 - occurrence 10% No reported occurrences

Occurrence ≥ 10%



b. Propellants

Fig 11 Probable causative stimuli for accidents occurring in specific process operations for two types of materials

The first category includes all cases involving blast and/or fragment hazard, whereas the second category includes incidents resulting only in fire and thermal radiation hazards. The results of the analysis are presented in Table 10 and Fig 12.

As an example of how the data in Table 10 should be interpreted, 93 percent of the sampled accidents of secondary explosives in a reactor operation resulted in explosions. The data base in some areas, particularly for primary explosives and black powder, was scant. Therefore, a larger data base could significantly alter the results. The analysis which was conducted does indicate that most process accidents (ignitions) result in explosions. Propellants are somewhat less likely to be involved in an explosion than primary explosives, secondary explosives, or black powder. Apparently primary explosives, if ignited, are almost certain to explode, probably because their critical dimensions are smaller than the dimensions of the process vessel.

Development of Sensitivity Criteria

The accident data along with material sensitivity data from various literature sources (Refs 24 to 38), were used to develop criteria for evaluating the sensitivity of in-process materials.

The initial step was to review the accident data and to determine the probable cause(s) of each accident. These probable causes were usually given in the accident report. However, when the probable causes were not given, they were assumed to be the same as those most frequently responsible for similar accidents in known instances, as previously summarized in Fig 10. For instance, when the probable causes of an accident involving a mixing operation were unknown; friction, impact and ESD would be used (see Fig 10). The exact material involved in each accident also was identified.

Next, for each accident, data were compiled on the material's sensitivity to ignition by the various stimuli. Occasionally either the material was not described in sufficient detail to allow compiling sensitivity data (e.g. - double base propellant) or the sensitivity data were unavailable. In these cases, the accident was not considered.

Table 10

Accident effects analysis (numbers are percent of sampled DDESB accidents resulting in explosions, fire/explosion or deflagrations)

Material type / Process operation	Primary explosive	Secondary explosive	Black powder	Propellants	All materials
Pressing	100	100	100	79	89
Mixing	100	96	100	71	82
Reactor	-	93	100	50	90
Conveying	-	87	100	100	91
Drying	100	89	100	78	88
Filling	100	79	50	64	72
Casting	100	64	100	100	73
Screening	100	0	-	100	60
Machining	100	67	82	64	72
All Operations	100	88	88	73	83

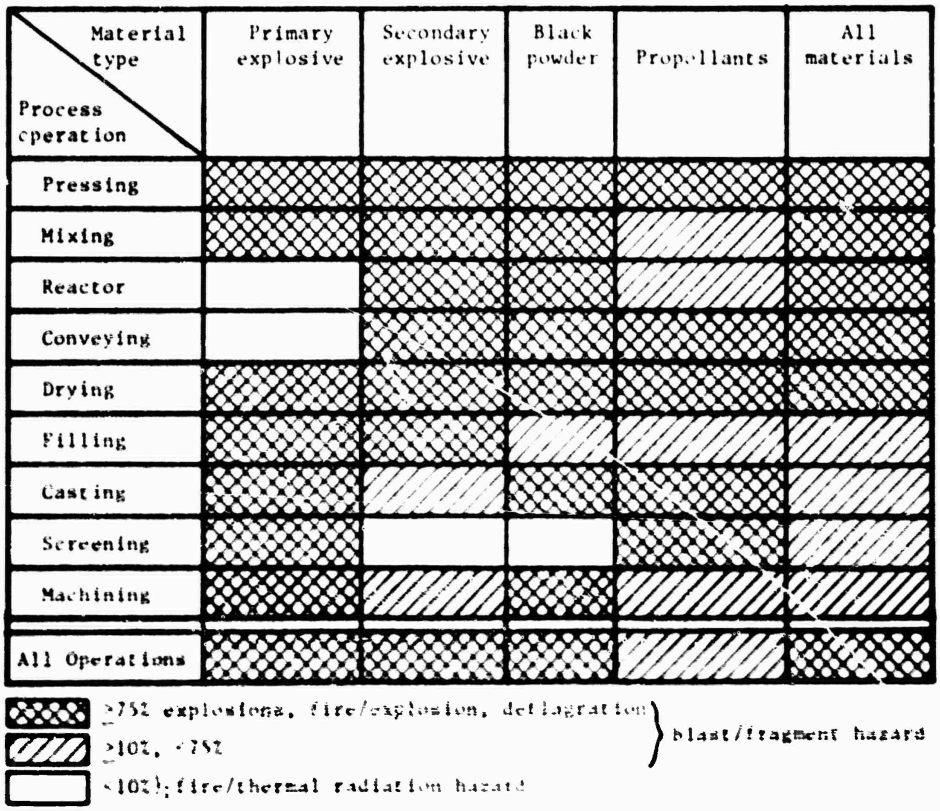


Fig 12 Accident effects analysis

Finally, the mean and standard deviation of the ignition sensitivities were calculated using different sample populations. The populations most frequently used were:

- Population 1 - All accidents in which the particular stimulus of interest was a probable cause.
- Population 1A - Population 1 accidents involving a particular type of material (e.g., secondary explosives).
- Population 1B - Population 1 accidents involving a particular type of process operation (e.g., mixing).
- Population 2 - All accidents regardless of the stimuli given as probable causes.
- Population 3 - All materials of a particular type, regardless of whether or not the material had been involved in an accident.

The terminology used to describe the populations is somewhat awkward as the words 'accidents' and 'materials' may seem to be used interchangeably. Populations 1 and 2 refer to accidents, and population 3 refers to materials. However, each accident involved some material. Thus, in actuality we are talking only about materials. The word 'material' could have been used in defining populations 1 and 2; however, this would have led even to more confusing language. For instance, population 1 would have been: all materials involved in an accident in which the particular stimuli of interest were probable causes.

Tables 11 to 14 summarize the means and standard deviations that were calculated for ignition by impact, friction, ESD, and heat. Insufficient data were available for any similar analyses for ignition by impingement or adiabatic compression. The pertinent data from these tables have been extracted and are summarized in Fig 13 to 18.

Figure 13 shows the mean $\pm$ one standard deviation of the impact sensitivities of population 1A and 3 materials. The figure shows that population 1A materials are more sensitive to impact ignition (lower impact sensitivities) than population 3 materials. Using method 3-3.13 of ORDP 20-110 (Ref 39), the means are different at a level of significance of 0.05. Thus, the materials involved in accidents in which

Table 11

Impact sensitivity means and standard deviations
for various sample populations

Sample	Material		$\bar{x}$ ($\times 10^4$)	s ($\times 10^4$)	n
Population 1A					
Materials involved in accidents in which impact was a probable cause	All materials		1.54	0.97	40
	All propellant types		1.48	0.81	13
	Secondary explosives		2.11	0.98	18
	Primary explosives		0.59	0.25	9
Population 1B					
Processes involved in accidents in which impact was a probable cause	Pressing	Group A	2.36	1.35	18
	Mixing	Group A	2.03	0.88	4
	Casting	Group A	1.85	1.02	5
	Reacting	Group B	1.22	0.96	3
	Machining	Group B	0.83	0.35	6
	Filling	Group B	0.95	0.51	6
	Conveying	Group B	1.15	1.21	2
	Drying	Group C	0.50	0.26	5
	Screening	Group C	0.26	0.11	2
	Group A		2.21	1.22	27
	Group B		0.98	0.59	17
	Group C		0.44	0.25	7
Population 2					
Processes involved in accidents regardless of cause	Pressing		2.04	1.32	15
	Mixing		2.03	0.88	4
	Coating		1.91	0.93	6
	Reacting		1.22	0.96	3
	Machining		0.98	0.45	5
	Filling		1.25	1.07	8
	Conveying		1.15	1.21	2
	Screening		0.26	0.11	2
	Drying		0.50	0.26	5
Population 3					
Materials involved or not involved in accidents	All materials		3.07	2.17	84
	All propellants		3.31	1.96	58
	All propellants-finished		2.84	2.00	35
	All propellants-in process		4.02	1.71	23
	Secondary explosives		3.06	2.57	21
	Primary explosives		2.09	1.15	5
	Single base-finished		2.42	1.75	12
	Double base-casting, finished		2.08	2.08	4
	Double base-solvent, finished		3.57	2.28	11
	Double base-solventless, finished		2.94	2.11	7
	Single base-in process		4.83	1.08	7
	Double base-solvent, in process		3.15	1.53	4
	Double base-solventless, in process		3.87	2.05	11

$\bar{x}$ = mean (J/m^2)
s = standard deviation
n = sample size

Table 12

Friction sensitivity means and standard deviations
for various sample populations

Sample	Material		$\bar{x}$ ($\times 10^6$)	s ($\times 10^8$)	n
Population 1A					
Materials involved in accidents in which friction was a probable cause	All materials		2.63	1.82	41
	All propellant types		2.15	1.90	18
	Secondary explosives		3.55	1.51	18
	Primary explosives		1.08	0.00	5
Population 1B					
Processes involved in accidents in which friction was a probable cause	Pressing	Group D	3.70	2.01	18
	Casting	Group D	4.00	1.50	3
	Mixing	Group E	3.04	1.75	3
	Reacting	Group E	2.52	2.11	3
	Machining	Group F	0.71	1.02	10
	Conveying	Group F	1.50	0.55	2
	Filling	Group F	1.68	0.82	8
	Drying	Group F	1.57	0.00	1
	Group D		3.74	1.92	21
	Group E		2.78	1.76	6
	Group F		1.71	0.84	22
Population 2					
Processes involved in accidents regardless of cause	Pressing		3.61	2.10	14
	Casting		2.95	1.64	4
	Mixing		3.04	1.75	3
	Reacting		2.52	2.11	3
	Machining		1.41	1.30	4
	Conveying		1.54	0.55	2
	Filling		1.59	0.84	7
	Drying		1.83	0.61	4
Population 3					
Materials involved or not involved in accidents	All materials		4.22	1.96	82
	All propellants		4.48	1.83	61
	All propellants-finished		3.67	1.17	36
	All propellants-in process		5.64	2.00	25
	Secondary explosives		4.07	2.16	16
	Primary explosives		1.58	0.49	5
	Single base-finished		3.83	1.18	12
	Double base-casting, finished		3.32	0.85	4
	Double base-solvent, finished		3.48	1.01	12
	Double base-solvent-less, finished		3.65	1.51	7
	Single base-in process		6.61	1.89	8
	Double base-solvent, in process		4.55	2.66	5
	Double base-solvent-less, in process		5.48	1.70	11

$\bar{x}$ = mean (N/m^2 @ 1.6 m/s)
s = standard deviation
n = sample size

Table 13

ESD sensitivity means and standard deviations for various sample populations

Sample	Material	$\bar{x}$	s	n
Population 1A				
Materials involved in accidents in which ESD was a probable cause	All materials	0.07	0.10	16
Population 2				
Materials involved in accidents regardless of cause	All materials	0.24	0.39	19
Population 3				
Materials involved or not involved in accidents	All materials	1.36	2.09	50

$\bar{x}$ = mean (J)

s = standard deviation

n = sample size

Table 14

Thermal sensitivity* means and standard deviations for various sample populations

Sample	Material	$\bar{x}$	s	n
Population 1A				
Materials involved in accidents in which heating was a probable cause	All materials	308.56	113.29	25
	Propellants	245.00	127.48	4
	Secondary explosives	325.47	112.29	19
	Primary explosives	275.00	91.92	2
Population 2				
Material involved in accidents regardless of cause	All materials	275.94	94.32	18
	Propellants	223.80	119.50	5
	Secondary explosives	301.60	85.29	10
	Primary explosives	277.33	65.13	3
Population 3				
Material involved or not involved in accidents	All materials	297.34	85.91	58

* Autoignition temp (°C at 5 s)

$\bar{x}$ = mean

s = standard deviation

n = sample size

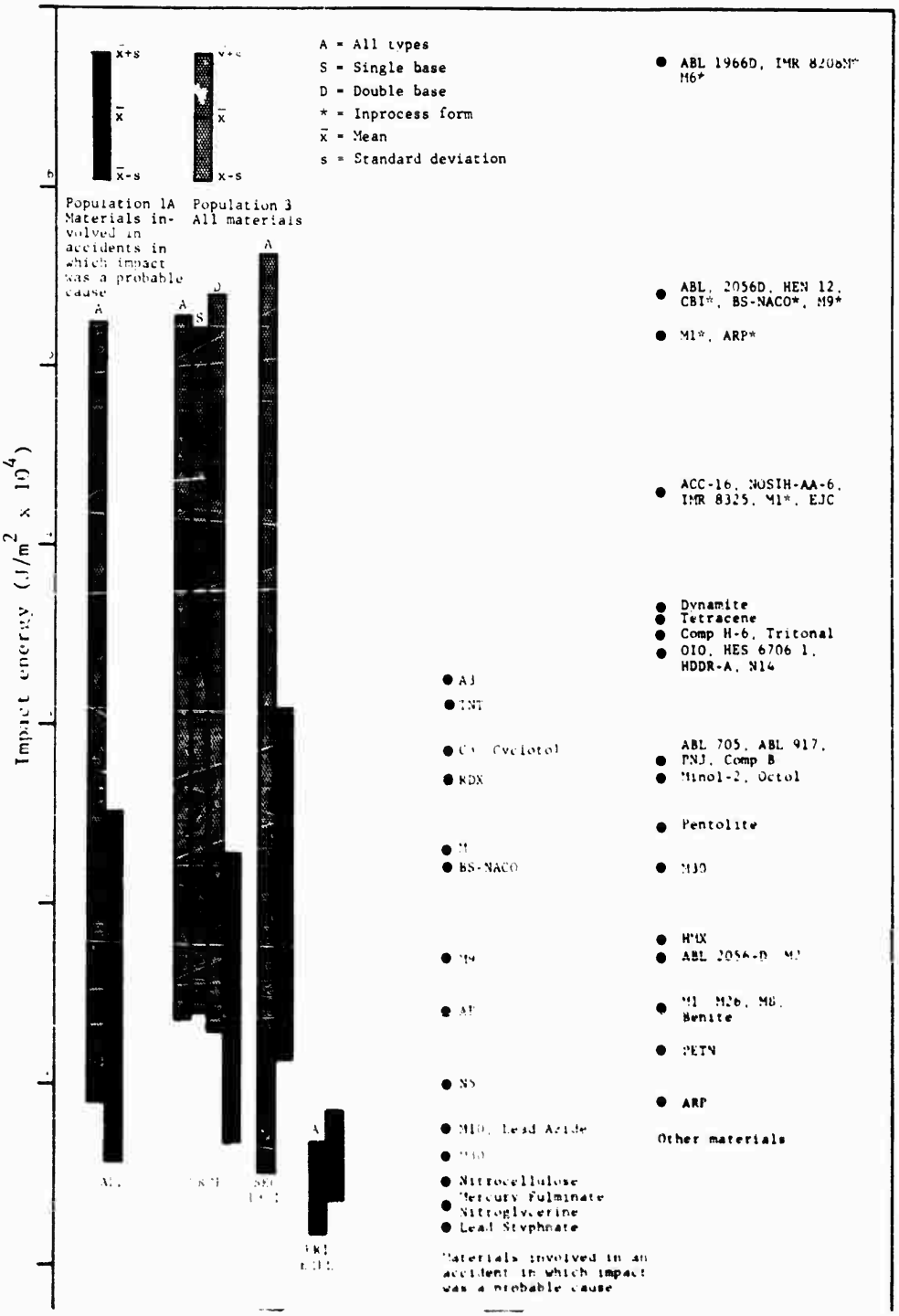


Fig 13 Impact sensitivities for population 1A and population 3 materials

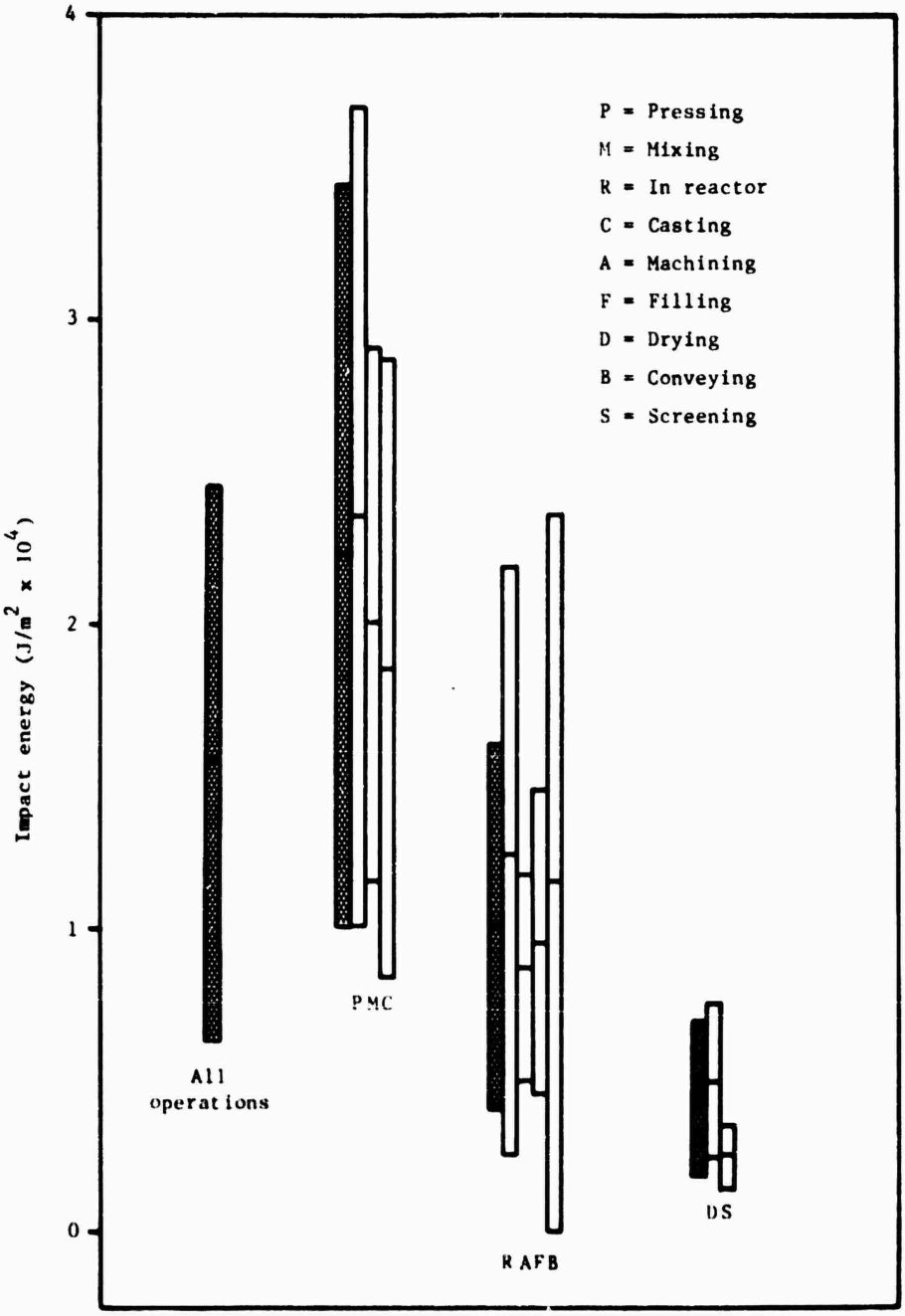


Fig 14 Impact sensitivities for population 1B materials

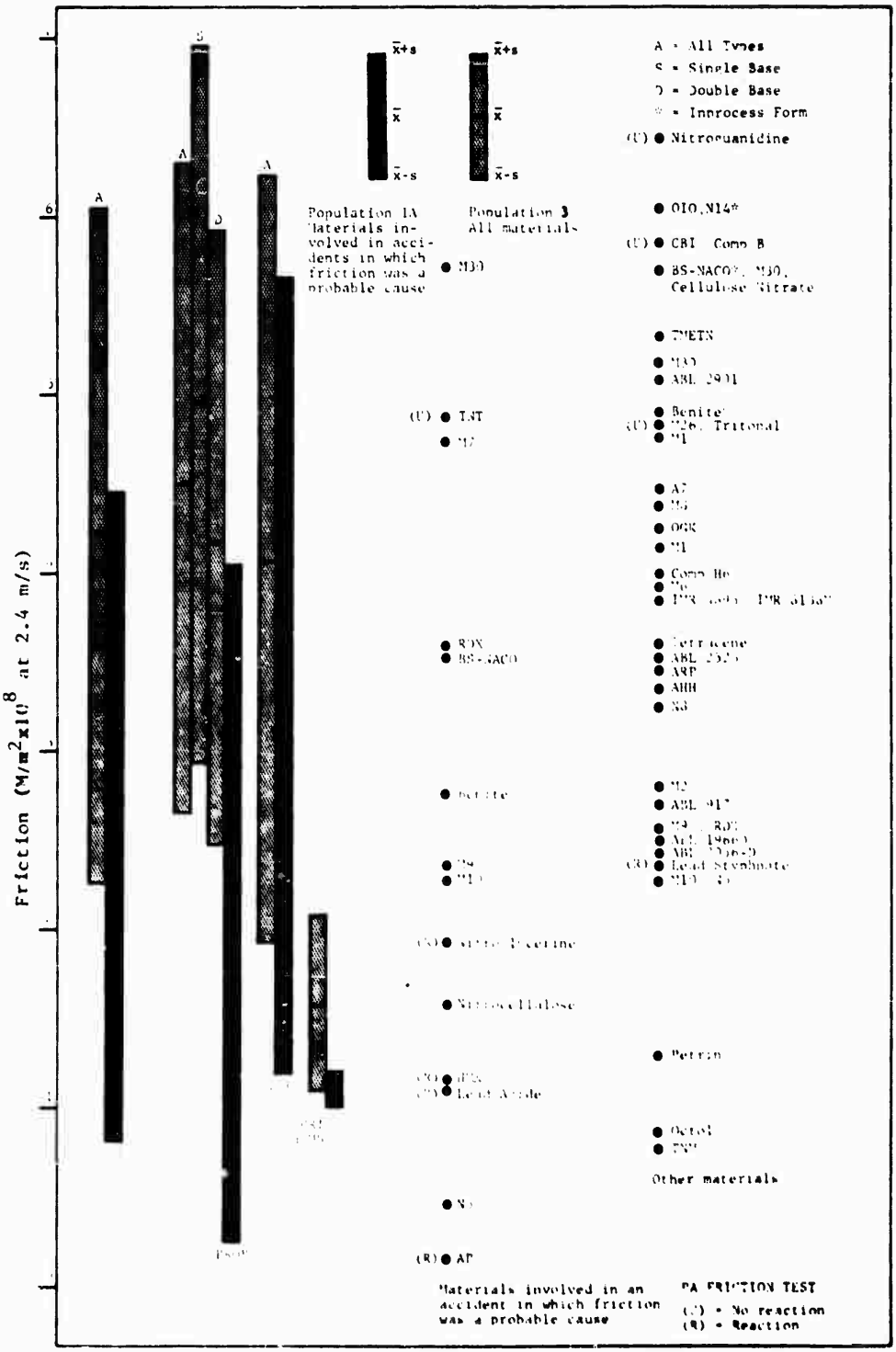


Fig 15 Friction sensitivities for population 1A and population 3 materials

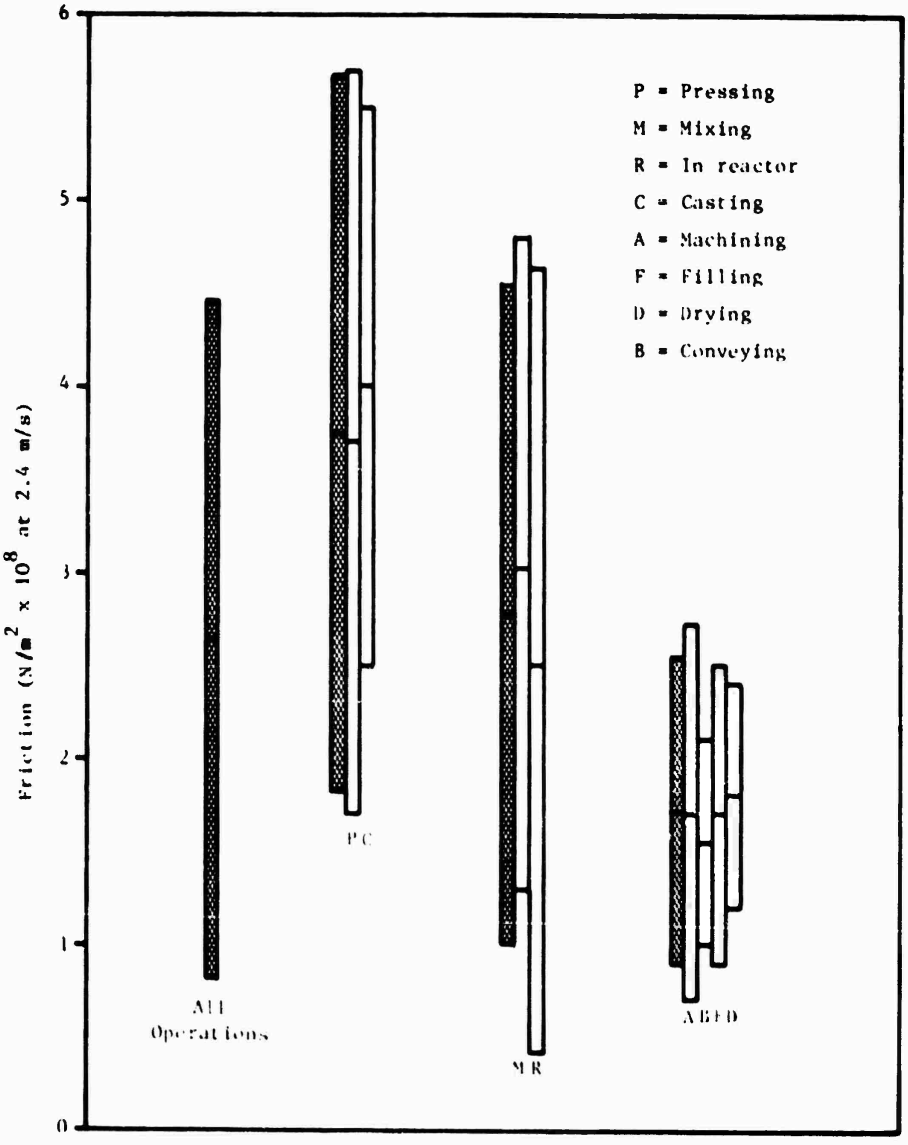


Fig 16 Friction sensitivity of population 1B materials

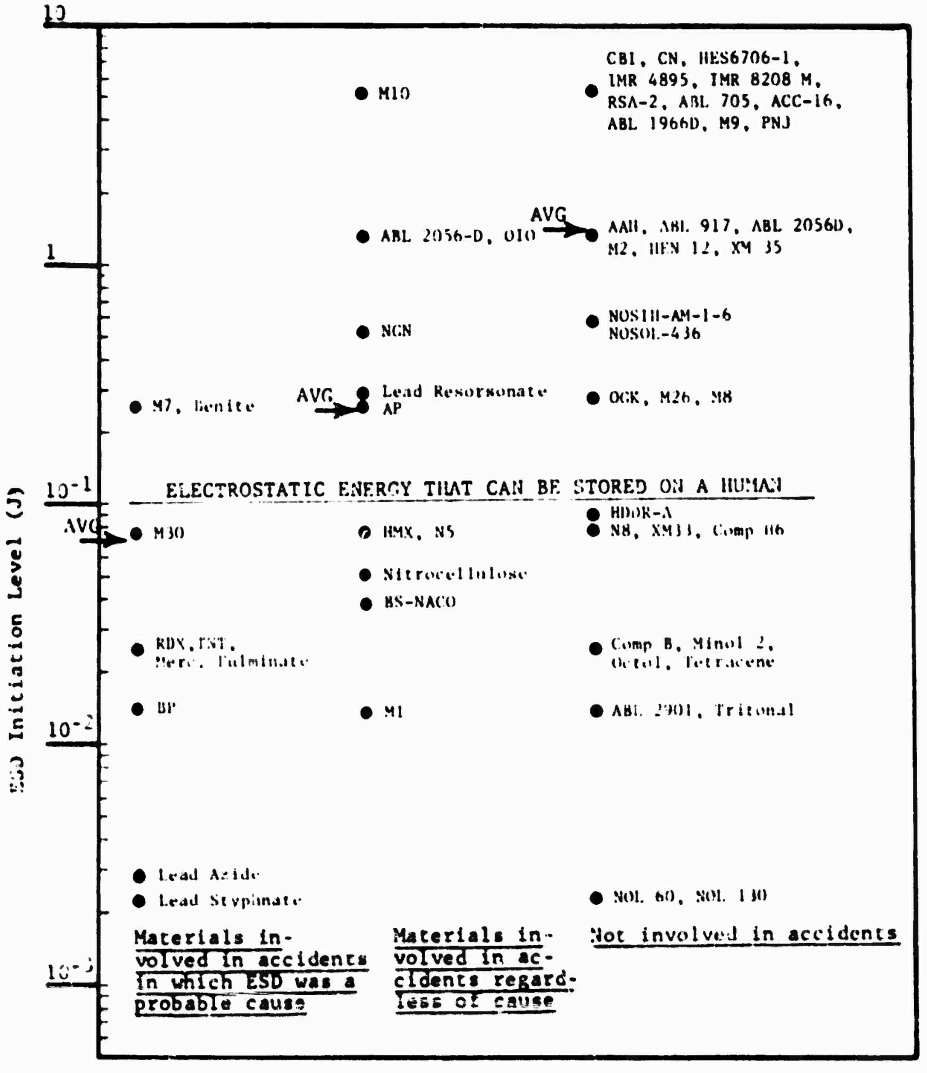


Fig 17 Sensitivity to electrostatic discharge (ESD) for population 1A, 2 and 3 materials

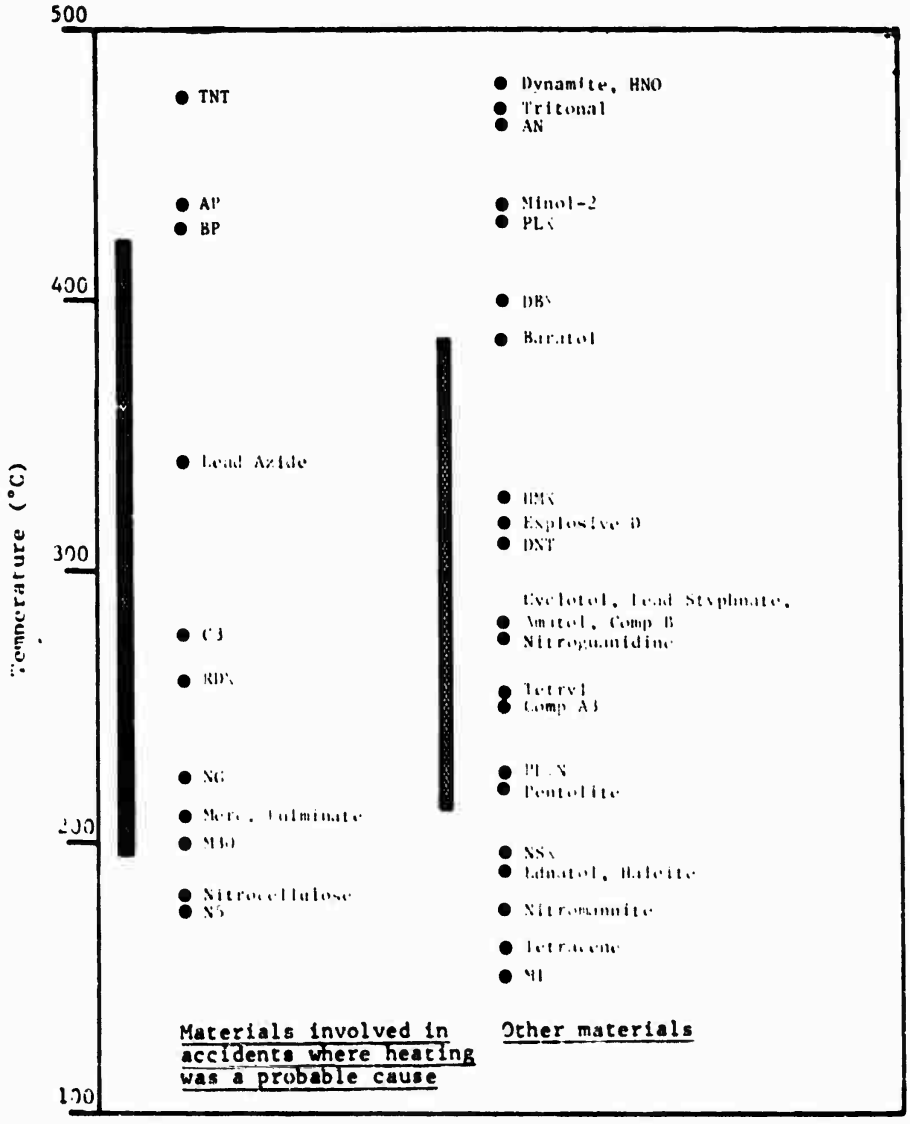


Fig 18 Sensitivity to heat for population 1A and population 3 materials

impact was a probable cause are significantly more sensitive to ignition by impact than most materials.

The impact energy for various materials representative of those in population 1A are shown as individual plotted points on the right side of Fig 13. These points show how the populations overlap and indicate the degree to which population 1A materials are concentrated in the lower (more sensitive) portion of the diagram.

Figure 14 shows the mean $\pm$ one standard deviation of the impact sensitivities for population 1B materials. The process operations whose means are not different at a level of significance of 0.05, method 3-4 of ORDP 20-110 (Ref 39), are grouped together. Combined mean $\pm$ one standard deviation are shown for each group by the shaded bar. Thus, the process operations can be combined into the following three groups which have statistically similar impact sensitivities.

These groups are:

- Group A - pressing, mixing and casting
- Group B - reacting, machining, filling and conveying
- Group C - drying and screening

The bands which are shown in Fig 14 contain over 68 percent of the sample population (mean $\pm$ one standard deviation). Thus, less than 16 percent of the population lies either above or below the band. Therefore, by choosing the top of the band (mean + one standard deviation) as the impact sensitivity criterion, we have included about 84 percent of the population 1B materials. The criterion could be changed to include about 97 percent of the population by raising the top of the band to the mean + two standard deviations. However, then the criterion would also include virtually every material, and would not discriminate between those likely to be a problem and those which are not. On this basis, the following impact sensitivity criteria were chosen, as listed in Table 15.

Table 15

Impact sensitivity criteria

Group	Process operation	Sensitivity (J/m^2)
A	Pressing, mixing, casting	3.34×10^4
B	Reacting, machining, filling, conveying	1.57×10^4
C	Drying, screening	0.69×10^4

Figure 15 shows the mean $\pm$ one standard deviation of the friction sensitivity of population 1A and population 3 materials. It can be seen that the materials involved in accidents in which friction was a probable cause are significantly more sensitive to friction ignition than most materials except for secondary explosives. Here, a difference does exist but the difference is not significant at a level of significance of 0.05, method 3-4, ORDP 20-110 (Ref 39). The friction sensitivities of materials representative of both populations are shown on the right side of the figure. The results of the Picatinny Arsenal Friction Test using a steel shoe are also shown for a number of materials. While not enough data are available for a meaningful comparison, the PA test does differentiate between materials.

Figure 16 shows the mean $\pm$ one standard deviation of the friction sensitivity of population 1B materials. The process operations whose means are not statistically different at a level of significance of 0.05 have been grouped together. The mean $\pm$ one standard deviation for the entire group is also shown (dark bar). These groups are

- Group D - pressing, casting
- Group E - mixing, reacting
- Group F - conveying, filling, machining, drying

These groups do not correspond with groups A, B and C for impact sensitivity. As with impact sensitivity, the mean $\pm$ one standard deviation was taken as the friction sensitivity criterion for each group. These criteria are given in Table 16.

Table 16

Friction sensitivity criteria

Group	Process operation	Sensitivity N/m^2 @ 2.4 m/s)
D	Pressing, casting	5.66×10^8
E	Mixing, reacting	4.54×10^8
F	Conveying, filling, drying, machining	2.55×10^8

Figure 17 shows the mean electrostatic discharge (ESD) sensitivity for population 1A, 2 and 3 materials. Materials representative of the populations are also shown, plotted on the basis of their ESD sensitivity. It is interesting to note that the mean ESD sensitivity for population 1A materials is below the 0.10 joule level, which is generally accepted as the amount of electrostatic energy that can be stored on a human. The mean $\pm$ one standard deviation (0.17 joule) is above that figure, and probably indicates that other energy sources may be the cause of some ignitions by ESD. The sample was too small to break out a 1B population, so 0.17 joule will be chosen as the ESD sensitivity criterion.

Figure 18 shows the mean $\pm$ one standard deviation of the thermal sensitivity (autoignition temp @ 5 s) for population 1A and population 3 materials. There is statistically no difference in the thermal sensitivities of the two populations.

In order to obtain a criterion for thermal sensitivity, other means must be used. In some hazards analyses where only limited data on the process are available, a temperature between 10 and 20 percent above the maximum process operating temperature is assumed to be the highest potential of the process. Thus, it would not be unreasonable to use as the thermal sensitivity criterion the maximum process operating temperature + 20 percent.

There were insufficient data on which an impingement sensitivity criterion could be based. Therefore, as was done with thermal sensitivity, the impingement sensitivity criterion is taken as the maximum process impingement velocity + 20 percent.

No data exist on which an adiabatic compression sensitivity criterion can be based. Adiabatic compression is quite possible during impact, but it is unknown how the two effects can be separated. Therefore, no adiabatic compression sensitivity criterion can be found.

The hazards classification sensitivity criteria which were obtained in this analysis are presented in Table 17.

There are several shortcomings to this analysis which should be noted. These can be summarized as follows:

- A sample rather than the entire DDESB accident base was used. Theoretically, the sample is indicative of the whole; however, no checks of the sample have been made.

- The populations on which some of the statistics have been based are rather small due to the accident sample size, lack of data on all the sampled accidents, and lack of an exact description of the materials. This small sample size implies that large changes in the statistics are possible with only a few additional data points.

- The Radford AAP data that was used had already been converted to engineering units without supplying the original data. Such original data would have been helpful in attempting to correlate Radford data with those from other sources.

These shortcomings may or may not affect the sensitivity criteria. It is recommended, however, that the shortcomings be investigated in greater detail in a later program by using the entire DDESB data base, and by using raw Radford AAP data to obtain correlations with other sources.

Table 17

Hazards classification sensitivity criteria

a. Mixing

Friction	4.54×10^8 newtons/m^2 @ 2.4 m/s
Impact	3.34×10^4 joules/m^2
ESD	0.17 joules

b. Pressing

Friction	5.66×10^8 newtons/m^2 @ 2.4 m/s
Impact	3.34×10^4 joules/m^2
Adiabatic compression	Unknown
ESD	0.17 joules

c. Reactor

Heating	Maximum process operating temperature + 20%
ESD	0.17 joules

d. Drying

Friction	2.55×10^8 newtons/m^2 @ 2.4 m/s
Impact	0.69×10^4 joules/m^2
Heating	Maximum process operating temperature + 20%
ESD	0.17 joules

e. Screening

Friction	2.55×10^8 newtons/m^2 @ 2.4 m/s
Impact	0.69×10^4 joules/m^2
ESD	0.17 joules
Impingement	Maximum process velocity + 20%

f. Filling

Friction	2.55×10^8 newtons/m^2 @ 2.4 m/s
Impact	1.57×10^4 joules/m^2
ESD	0.17 joules
Heating	Maximum process operating temperature + 20%

g. Conveying

Friction	2.55×10^8 newtons/m^2 @ 2.4 m/s
Impact	1.57×10^4 joules/m^2
Impingement	Maximum process velocity + 20%
ESD	0.17 joules

DEVELOPMENT OF PRELIMINARY HAZARD CLASSIFICATION PROCEDURES

Preliminary Procedures for In-Process Hazard Classification

Hazard classification is in reality a two-step process. The first step is to gather the data necessary to classify the material by the use of specified tests. The second step is to interpret the test results and determine the material's classification. The following sections present our current thoughts in both areas. It should be stressed that the ideas presented are preliminary in nature and are subject to change.

Testing

There is much information which could be gathered on a material depending on the tests and test instrumentation specified. Some of this data is necessary to properly classify the material and some is not. Due to the expense of testing, our philosophy is to conduct only those tests that are necessary and to structure or specify the tests in such a manner that the data can be used for more than just the hazards classification (e.g., hazards analysis also).

The testing that should be conducted can be divided into three basic classes:

- General material properties,
- Material sensitivity, and
- Effects of an accident

The tests are further divided into tests which will be conducted on all materials, and tests which will be conducted only when specific test results or process operations dictate that such tests be conducted.

The general material tests are to determine:

- Material characteristics such as particle size distribution or composition.
- Electrical properties which indicate the material's propensity to collect and retain electrostatic charges, and
- Whether or not dusting is a problem, and the range of dust concentrations that are likely.

The last test is not necessary if the material is a liquid or a slurry. However, in these cases vapor may be a problem.

The sensitivity tests are to determine the material's sensitivity to various stimuli. The stimuli used are determined from the results of the accident analysis which correlated stimuli with process operation. At this time it is expected that tests to determine sensitivity to friction, impact, heating and ESD will be conducted on all materials. Additional tests to determine sensitivity to impingement, adiabatic compression and ESD in dusts may be required when specific materials or process operations are involved.

The output tests are to determine the effects of an accident. These effects would be the hazards that could occur as a result of an accident--namely blast, fragments, and thermal effects.

Figure 19 shows how these tests would be combined to form an integrated test plan. A more detailed description of the data desired from the tests is presented in Table 18. This table also lists standard tests which may or may not provide the required data.

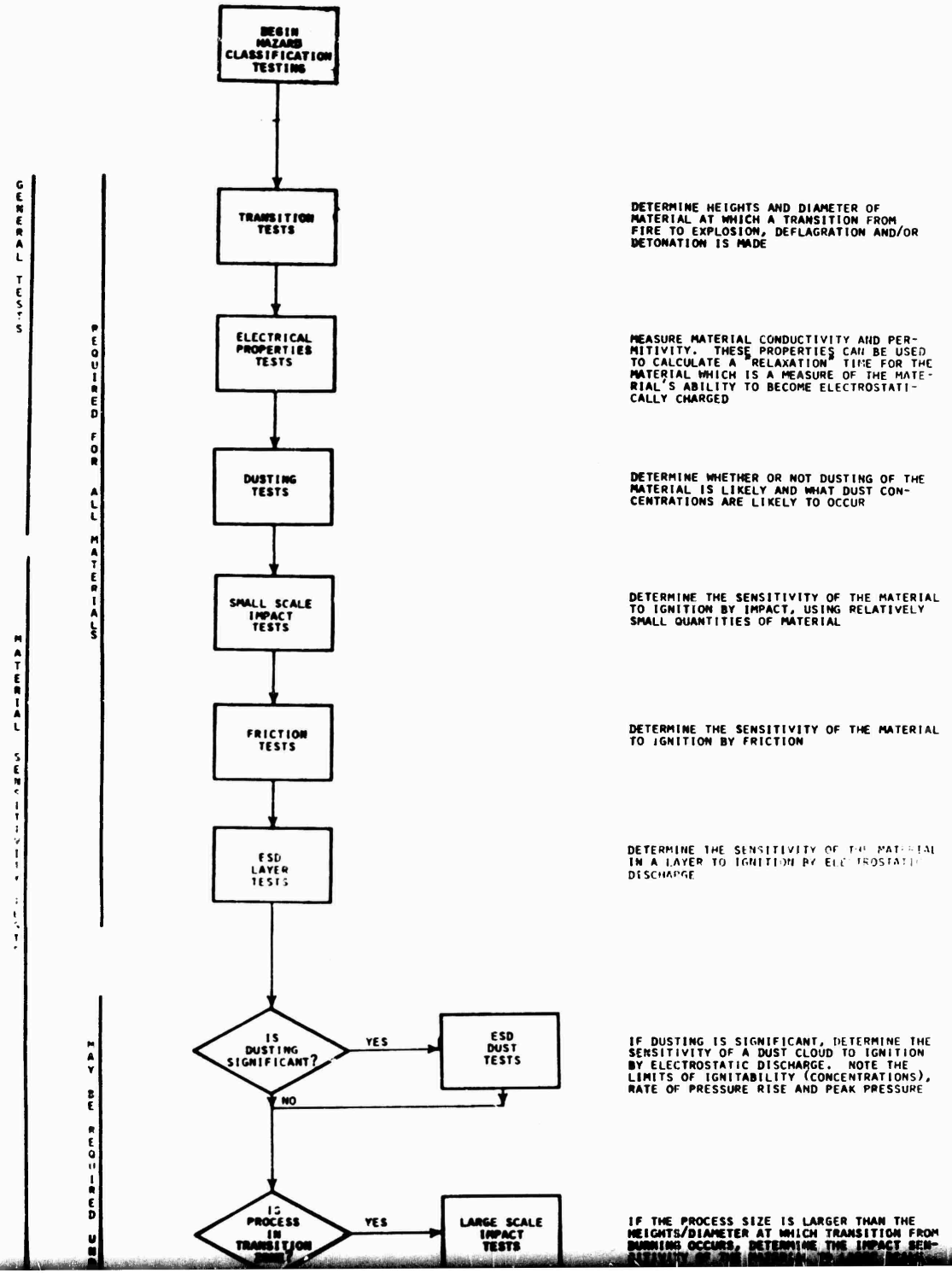
As can be seen in the table, there are many "standard" small-scale tests which could be suitable for the hazards classification procedure. An evaluation of these tests was conducted. This evaluation, in general, consisted of determining the following:

- Is the test capable of handling all the in-process material states (i.e., liquid, slurry, granular, or solid)?
- Does the test simulate a condition which exists in the process?
- Does the test provide meaningful data?

For sensitivity tests, an additional criterion was needed:

- Can the results be used as data input for a hazards analysis?

While this last criterion is not necessary for hazards classification, it would minimize the need for duplicating the


BEGIN HAZARDS CLASSIFICATION TESTING
TRANSITION TESTS
ELECTRICAL PROPERTIES TESTS
DUSTING TESTS
SMALL SCALE IMPACT TESTS
FRICTION TESTS
ESD LAYER TESTS
IS DUSTING SIGNIFICANT?
YES
NO
ESD DUST TESTS
IS PROCESS IN TRANSITION ZONE?
YES
LARGE SCALE IMPACT TESTS
GENERAL TESTS
REQUIRED FOR ALL MATERIALS
MATERIAL SENSITIVITY TESTS
MAY BE REQUIRED
DETERMINE HEIGHTS AND DIAMETER OF MATERIAL AT WHICH A TRANSITION FROM FIRE TO EXPLOSION, DEFLAGRATION AND/OR DETONATION IS MADE
MEASURE MATERIAL CONDUCTIVITY AND PERMITIVITY. THESE PROPERTIES CAN BE USED TO CALCULATE A "RELAXATION" TIME FOR THE MATERIAL WHICH IS A MEASURE OF THE MATERIAL'S ABILITY TO BECOME ELECTROSTATICALLY CHARGED
DETERMINE WHETHER OR NOT DUSTING OF THE MATERIAL IS LIKELY AND WHAT DUST CONCENTRATIONS ARE LIKELY TO OCCUR
DETERMINE THE SENSITIVITY OF THE MATERIAL TO IGNITION BY IMPACT, USING RELATIVELY SMALL QUANTITIES OF MATERIAL
DETERMINE THE SENSITIVITY OF THE MATERIAL TO IGNITION BY FRICTION
DETERMINE THE SENSITIVITY OF THE MATERIAL IN A LAYER TO IGNITION BY ELECTROSTATIC DISCHARGE
IF DUSTING IS SIGNIFICANT, DETERMINE THE SENSITIVITY OF A DUST CLOUD TO IGNITION BY ELECTROSTATIC DISCHARGE. NOTE THE LIMITS OF IGNITABILITY (CONCENTRATIONS), RATE OF PRESSURE RISE AND PEAK PRESSURE
IF THE PROCESS SIZE IS LARGER THAN THE HEIGHTS/DIAMETER AT WHICH TRANSITION FROM BURNING OCCURS, DETERMINE THE IMPACT SEN-

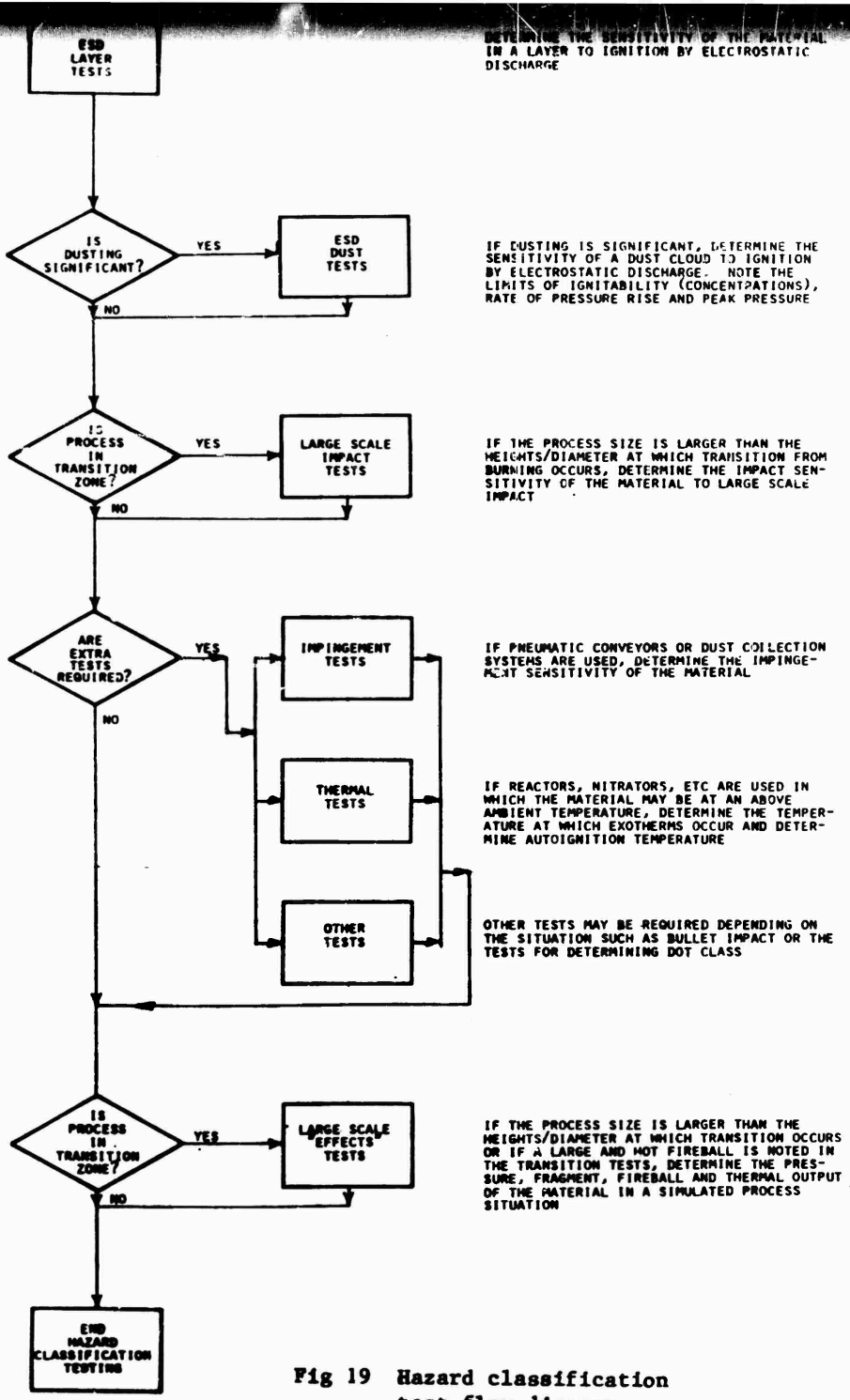


Fig 19 Hazard classification test flow diagram

Table 18

Descriptions of potential hazard classification tests

Type	Objective of test	Measured parameters	Candidate tests
Transition	Measure the height and diameter at which transition from fire to explosion, deflagration, or detonation occurs	1) Height 2) Diameter 3) Weight of material 4) Burning or detonation velocity vs height 5) Confinement	1) Closed pipe 2) Paar bomb 3) Critical height 4) Closed bomb 5) Flame spread 6) BuMines burning tests for inorganic oxidizers
Electrical properties	Measure the conductivity and permittivity of material	1) Conductivity 2) Permittivity	1) Conventional laboratory apparatus
ESD layer	Determine the sensitivity of material to ignition by electrostatic discharge (TIL and 50%)	1) Voltage vs time 2) Energy	1) BuMines 2) Picatinny Arsenal 3) NOL 4) ABL 5) GE 6) SwRI 7) BRL 8) IITRI
Small scale impact	Determine the sensitivity of small samples of material to ignition by impact (TIL and 50%)	1) Energy density	1) BuMines 2) Picatinny Arsenal 3) NOL 4) CPIA #6 5) RARDE
Friction	Determine the sensitivity of material to ignition by friction (TIL and 50%)	1) Pressure 2) Sliding	1) Rotary friction 2) Locked wheel 3) New test

Table 18 (concl)

Descriptions of potential hazard classification tests

Type	Objective of test	Measured parameters	Potential tests
Dusting	Determine the percentage of material that remains in the air after being poured into a container	1) Weight vs time 2) Light obscuration	1) New test
ESD-dust	Determine the sensitivity of dust to ignition by electrostatic discharge (TIL and 50%)	1) Energy 2) Dust concentration 3) Peak pressure 4) Rate of pressure rise	1) BuMines
Large scale impact	Determine the sensitivity of large samples of material to ignition by impact (TIL)	1) Energy density 2) Impact velocity	1) Flyer plate 2) Susan 3) Tower drop
Impingement	Determine the sensitivity of material to ignition by impingement (TIL)	1) Particle velocity 2) Particle size	1) ABL
Thermal	Determine the effect of elevated temperatures on the material	1) Autoignition temp 2) Temp at which exotherms occur	1) DTA 2) DSC
Large scale effects	Determine the hazardous effects that could result from an incident	1) Fragment map 2) Heat flux 3) Fireball diameter 4) Blast overpressure and impulse	1) New test

tests for other purposes. The results of this evaluation are summarized in Appendix B.

The test results will be most useful if they are given in basic units, and sufficient replicate tests are conducted to be meaningful statistically. The strength or intensity of a stimulus would have to be varied over a sufficient range to permit drawing curves of probability of initiation versus intensity of stimulus. Such data would be in a form directly applicable to hazards analyses. Unfortunately, the number of tests may be excessive for a hazard classification test program. Much of the current data on material sensitivity are presented for threshold initiation levels (TIL), which are the largest possible stimuli at which no ignitions are observed in either 10 or 20 tests. Such data can be used in hazards analyses, and since they require fewer tests, TIL data will generally be specified.

It should be noted that there could be a problem with the effects tests. The process materials and operations are almost always enclosed in a building or other structure. If the process is a new one, the building could be substantial ("TM walls"). Therefore the blast, fragments, and fireball measurements taken during the effects testing may be difficult to apply as they do not include the effects of the building--especially in stopping primary fragments and contributing secondary fragments.

Interpretation of Results

The tests that are performed are intended to gather sensitivity and effects data on which a hazards classification can be based. Unfortunately, such data can be likened to the proverbial apples and oranges which, as the saying goes, cannot be compared. The sensitivity data indicate the likelihood of an ignition (apples) and the effects data the results of an ignition (oranges).

The hazards classification assigns the material to a class which is indicative of the degree of hazard associated with the material. The class determines the quantity-distance relationship. Thus, hazards classification is primarily aimed at the effects of an accident.

Currently, the only effect of an accident that is addressed is blast overpressure. The current quantity-distance tables are intended to provide sufficient distance that the

blast overpressure is reduced to about 1.0 psi (7×10^3 Pa) at the inhabited-building distance. Unfortunately, because of the emphasis on blast overpressure, equally hazardous effects such as fragments, thermal radiation, and fireball are virtually ignored. Thus the classification procedure must be changed to account for all hazards.

In order to consider all hazards, it is necessary to adopt terminology which is descriptive of the hazards and their effects. Temporarily, we will adopt the following terms and definitions.

Threat- any hazard which is capable of causing fatalities, injuries,or property damage.

Threat Distance - the distance at which a hazard ceases to be a threat due to natural reductions in the hazard's available energy.

In order for the quantity-distance requirements to supply any protection, it is necessary that the threat distance be less than or equal to the required separation distance. This can be accomplished by using a threat equivalency analogous to TNT equivalency. This would be the ratio of a standard weight to a process charge weight which would produce equivalent damage at the same radial distance from each charge (equal threat distances).

Scaled distance is usually defined as the ratio of distance to the cube root of the charge weight. If W is the charge weight and L is the threat distance, the scaled distances are:

$$\lambda_s = \frac{L_s}{W_s^{1/3}} \tag{11}$$

and $$\lambda_p = \frac{L_p}{W_p^{1/3}} \tag{12}$$

where the subscripts s and p refer to the standard charge and the process simulated charge, respectively.

From the definition of threat equivalency, TE, we have:

$$TE = \frac{W_s}{W_p} \quad \text{at } L_p = L_s \tag{13}$$

By combining Equations 11, 12 and 13, we have

$$TE = \left(\frac{\lambda_p}{\lambda_s}\right)^3 = \frac{L_p^3}{\lambda_s^3 W_p} \tag{14}$$

In order to use Equation 14, we must define our threats. Using the criteria given in the UN-NATO document, fragments are a threat if the fragments have energies of 79 J or more and occur at a density of per 1 per 56 m^2 or more, thermal radiation is a threat if the radiant heat flux is 1.3×10^4 $J/m^2/s$ or more, and the fireball is a threat out to its outermost radius. Overpressure is a threat to structures at 7×10^3 Pa and to glass at pressures less than 1.4×10^3 Pa. For now, a criterion of 3.5×10^3 Pa will be used. By definition, these threats are all equivalent in that they all can produce fatalities, injuries,or property damage albeit by different mechanisms.

If we take the standard charge as a bare hemispherical charge of TNT, the scaled distance, λ_s, necessary to produce the overpressure threat can easily be obtained from tables of overpressure vs scaled distance. The threat distance, L_p, would be the maximum of the threat distances for overpressure, fragments, thermal radiation,or fireball as measured in the effects tests; and the charge weight, W_p, would be the charge weight used in the effects tests. Thus, all the data necessary for calculating the threat equivalency are easily obtained. It should be noted that the overpressures used to define the threat distance L_p and the standard scaled distance λ_s need not be the same. Different overpressure standards would provide either increased or decreased margins of safety depending on the overpressures chosen.

The threat equivalency can be used to scale the weight of process material when applying the quantity-distance standards. This would eliminate the problem of materials with different outputs, such as two mass detonation materials with TNT pressure equivalencies of 30 percent and 120 percent (e.g., black powder and C4). This would also eliminate the need for specifying minimum separation distances as is done in the UN-NATO classification procedure.

The threat equivalency can also be used to determine the appropriate hazards classification by specifying ranges of threat equivalencies for each class. If there are four hazards classes, the ranges of equivalencies may be those given in Table 19.

Table 19

Standards for classifying materials

Threat equivalency	Class
10% or more	1.1
1% to 10%	1.2
0.2% to 1%	1.3
Less than 0.2%	1.4

These breakdowns are similar to those in the UN-NATO hazard classification procedure. With this classification scheme, it is expected that Class 1.1 will represent primarily a fragment or overpressure threat, Class 1.2 primarily a fragment threat, Class 1.3 primarily a thermal radiation threat, and Class 1.4 primarily a fireball threat.

There is a certain amount of uncertainty built into this classification procedure due to the uncertainties in the data from the effects tests. This is particularly important when a material is near the boundary between classes. Thus, we anticipate using the results of the sensitivity tests to decide in which class to place borderline materials. Basically, this will be done by moving boundary line materials with low sensitivity test results into the next higher class. The philosophy behind this decision is that a more sensitive material is more likely to become involved in an accident. Thus, over a long period of time, a more sensitive material will participate in more accidents than a less sensitive material, and there will be more of a chance that the effects of the accident will be greater than those predicted in the effects tests. Therefore the material should be placed in the next highest class.

Whether or not a material has low sensitivity will be determined by comparing the material's sensitivity as determinted in the sensitivity tests with the sensitivities of

materials which have been involved in accidents. This involves the use of the sensitivity criteria described earlier.

A preliminary worksheet has been prepared to illustrate how the classification procedure would work. The worksheet is presented in Fig 20 and a table of criteria from the previous section is repeated as Table 20.

Step 1 of the procedure requires the person to fill in the threat distances associated with fragments, thermal radiation, overpressure and fireball. Criteria are given for what is a threat situation. Step 2 selects the maximum of the threat distances for later use in calculating a threat equivalency. Step 3 requires entering the weight of process material utilized in the test. This will also be used in calculating the threat equivalency.

Step 4 requires specifying a description of the process. With this information, Table 10 which contains the criteria for sensitivity can be used. These criteria were determined from accident and sensitivity data. As there may be more than one process operation in a given building, Step 5 is provided as a "scratch sheet" for writing down all the criteria. The smallest of the criteria for each type of stimulus is the worst case--the most sensitive. This is listed in Step 5 along with actual sensitivity test data. The sensitivities are compared, and the number of times that the process material is more sensitive than the criterion for each stimulus is summed and entered in Step 7.

Step 8 calculates the threat equivalency. This is used to scale the weight of process material when applying the quantity-distance standards. The constant used in the equation is $1/\lambda_s^3$ where λ_s is the scaled distance for 1.00 psi (7×10^3 Pa). Step 9 calculates the classification equivalency by multiplying the threat equivalency by 1000 and adding the penalty factor. The penalty corresponds to an addition of 0.2 percent threat equivalency for each time the process material is more sensitive than the criterion.

Step 10 converts the classification equivalency to a hazard class and is identical to the criteria given in Table 20. Due to the scale factor of 1000, a classification equivalency of 100 corresponds to a threat equivalency of 0.10 or 10 percent.

The procedure is somewhat confusing, so an example is presented which utilizes assumed numbers.

1. Fill in the following large-scale test results using average data from the three tests. If the large-scale tests were not required, enter zeros.

   a. Distance at which fragments with energies of 58 ft-lb (79 J) or more have a density of no more than one fragment per 600 square feet (56 m^2) — a._____ft (m)

   b. Distance at which the radiant heat flux was no more than 0.3 $cal/cm^2/sec$ (1.26×10^4 J/m/s) — b._____ft (m)

   c. Distance at which the blast overpressure was no more than 1 psi (7×10^3 Pa) — c._____ft (m)

   d. Maximum measured fireball radius (do not use average results) — d._____ft (m)

2. Enter the biggest number from Step 1 a to d.

   L = _____ft (m)

3. Enter the weight of material used in the large-scale tests.

   W = _____pounds (kg)

4. From the following list, select the process operations which best describe your process building or area. Enter the letter which precedes the operation in the top row of Step 5.

a. Mixing	e. Screening
b. Pressing	f. Filling
c. Reactor	g. Conveying
d. Drying	h. Machining

Fig 20 Preliminary hazard classification worksheet

5. Table 20 contains a list of criteria for the process operations listed in Step 4. Refer to Table 20 and enter the given criteria, for your process operations, in the spaces provided below. If no criterion is given, leave the space blank.

	Process Operation →	___	___	___
a.	ESD layer	_____	_____	_____
b.	Small-scale impact	_____	_____	_____
c.	Friction	_____	_____	_____
d.	ESD dust	_____	_____	_____
e.	Large-scale impact	_____	_____	_____
f.	Impingement	_____	_____	_____
g.	Thermal	_____	_____	_____
h.	Adiabatic compression	_____	_____	_____

6. Choose the smallest number in each line (row) from Step 5 and enter that number in the appropriate space in Col. 1 below. Record your test results (threshold initiation level) in Col. 2.

		Col. 1 (from Step 5)	Col. 2 (your data)	Col. 3
a.	ESD layer	_____	_____	_____
b.	Small-scale impact	_____	_____	_____
c.	Friction	_____	_____	_____
d.	ESD dust	_____	_____	_____
e.	Large-scale impact	_____	_____	_____
f.	Impingement	_____	_____	_____
g.	Thermal	_____	_____	_____
h.	Adiabatic compression	_____	_____	_____

Subtract Col. 2 from Col. 1. In Col. 3, enter a zero (0) if the result is negative or a one (1) if the result is positive. If no number appears in either Col. 1 or Col. 2, enter a zero (0).

Fig 20 Preliminary hazard classification worksheet (contd)

7. Sum the numbers in Step 6 Col. 3 and enter in the space below.

   N = _____

8. Calculate the threat equivalency, TE, using data from Steps 2 and 3.

   $TE = (1.055 \times 10^{-5}) \frac{L^3}{W}$ (constant = 1.69×10^{-4} if SI united are used)

   $TE = (1.055 \times 10^{-5}) \left(\frac{\quad\quad}{\quad\quad}\right)^3$

   TE = _____

   This threat equivalency will be used to scale the weight of process material when applying the quantity-distance standards.

9. Calculate the classification equivalency, CE, using data from Steps 7 and 8.

   CE = 1000 x TE + 2 x N

   CE = 1000 x ( ) + 2( )

   CE = _____

10. Using the classification equivalency, find the hazard classification from the table below.

CE	Hazard Classification
100 or more	Class 1.1 Mass detonating
$10 \leq CE < 100$	Class 1.2 Explosion -
$1 \leq CE < 10$	deflagration
$0 < CE < 1$	Class 1.3 Intense fire
0	Class 1.4 Minor fire

Fig 20 Preliminary hazard classification worksheet (concl)

Table 20

Hazards classification sensitivity criteria

a. Mixing

Friction	4.54×10^8 newtons/m^2 @ 2.4 m/s
Impact	3.34×10^4 joules/m^2
ESD	0.17 joules

b. Pressing

Friction	5.66×10^8 newtons/m^2 @ 2.4 m/s
Impact	3.34×10^4 joules/m^2
Adiabatic compression	Unknown
ESD	0.17 joules

c. Reactor

Heating	Maximum process operating temperature + 20%
ESD	0.17 joules

d. Drying

Friction	2.55×10^8 newtons/m^2 @ 2.4 m/s
Impact	0.69×10^4 joules/m^2
Heating	Maximum process operating temperature + 20%
ESD	0.17 joules

e. Screening

Friction	2.55×10^8 newtons/m^2 @ 2.4 m/s
Impact	0.69×10^4 joules/m^2
ESD	0.17 joules
Impingement	Maximum process velocity + 20%

f. Filling

Friction	2.55×10^8 newtons/m^2 @ 2.4 m/s
Impact	1.57×10^4 joules/m^2
ESD	0.17 joules
Heating	Maximum process operating temperature + 20%

g. Conveying

Friction	2.55×10^8 newtons/m^2 @ 2.4 m/s
Impact	1.57×10^4 joules/m^2
Impingement	Maximum process velocity + 20%
ESD	0.17 joules

Example

One building at the XXXX Ammunition Plant processes 10,000 pounds of propellant MX a day. At any given time, there is not more than 500 pounds (227 kg) of propellant in the building. The process operations which occur are extruding (pressing) the propellant into strands and conveying the strands to storage bins at 164 fps (50 m/s). The appropriate tests were conducted on the material with the following results.

Sensitivity:	ESD-layer	0.20 joules
	Impact	2.80×10^4 newtons/m^2
	Friction	1.33×10^8 joules/m^2
	Heating	300°C
	Impingement	1000 m/s
Effects:	Fragments	167 ft (50.9 m)
	Overpressure	125 ft (38.1 m)
	Thermal radiation	105 ft (32.0 m)
	Fireball	40 ft (12.2 m)
	Test weight	500 lb (227 kg)

Referring to Fig 21 which is the example of a completed hazard classification worksheet, we have completed the following:

Step 1 - Entered the threat distances of 167, 105, 125 and 40 ft

Step 2 - Entered the biggest of the threat distances from Step 1 - 167 ft

Step 3 - Entered the test weight of 500 lb

Step 4 - Marked process operations b and g in the row provided in Step 5

Step 5 - Entered the criteria from Table 20 lines b and g in the appropriate column

Step 6 - Entered the minimum criterion for each stimulus in Column 1 and the test data in Column 2. Zeros and ones were entered in Column 3 on the basis of subtracting Column 2 from Column 1. The number 1 in Column 3 indicated that the material did not pass the friction test.

Step 7 - Entered the total number of sensitivity test failures (one) into the space

Step 8 - Calculated the threat equivalency of 0.0983 (9.9%)

Step 9 - Calculated the classification equivalency of 100.3 and rounded it off to 100

Step 10 - Looked in the table and found that the material was Class 1.1

Preliminary hazard classification worksheet

1. Fill in the following large-scale test results using average data from the three tests. If the large-scale tests were not required, enter zeros (0).

   a. Distance at which fragments with energies of 58 ft-lb or more have a density of no more than one fragment per 600 square feet — a. 167 ft

   b. Distance at which the radiant heat flux was no more than 0.3 $cal/cm^2/s$ — b. 105 ft

   c. Distance at which the blast overpressure was no more than 0.5 psi — c. 125 ft

   d. Maximum measured fireball radius (do not use average results) — d. 40 ft

2. Enter the biggest number from Step 1 a to d

   L = 167 ft

3. Enter the weight of material used in the large-scale tests.

   W = 500 pounds

4. From the following list, select the process operations which best describe your process building or area. Enter the letter which precedes the operation in the top row of Step 5.

a. Mixing	e. Screening
b. Pressing	f. Filling
c. Reactor	g. Conveying
d. Drying	h. Machining

Fig 21 Sample classification problem

5. Table 20 contains a list of criteria for the process operations listed in Step 4. Refer to Table 20 and enter the given criteria, for your process operations, in the spaces provided below. If no criterion is given, leave the space blank.

Process Operation →	b	g	___
a. ESD layer	0.17	0.17	
b. Small-scale impact	3.43×10^4	1.57×10^4	
c. Friction	5.66×10^8	2.55×10^8	
d. ESD dust			
e. Large-scale impact			
f. Impingement		60	
g. Thermal			
h. Adiabatic compression			

6. Choose the smallest number in each line (row) from Step 5 and enter that number in the appropriate space in Col. 1 below. Record your test results (threshold initiation level) in Col. 2.

	Col. 1 (from Step 5)	Col. 2 (your data)	Col. 3
a. ESD layer	0.17	0.20	0
b. Small-scale impact	1.57×10^4	2.80×10^4	0
c. Friction	2.55×10^8	$1.33 c10^8$	1
d. ESD dust			0
e. Large-scale impact			0
f. Impingement	60	1000	0
g. Thermal		300	0
h. Adiabatic compression			0

Subtract Col. 2 from Col 1. In Col. 3, enter a zero(0) if the result is negative or a one (1) if the result is positive. If no number appears in either Col. 1 or Col. 2, enter a zero (0).

Fig 21 Sample classification problem (contd)

Preliminary hazard classification worksheet

7. Sum the numbers in Step 6 Col. 3 and enter in the space below.

   N = 1

8. Calculate the threat equivalenvy, TE, using data from Steps 2 and 3.

   $TE = (1.055 \times 10^{-5}) \frac{L^3}{W}$

   $TE = (1.055 \times 10^{-5}) \frac{(\ 167\ )^3}{(\ 500\ )}$

   TE = 0.0983

   This threat equivalency will be used to scale the weight of process material when applying the quantity-distance standards.

9. Calculate the classification equivalency, CE, using data from Steps 7 and 8.

   CE = 1000 x TE + 2 x N

   CE = 1000 x (0.0983) + 2 (1)

   CE = 100.3

10. Using the classification equivalency, find the hazards classification from the table below.

	CE	Hazards classification	
100.3	100 or more	Class 1.1	Mass detonating
	$10 \leq CE < 100$	Class 1.2	Explosion-deflagration
	$1 \leq CE < 10$	Class 1.3	Intense fire
	$0 \leq CE < 1$	Class 1.4	Minor fire

Fig 21 Sample classification problem (concl)

This example was set up so that a borderline case would be illustrated. Here, the penalty caused the material to be Class 1.1 rather than Class 1.2. If the material had not "failed" any of the sensitivity tests, the classification equivalency would have been 98.3 or 98 which is a Class 1.2 material.

Figure 22 shows the current quantity-distance requirements for DoD hazard classes. The threat distances taken from this example are plotted on the figure. By inspection, a military Class 7 designation would have to be given to this material as Class 2 does not provide protection from the threat. Using the UN-NATO criteria, the material would be assigned to Class 1.2 with a minimum separation distance of 167 ft (50.9 m). Thus, our procedure yields essentially equivalent hazard classes as the current DoD and UN-NATO systems. However, our system has the added benefit of a threat equivalency which scales the weight to ensure that only the minimum separation distance is called for.

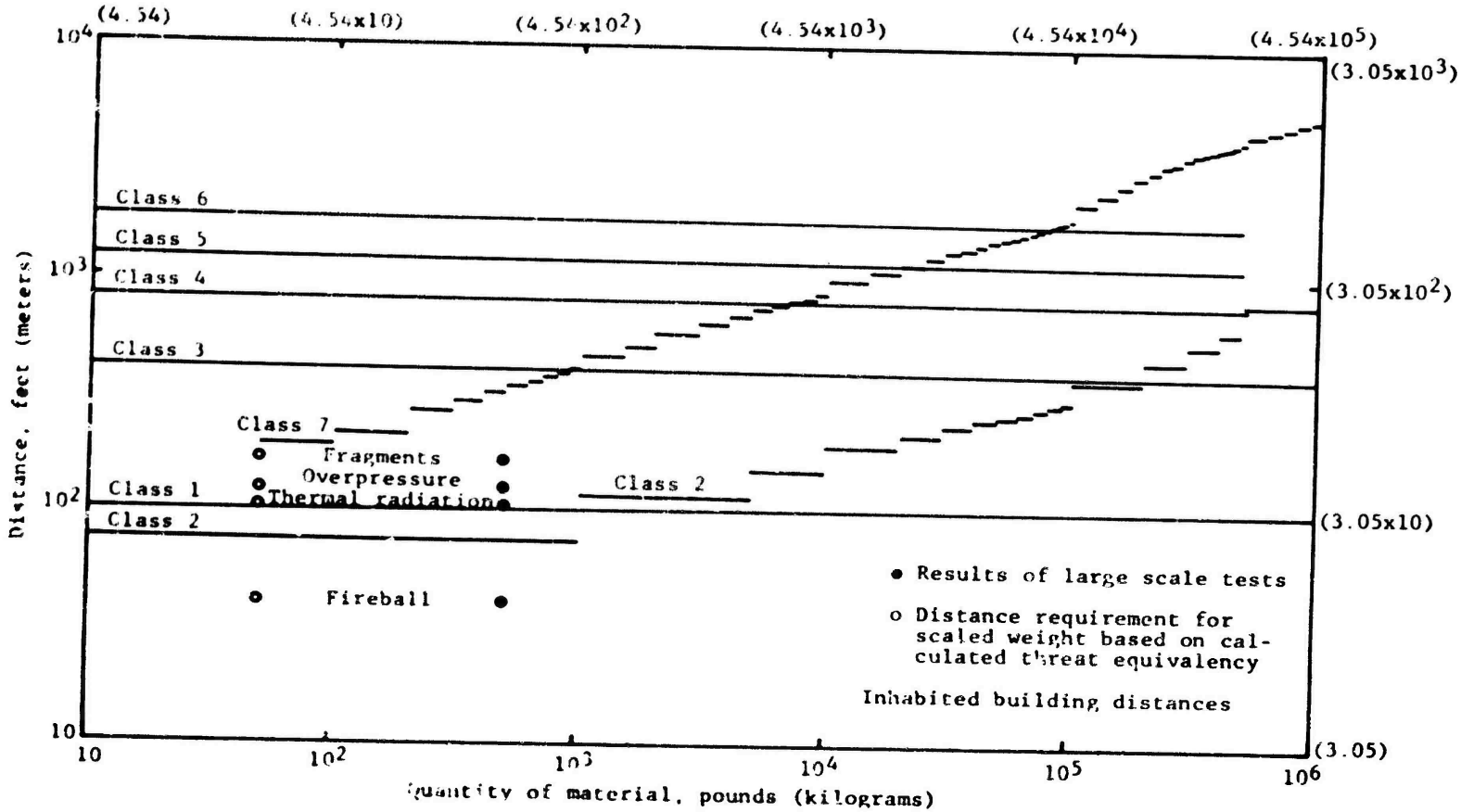


Fig 22 Relationship between the example threat distances and the current DoD quantity-distance requirements

EVALUATION OF SELECTED SMALL-SCALE TESTS

An experimental program was designed to evaluate the feasibility of using selected small-scale tests in hazards classification procedures. Numerous small-scale and some large-scale tests exist which could be applicable to a hazards classification procedure such as the one described in the previous section. These tests were surveyed and the most promising candidates selected for experimental evaluation. Four representative in-process propellant and explosive materials were chosen for the program. Detailed evaluations were completed for eight types of sensitivity tests, but not all of these reached the experimental stage. The detailed evaluations are described below.

Selection of Small-Scale Tests to Be Evaluated

A survey was made of existing and proposed tests which could be applicable to the described hazards classification procedures. The categories of tests surveyed included:

- Friction sensitivity tests, including viscous friction
- Small-scale impact and adiabatic compression sensitivity tests
- Dusting tests
- Transition tests
- Electrostatic discharge (layer) tests
- Electrostatic discharge (dust) test
- Thermal tests
- Electrical properties tests
- Large-scale impact tests

The results of the survey are presented in Appendix B, where the following information is outlined for each test category:

- test objective
- operating procedure
- test description
- applicability to hazards classification
- general discussion

- best standard tests, and in some cases a need for a new test, and
- references.

Selection of Materials Used in the Evaluation

To gain the most information from the small-scale test program, it was desirable to select materials from different stages of processing with different forms and different characteristics. It was also desirable to use materials which have known sensitivity characteristics. A number of materials were evaluated, and the following were selected:

1. M1 single perforated extruded strands, solvent wet, 12 to 25 percent total volatiles (TV)
2. M26 premixed paste, solvent wet, 13 percent TV
3. M30 air dried pellets (grains), 9 percent TV
4. RDX-H_2O slurry, 15 percent solids

The choice permitted evaluation of a single base propellant (M1), a double base propellant (M26), a triple base propellant (M30), and an explosive (RDX). Three different material forms were involved: 1) solid, 2)paste and 3) slurry. Three different solvent levels were represented. In addition, pressing/extrusion (M1), mixing (M26), drying (M30), and conveying (RDX-H_2O) were covered. Impact and friction sensitivity data from Radford AAP were available for all four materials.

Discussion of Detailed Evaluation of the Selected Tests

Detailed evaluations were completed for these eight selected tests:

Test Evaluated	Subsection
Transition	5.3.1
Impact Sensitivity	5.3.2
Friction Sensitivity	5.3.3
Dusting Propensity	5.3.4
Dust Explosibility	5.3.5
Electrical Properties	5.3.6
Electrostatic Discharge	5.3.7
Thermal	5.3.8

Transition Tests

The objectives of transition tests are to determine the critical diameter of the material for propagation of an explosive reaction, and to determine the critical height at which transition from burning to a detonation takes place. Knowing the critical charge dimensions is important in assessing in-process hazards. For example, if the process never utilizes a diameter larger than the critical diameter, it would be quite unlikely that a detonation would occur under any conditions in that process. Also, if all process charge lengths are less than the critical height, then it is unlikely that a deflagration will progress to a detonation.

The transition from a flame or other type of thermal initiation to a deflagration or detonation is affected not only by the dimensions of the material, but also by material density and confinement. Because of this, transition tests should be flexible and consider a range of densities and degrees of confinement.

Test Procedure: General

All transition tests used steel seamless mechanical tubing of various diameters and wall thicknesses. The ranges and sizes are listed in Table 21.

Each tube assembly was weighed prior to loading the sample material. The initiators were also weighed prior to loading. The sample materials were then loaded into the tubes and weighed again. The total volume occupied by the sample was noted so that the bulk density could be determined. Various densities can result depending on the amount of packing or tamping done during loading. A few trial loadings may be necessary to achieve the proper bulk density equivalent to that in the process plant.

Table 21

Tube Characteristics

Inside diameter (cm)	Outside diameter (cm)	Wall thickness (cm)	t/D_i
12.7	14.0	0.64	0.050
8.89	10.2	0.64	0.071
6.35	7.3	0.48	0.075
4.92	5.7	0.40	0.081

Table 21 (cont)

Tube Characteristics

Inside diameter (cm)	Outside diameter (cm)	Wall thickness (cm)	t/D_i
3.52	4.1	0.30	0.087
2.57	3.2	0.30	0.119
1.99	2.5	0.28	0.139
1.85	2.1	0.11	0.059
1.43	1.6	0.081	0.057
1.26	1.7	0.24	0.191
0.85	1.1	0.13	0.155
0.62	0.81	0.089	0.142
0.38	0.48	0.048	0.126
0.16	0.65	0.25	1.565

Test Description: Critical Diameter

For the critical diameter test, the sample material is initiated using a high explosive booster. The material diameter is varied and the ability of a detonation to propagate through the charge is determined. The critical diameter is the smallest tube inside diameter, for the given tube material and wall thickness, at which a stable detonation occurs.

Typical test arrangements for determining the critical diameter are shown in Fig 23. The arrangements consist of tubes filled with the test material, an explosive donor system, and a means of measuring the reaction propagation velocity. The test can simulate various process configurations by choosing the tube material and wall thickness which best simulates the type of confinement in the process.

Two methods to measure detonation velocity were evaluated experimentally. One method used a continuous wire resistance probe and the other used ionization probes. The resistance probes, although more expensive, give more reliable results than the ion probes and therefore should be used in hazards classification tests. This holds true even if more ion probes are used than the number shown here.

In each critical diameter test, the C4 explosive booster was cylindrical with the diameter equal to the pipe inside diameter, and a length to diameter ratio of 1. A tetryl pellet, 1.3 cm dia x 1.3 cm long, and a number 6 blasting cap initiated the C4 booster.

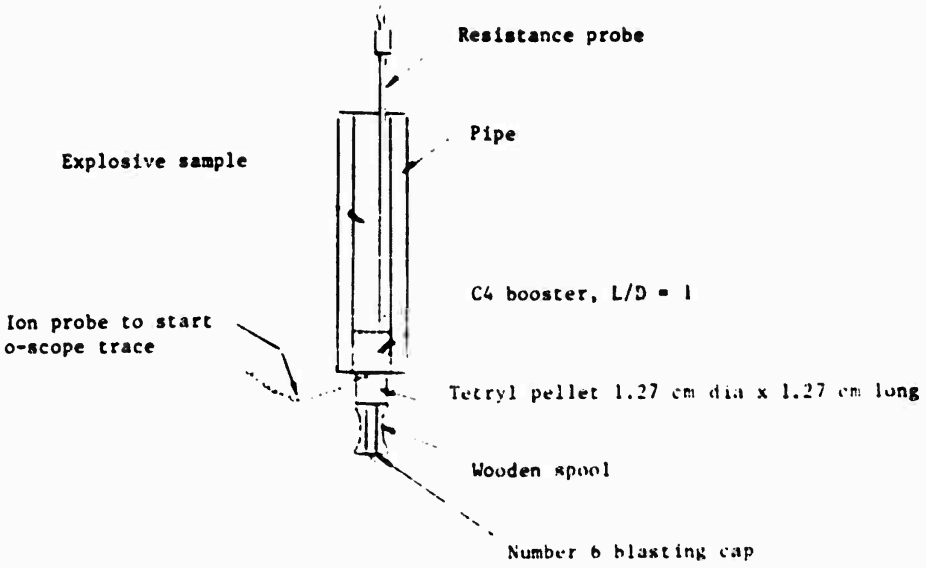


Fig 23a Critical diameter test apparatus
velocity from resistance probe

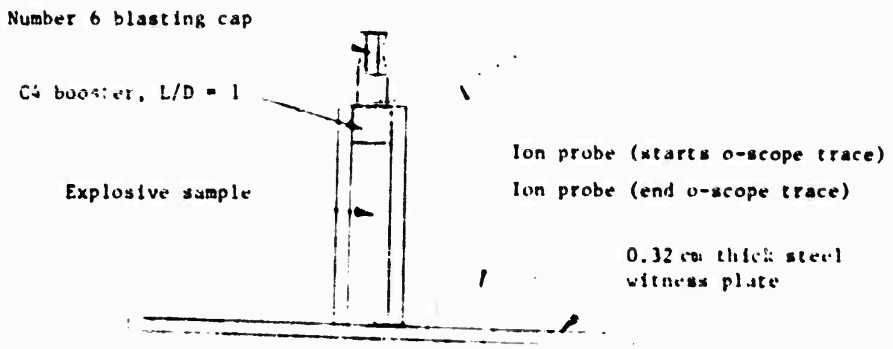


Fig 23b Critical diameter test apparatus
velocity from ion probes

For the apparatus shown in Fig 23b, the charge was placed on a witness plate so that the blast was directed downward. All witness plates were 0.32 cm thick steel plates. The intensity of the reaction was determined by velocity measurements. The witness plate damage provided additional evidence as to the severity of the reaction. Posttest fragment size also assisted in determining the severity of the reaction for both types of apparatus, (Fig 23a and Fig 23b).

Test Description: Critical Height

For the critical height test, the test material is subjected to a flame ignition. The critical height is defined as the distance (height) at which a transition occurs from burning to deflagration or to detonation.

A flame or other type of thermal ignition source is a more likely initiating event within a process than a shock wave of the type produced by a high explosive. Therefore it is important to determine the distance required for the material to make the transition from burning to deflagration or detonation. If the largest process length is less than this transition distance, then it is improbable that a deflagration will propogate into an explosion.

A typical setup for determining the critical height is shown in Fig 24. This apparatus consists of a tube filled with test material, a flame ignition source, and a means of measuring the reaction velocity. This test uses the same tube materials as the critical diameter test and begins at a diameter larger than the critical diameter to assure that a detonation can result.

The critical height is expected to depend upon the quantity of ignited material, its energy content, and rate of energy release. Therefore the igniter used in the tests should be standardized. For these tests, various amounts of black powder were used to initiate a burning reaction in the sample. A squib (Dupont S-65) was used to ignite the black powder igniter bag. Also for these tests, a 0.64 cm thick steel plate was welded to the bottom of the tube and a 0.64 cm dia hole was drilled in the center for the squib to fit snugly.

In addition to velocity measurements, posttest recovery of fragments and tube sections is important for evaluating the test results. The portion of the tube which experiences a burning reaction remains essentially intact, whereas a

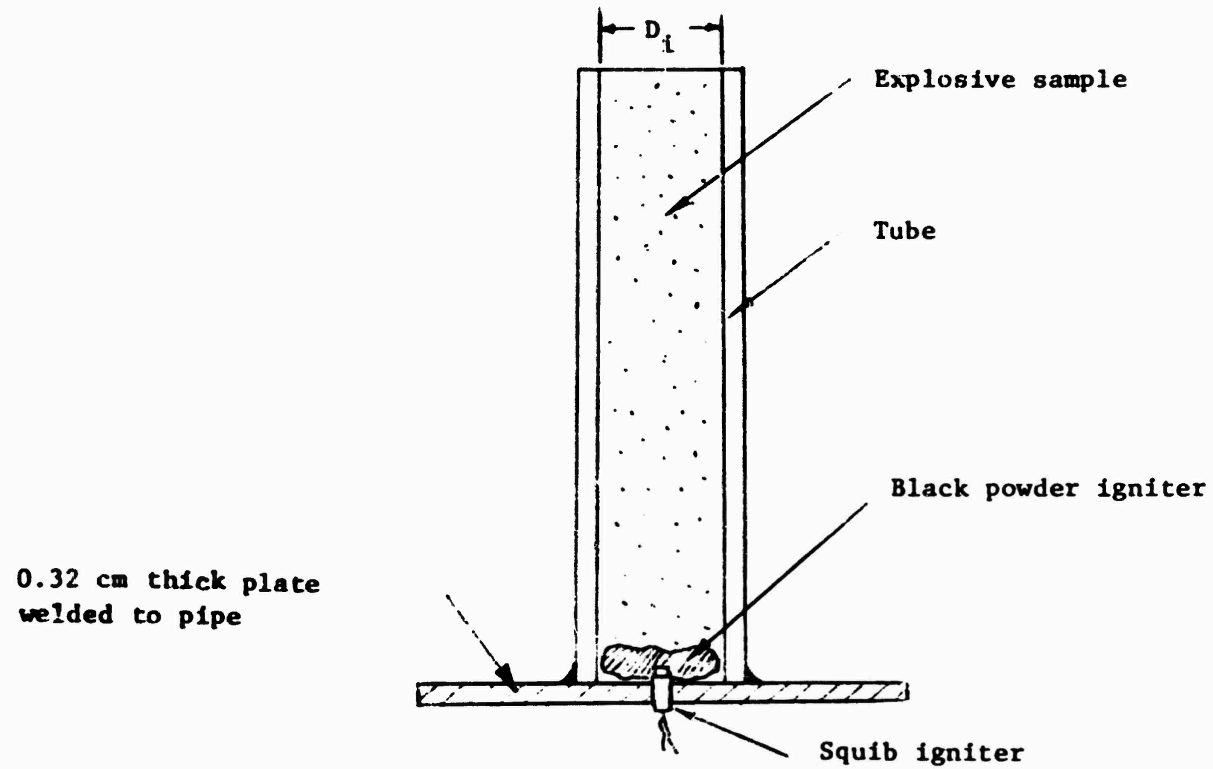


Fig 24 Critical height test apparatus
(probes for measuring propagation velocity are not shown)

detonation or explosion causes the adjacent tube material to be fragmented into small pieces. Therefore the critical height could be determined approximately by measuring the length of the recovered section of tube. Beyond that length, detonation or a violent explosion must have occurred destroying the additional section of tube.

Test Results: Critical Diameter

Forty one tests were conducted on the four pilot materials to determine their critical diameter. The data are presented in Table 22. Velocity calculations were based on data from ion probes and continuous resistance probes. The measurements from these data are listed in Table 23. Figures 25a through 25f show some typical records obtained using both methods. The oscilloscope trace is a measure of the distance the reaction front travels as a function of time. The velocity of the reaction front is determined from the slope of the oscilloscope trace. Some records indicated that there was an initial high velocity caused by the booster and then a gradual tapering off (Fig 25c and 25d).

Figure 25e and 25f show records obtained using ion probes. The signal is initiated by the ion probe in the booster and is terminated by a probe placed in the explosive or propellant material. The distance between the two probes is measured before the test, and the time for the reaction to traverse the distance is measured from the oscilloscope trace. The ion probe data must be adjusted for the booster effects, since the total time includes the time it takes the detonation front to propagate through the booster. An average detonation velocity for C4 was chosen from the literature. With the booster length known, the time to propagate through the booster was computed and subtracted from the measured total time difference. Table 23 summarizes the velocity calculations obtained from the test data using both types of probes.

The data listed in Table 22 also show the severity of the reaction by the comments on witness plate damage. Usually there is a fairly clear indication as to the critical diameter from this information. A severe dent with metal flow indicates a high order reaction, whereas only a dent indicates a low order reaction.

In addition to witness plate damage, the reaction front velocity also can be used to determine if the critical diameter has been reached. Fig 26, reproduced from Ref 40, shows that the detonation velocity reaches a plateau above a critical

Table 22

Critical diameter test results

M1 Strands

Inside diameter (cm)	t/D	Bulk density (g/cm^3)	Estimated velocity (m/s)	Witness plate damage
8.89	0.071	0.410	-	Punched hole
4.92	0.080	0.393	-	Punched hole
4.92	0.080	0.332	2330	-
3.52	0.086	0.457	-	Severe dent
3.52	0.087	0.296	2640	-
2.57	0.119	0.429	3390	Small fragments
1.85	0.059	0.432	-	Severe dent with metal flow
1.85	0.059	0.706	3260	Dent
1.26	0.191	0.329	5620	Dent, some propellant left on plate, pipe peeled

M26 Premix Paste

Inside diameter (cm)	t/D	Bulk density (g/cm^3)	Estimated velocity (m/s)	Witness plate damage
8.89	0.071	0.770	-	Punched hole
4.92	0.080	0.803	-	Punched hole
3.52	0.087	0.722	-	Punched hole
2.57	0.119	0.019	-	Punched hole
1.99	0.139	0.972	-	Punched hole
1.99	0.139	0.905	1140	-
1.85	0.059	0.614	-	Dented
1.85	0.059	1.002	5410	Punched hole
1.26	0.191	0.772	-	Severe dent with metal flow
1.26	0.191	0.681	1480	-
0.85	0.155	0.263	3470	Dent with metal flow

Table 22 (concl)

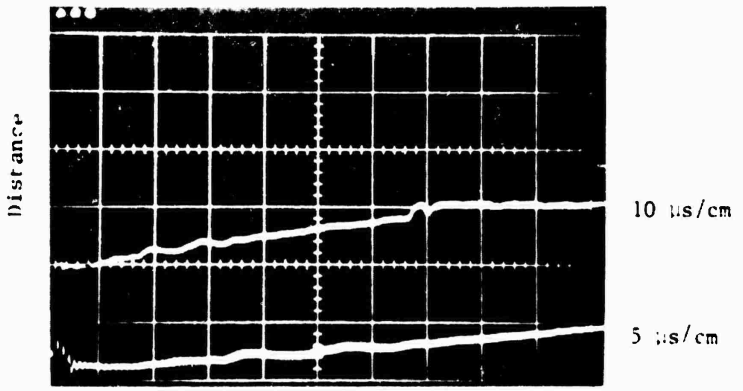
Critical diameter test results

M30 Grains

Inside diameter (cm)	t/D	Bulk density (g/cm^3)	Estimated velocity (m/s)	Witness plate damage
8.89	0.071	0.819	-	Punched hole
4.92	0.080	0.814	-	Punched hole
4.92	0.080	0.725	3170	-
3.52	0.087	0.775	-	Severe dent
3.52	0.087	0.714	3610	-
2.57	0.119	0.626	3290	Small fragments
1.85	0.059	0.614	-	Small dent
1.85	0.059	0.955	9620	Small dent
1.43	0.057	1.248	3150	No dent, large fragments
1.26	0.191	0.692	-	Small dent
1.26	0.191	1.193	3020	Small dent, pipe bananna peeled at end

RDX Slurry

Inside diameter (cm)	t/D	Bulk density (g/cm^3)	Estimated velocity (m/s)	Witness plate damage
2.57	0.119	0.999	-	Punched hole
1.99	0.139	0.938	-	Punched hole
1.85	0.059	0.974	-	Punched hole
1.43	0.057	0.770	-	Punched hole
1.26	0.191	0.897	-	Punched hole
0.85	0.155	1.107	-	Punched hole
0.62	0.142	0.855	-	Dent with metal flow
0.62	0.142	1.099	5500	-
0.38	0.126	1.204	-	Dent with metal flow
0.38	0.126	1.323	4930	-



Time

Fig 25a Continuous probe data for M30, 9 cm I.D.

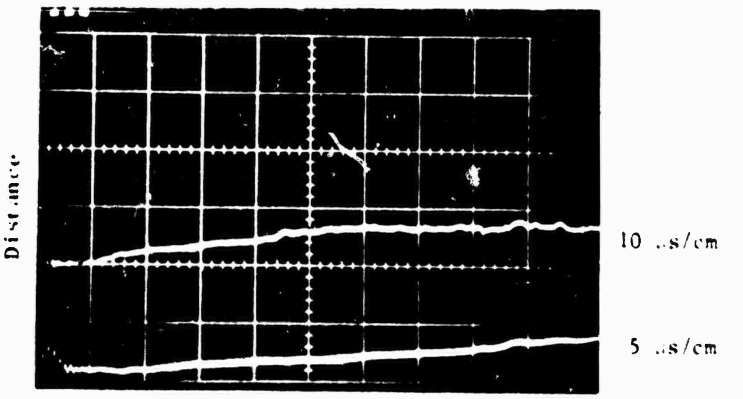


Fig 25b Continuous probe data for M30, 9 cm I.D.

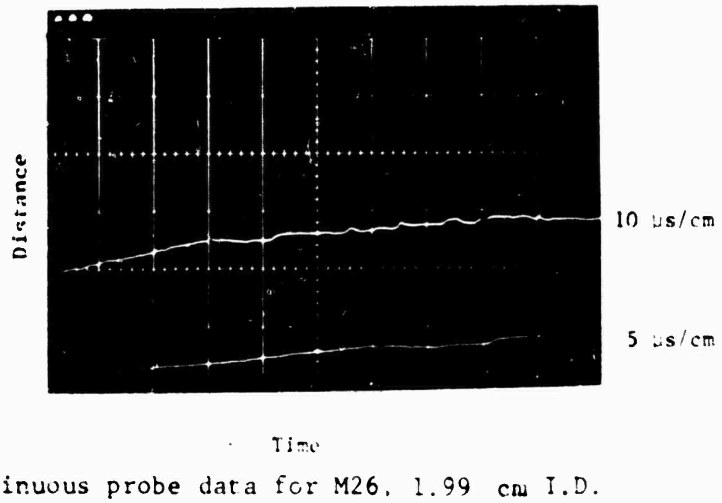


Fig 25c Continuous probe data for M26, 1.99 cm I.D.

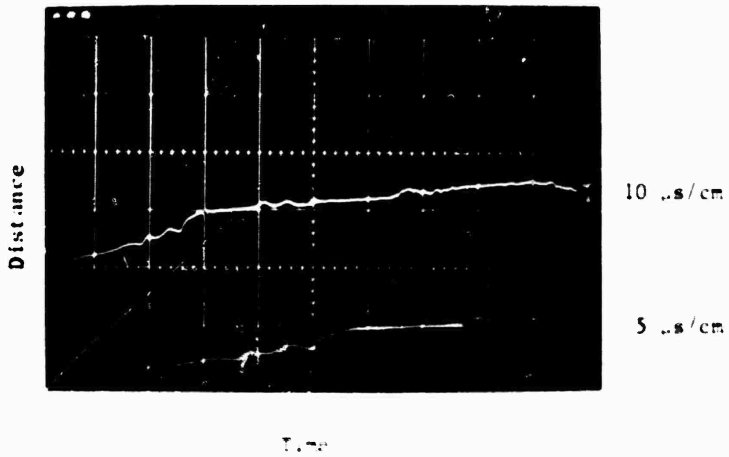


Fig 25d Continuous probe data for M1, 4.92 cm I.D.

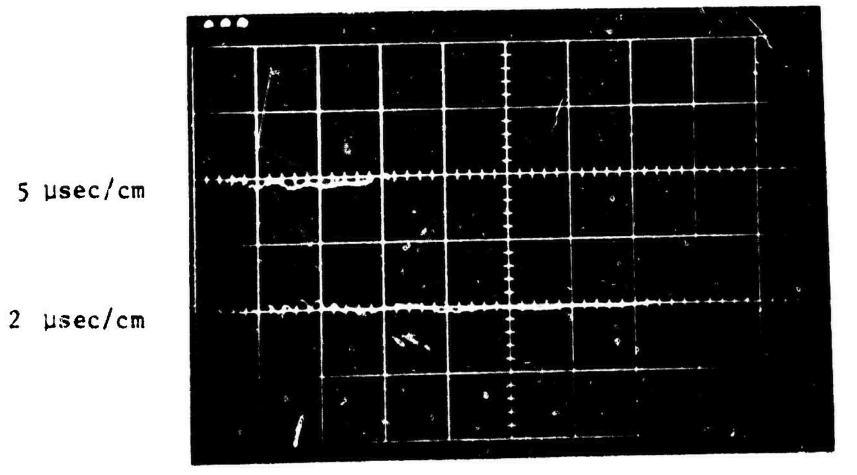


Fig 25e Ionization probe data for M26. 0.851 cm I.D.

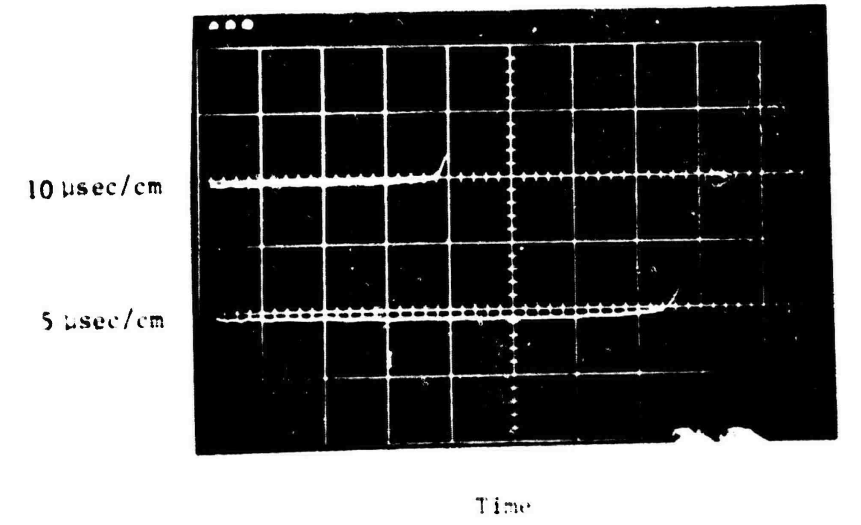


Fig 25f Ionization probe data for M1. 2.57 cm I.D.

Table 23

Velocity calculations for critical diameter tests

Material	Tube inside diam. (cm)	Displacement (m)	Time (μs)	Velocity (m/s)
M1 Strands	4.92	0.203	87.0	2340
	3.52	0.143	54.0	[illegible]
	2.57	0.108	31.8*	[illegible]
	1.85	0.074	22.7*	3260
	1.26	0.089	15.8*	5620
M26 Paste	1.99	0.089	78.0	1140
	1.85	0.074	13.7*	3460
	1.26	0.089	60.0	1480
	0.85	0.048	13.7*	5410
M30 Grains	4.92	0.203	64.0	3170
	3.52	0.143	39.5	3610
	2.57	0.108	32.8*	3290
	1.85	0.074	7.7*	9620
	1.43	0.089	28.2*	3150
	1.26	0.089	29.4*	3020
RDX Slurry	0.62	0.025	4.5	5490
	0.38	0.033	6.7	4920

* Measured using ion probes; time adjusted for booster as follows: C4 detonation velocity = 8040 m/s (no confinement, hand tamped, 2.54 cm diameter, 1.59 gm/cc density); time adjustment = Δt_b = booster length/8040; actual time = measured time - Δt_b.

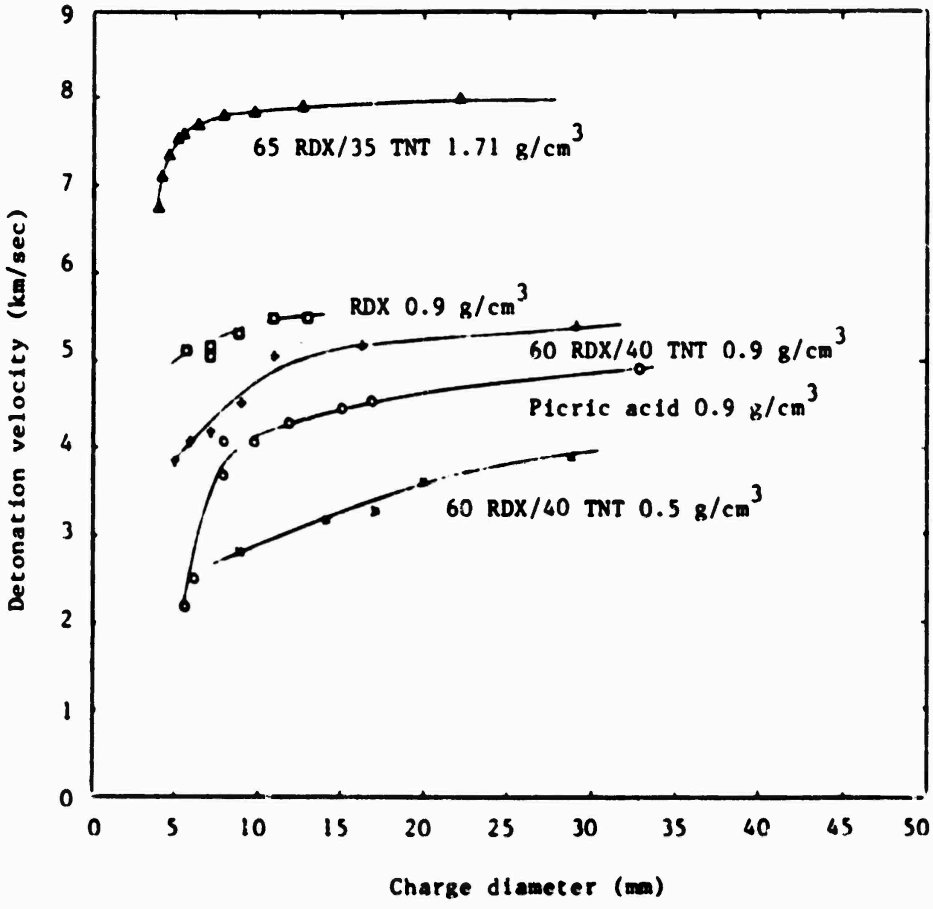


Fig 26 Detonation velocity versus charge diameter for unconfined charges (from Ref 40)

charge diameter. These data are for unconfined explosives but the curve for encased explosives should follow the same trends. The data produced during the present program using continuous probes are plotted in Fig 27. The ion probe data exhibited inconsistent fluctuations and were judged to be less reliable than the continuous probe data. It is seen that the propellants (M1, M26 and M30) seem to exhibit an opposite trend compared to the secondary explosive RDX. However, no conclusions can be drawn since there are only two experimental points for each of these materials.

Test Results: Critical Height

Fourteen tests were conducted to determine critical height. The same type of tubes were used as in the critical diameter tests. The test results are given in Table 24. It was evident in some cases that when a burning reaction started and built up sufficient pressure, the remainder of the charge not yet involved in the chemical reaction was blown out of the open end of the tube. This condition is indicated in the data table as "burned — not fully consumed". Sufficient tests have not been completed on all the materials to define a critical height. In summary, however, the following results were obtained:

Material	Diameter (cm)	Critical height (cm)
M1 strands	4.92	> 59
M1 strands	1.99	> 89
M26 paste	2.57	33
M26 paste	1.99	53
M30 grains	6.35	> 99
M30 grains	4.92	> 58
RDX slurry	0.63	8

These values depend on pipe wall thickness. It is seen that the critical height decreases with the increasing charge diameter for the M26 propellant, and possibly also for the M1 strands.

Recommendations: Transition Tests

It is recommended that a much larger test program be undertaken to clarify the effects of the numerous variables in the critical diameter and critical height tests. Detonation velocity measurements are suggested for every test. A factorial test plan is recommended to minimize the actual number of tests to determine the relationships between

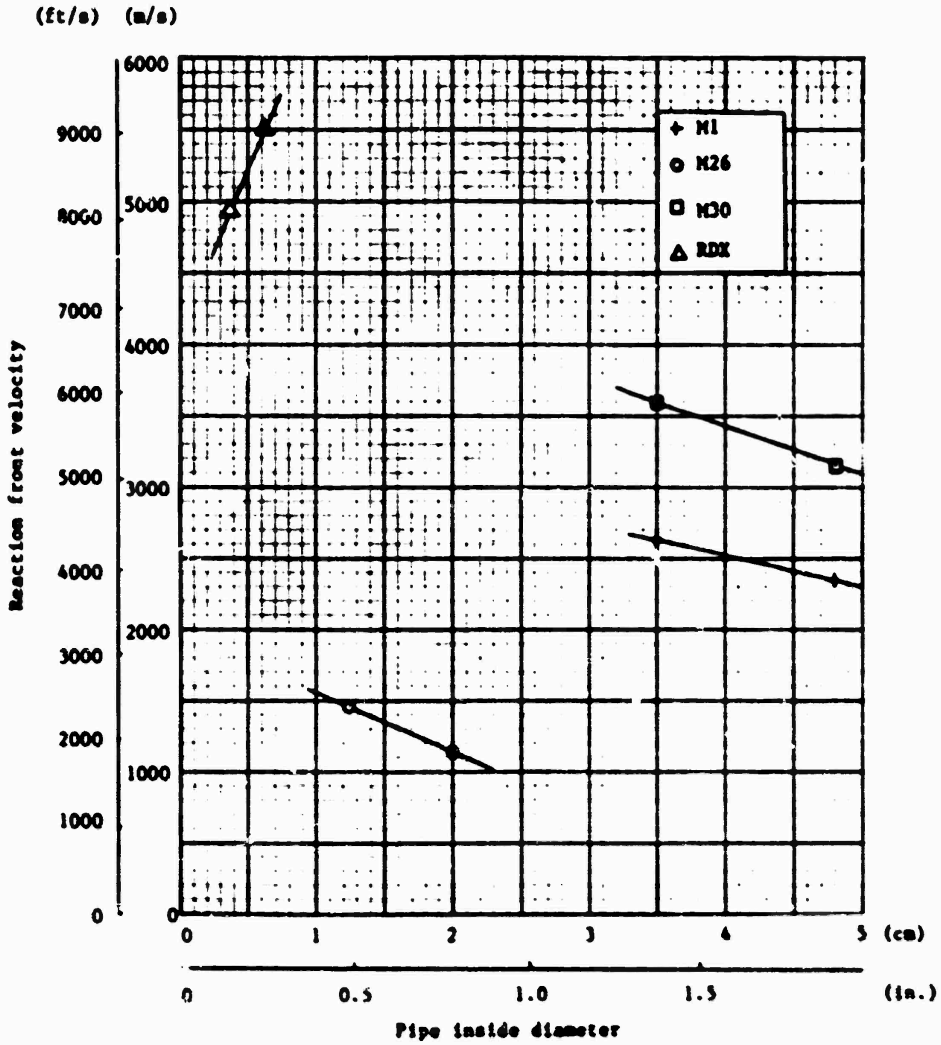


Fig 27 Reaction front velocity versus pipe inside diameter(from continuous resistance probe data)

Table 24

Critical height test data

Material	Tube inside diameter (cm)	Black powder weight (gm)	Charge length (cm)	Bulk density (gm/cm^3)	Results
M1 strands	4.92	12	58	0.321	Burned, not fully consumed (tube Horizontal)
	4.92	12	58	0.401	Burned, not fully consumed (tube vertical)
	1.99	12	89	0.659	Burned, not fully consumed
M26 paste	2.57	12	41	0.808	No initiation, squib malfunctioned
	2.57	12	41	0.412	Burned, all consumed
	2.57	12	91	0.695	Low order reaction 2.5 to 7.6 cm, detonation at 33 cm
	1.99	12	89	0.424	Low order reaction 2.5 to 53 cm, detonation beyond 53 cm
M30 grams	6.35	12	99	0.770	Burned, not fully consumed
	6.35	12	99	0.844	Burned, not fully consumed
	4.92	12	57	0.573	Burned, fully consumed
RDX slurry	0.62	0.27	7.0	0.974	No initiations, not enough black powder
	0.62	0.38	6.7	0.955	No initiations, not enough black powder
	0.62	1.0	7.3	0.830	Started detonation at 2.9 cm
	0.62	1.0	20.0	1.049	Low order reaction at 2.9 to 8 cm, detonation beyond 8 cm.

critical dimensions, detonation velocity, density, charge diameter, casing thickness and booster size. Wide ranges of each parameter are recommended to cover all possible conditions that might exist in the in-process environment.

Impact Test

The objective of this test is to determine the energy density necessary to initiate a material by impact. According to the accident analyses, impact was shown to be a primary cause of in-process accidents. Accordingly, a test to determine the sensitivity of in-process materials to impacts that might occur in an accident situation is imperative. Impacts caused by dropping hand tools or the impact of elements within process machinery are two examples of such in-process stimuli.

As outlined in Table 15 for the four test explosives evaluated in this pilot test series, the hazards classification impact sensitivity criteria are listed:

M1 extruded strands pressing operation	3.3×10^4 J/m^2
M26 premixed paste mixing operation	3.34×10^4 J/m^2
M30 air-dried pellets drying operation	0.69×10^4 J/m^2
RDX slurry conveying operation	1.57×10^4 J/m^2

These are the energy levels to which the material could be exposed in the specified process operation. Therefore, the material sensitivities should be well below these levels.

Test Description

Many test machines have been constructed to evaluate the relative sensitivity of materials to impact. The basic operation of these machines, however, is very similar. Usually a small sample of material (about 35 mg) is placed on an anvil, or in a cup on an anvil. A hammer of known mass is raised to a predetermined height above the sample and is released. The falling hammer or drop weight impacts the sample either directly or indirectly through one or more intermediate strikers.

These machines have a common problem of not producing quantifiable results that can be correlated with accident stimuli or between different machines. Each machine is unique with regard to materials of construction. The drop weight and its height for 50 percent probability of ignition give ambiguous results for engineering usage. The output

should be in quantifiable terms, such as energy density (J/m^2), energy (J),or rate of energy transfer (J/s). With data in these forms, results of various machines could be correlated better.

The impact machine used in the present tests was designed to provide useful output for hazards classification and hazards analysis. The basic features of the impact machine are shown in Fig 28b. Drop heights up to about 2 m are attainable. A piezoelectric-type force gage is mounted on the anvil directly under the sample cup and striker. The cup, striker, and intermediate weight are identical to those of the Bureau of Mines impact machine. An electromagnet lifts the drop weight to the desired height which is indicated by markings on the drop tube and cable. The windlass assembly was designed so that one rotation of the windless crank raises the drop weight 30 cm. The weight can be fixed at 2.5 cm intervals. All impact parts are heat treated to a Rockwell hardness of 55 or greater.

Since the sample is placed in a cup, materials of almost any consistency can be tested (except liquids). Liquids respond much differently than solids to the impact stimulus. This necessitates using a separate fixture for liquid impact testing.

The test measurement is the force the sample material feels as a function of time. A typical data record is shown in Fig 29, which is a photograph of an oscilloscope trace. The vertical axis is calibrated in units of force and the horizontal axis represents time. With the impact area known, the pressure-time history can be calculated (pressure equals force per unit area).

Several indicators are available for determining whether or not ignition has occurred. The force-time record frequently shows a spike in the trace if ignition occurred,or ignition is detected by audio (sound), visual (smoke, char, flash or flame), or infrared analysis (decomposition products). Tarnishing of the polished striker surface is frequently an indication of ignition.

Analyzing the force trace from the piezoelectric gage will produce the desired quantities:

- energy
- rate of energy transfer, and
- energy density.

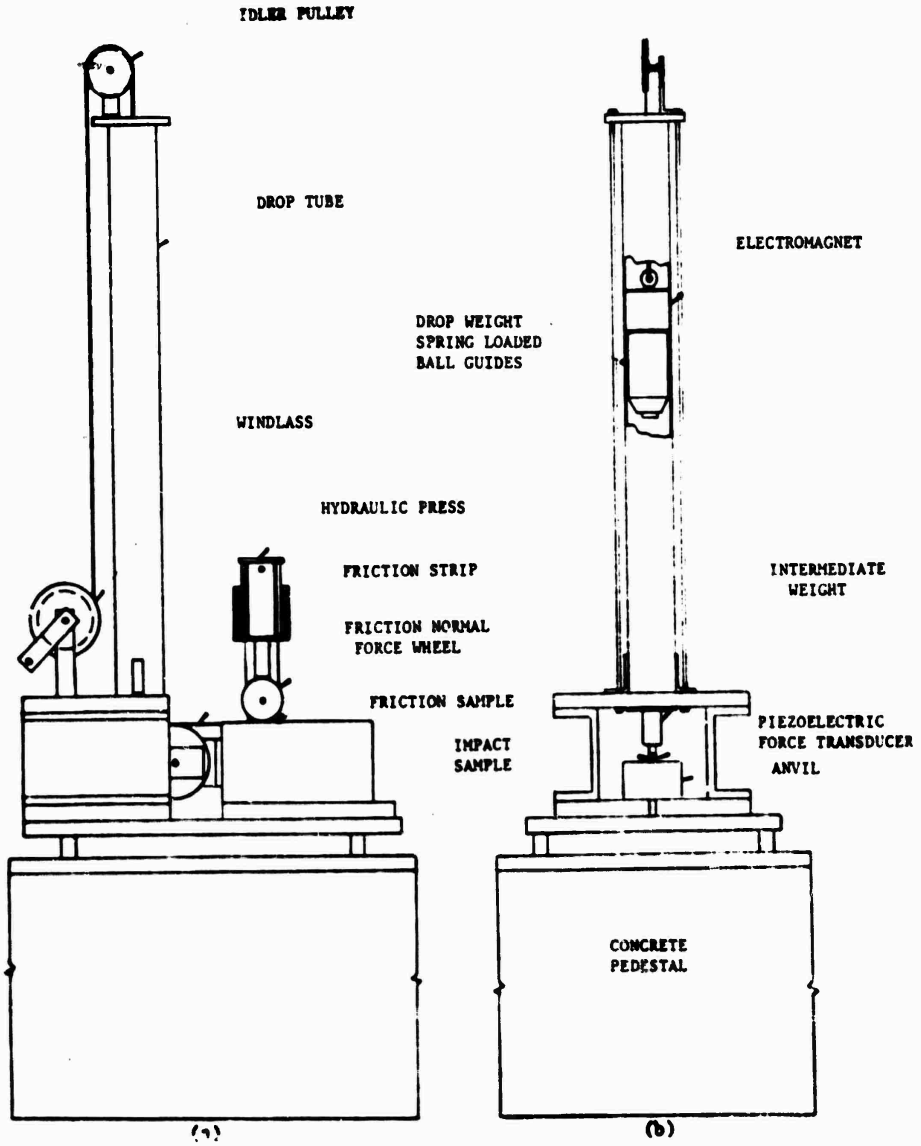


Fig 29 Impact test machine
(Including friction test adapter)

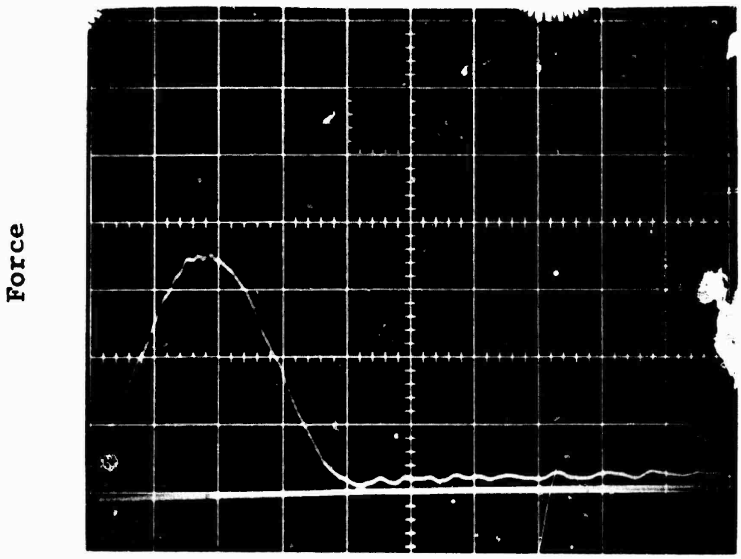
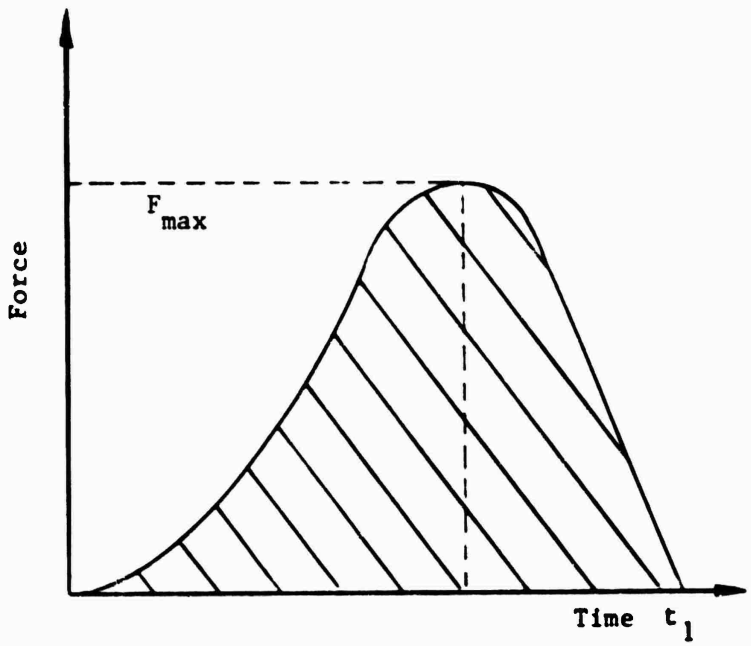


Fig 29 Impact test data of force versus time (no explosive in cup)



Shaded area equals positive impulse: Area = I = $\int F dt$

Fig 30 Analysis of impact sensitivity test data from force vs time measurements

The energy is determined from the area under the force-time curve. This area is indicated in Fig 30 and is equal to the positive impulse, I.

$$\text{Area} = I = \int_0^{t_1} F\,dt \tag{19}$$

Energy, E, is calculated by:

$$E = \frac{I^2}{2m} \tag{20}$$

where m is the drop mass. The rate of energy transfer, $\dot{E}$, is simply:

$$\dot{E} = \frac{E}{t_1} = \frac{I^2}{2m\,t_1} \tag{21}$$

The energy density, ε, is calculated as:

$$\varepsilon = \frac{E}{A} \tag{22}$$

where A is the area of the striker pin contacting the sample.

Two drop weight sizes were used with the present impact apparatus: small weight, 2.21 kg and large weight, 4.42 kg. The impact area was always the same. The striker pin diameter was 0.64 cm. The impact area was therefore 0.32 sq cm.

Test Procedures

Ambient temperature for testing should be 25 $\pm$ 5° C and the relative humidity less than 40 percent. For granular materials the sample size should be 35 $\pm$ 1 mg and should be tested "as received" (or in the actual in-process state). For cast, molded, extruded, and injected explosives, each specimen should be formed into a pellet or wafer not less than 0.64 cm (0.25 in) in diameter and 0.064 $\pm$ 0.025 cm thick. The sample in any form should be uniform over the total area of the sample cup and be less than 100 mg. Unfortunately, pelletizing the test material usually changes the in-process form of the material, either by changing its geometry, its density, moisture content, percent volatiles, etc. Thus it is questionable as to how meaningful this test is, when the form of the material is changed.

Of the four pilot test materials in this program, three were formed into pellets, (M1, M26, M30). A small press was used to press these materials to the desired shape. Pressures between 3.5×10^6 and 2.1×10^7 Pa were sufficient to make a uniformly appearing sample.

Since many in-process materials contain volatile solvents, testing should be conducted without undue delays between sample preparation and the time of impact. For many in-process forms, however, large changes in volatility occur during sample preparation.

The material sample wieght and description, drop weight height, and force-time trace should be recorded for each trial along with the test result (reaction or no reaction).

The force gage and striker pin should be cleaned after each trial. If the striker pin becomes pitted or roughened it should be removed, reground, and polished. A new sample should be used for each trial.

Testing should commence at a high enough drop weight height to cause initiation of the sample. A Bruceton or "up-down" test sequence should then be used to find the 50 percent ignition probability. The statistical method for determining the 50 percent ignition energy is detailed in Refs 41, 42 and 43.

Test Results

Before testing, the perpendicularity of the drop tube relative to the gage surface was checked. The drop tube was first aligned with a level. Then a piece of white paper with a piece of carbon paper overlaid was placed under the striker pin on the gage surface. The drop weight was then raised and released. The carbon imprint on the paper was observed. A uniformly darkened area indicated the machine was satisfactorily aligned.

Initial tests were conducted on RDX that had been dried in a dessicator. These initial tests emphasized the importance of the sample location in the sample cup. Table 25 shows the results for RDX. A sample piled in the middle of the cup showed a greater sensitivity to impact.

Additional impact test results are listed in Table 26. Preliminary tests on the three propellants also showed the importance of sample preparation. No reactions were obtained

TABLE 25

RDX impact test data

Sample description	Sample weight (mg)	Drop height (m)	Results
Dried granules, uniformly distributed in cup	25	0.30	No reaction
Dried granules, uniformly distributed in cup	25	1.52	No reaction
Dried granules, uniformly distributed in cup	25	1.52	Reaction
Dried granules, piled in center of cup	25	1.52	Reaction
Dried granules, piled in center of cup	25	0.91	Reaction
Dried granules, piled in center of cup	25	0.30	Reaction

Table 26

Propellant impact test data

Material	Sample description	Sample weight (mg)	Drop height (m)	Results
M1	Cut segments	119	1.52	No reaction
M1	Pressed at 3000 psi	100	1.52	No reaction
M1	Pressed at 3000 psi	44	1.52	Very slightly browned
M1	Pressed at 3000 psi	27	1.52	No reaction
M1	Same sample	27	1.52	Beginning of melting at one spot
M1	Pressed at 3000 psi	54	1.45	Burnt
M1	Pressed at 3000 psi	86	1.22	Melted, browned, smoke
M1	Pressed at 3000 psi	88	1.14	Melted, browned, smoke
M1	Pressed at 3000 psi	121	0.91	Melted, browned, smoke
M1	Pressed at 3000 psi	98	0.76	Melted, browned, smoke
M1	Pressed at 3000 psi	88	0.69	No reaction
M1	Pressed at 3000 psi	77	0.69	Melted, browned, smoke
M1	Pressed at 3000 psi	83	0.61	Browned, smoke
M1	Pressed at 3000 psi	73	0.53	Browned, smoke
M1	Pressed at 3000 psi	81	0.46	Melted, browned, smoke
M1	Pressed at 3000 psi	105	0.38	No reaction
M1	Pressed at 3000 psi	104	0.38	No reaction
M1	Pressed at 3000 psi	58	1.22	Melted, browned
M26	Pressed at 400 psi	24	1.52	No reaction
M26	Pressed at 500 psi	26	1.52	No reaction
M26	Pressed at 500 psi	21	1.52	No reaction
M26	Not pressed	25	1.52	No reaction
M26	Not pressed	76	1.52	No reaction
M26	Pressed at 500 psi	24	1.52	No reaction
M26	Pressed at 400 psi	26	1.45	Flash, burnt
M26	Pressed at 400 psi	31	1.14	No reaction
M26	Pressed at 400 psi	21	1.37	No reaction
M26	Pressed at 400 psi	11	1.45	No reaction
M26	Same sample	11	1.45	No reaction

Table 26 (cont)

Propellant impact test data

Material	Sample description	Sample weight (mg)	Drop height (m)	Results
M26	Pressed at 400 psi	50	1.45	Flash, burnt
M26	Pressed at 400 psi	29	1.37	Flash, burnt
M26	Pressed at 400 psi	44	1.30	Flash, burnt
M26	Pressed at 400 psi	30	1.22	Flash, burnt
M26	Pressed at 400 psi	31	1.14	Flash, burnt
M26	Pressed at 400 psi	47	1.07	Flash, burnt
M26	Pressed at 400 psi	49	0.99	Burnt smell, no flash
M26	Pressed at 400 psi	38	0.91	Pin burnt
M26	Pressed at 400 psi	53	0.43	No reaction
M26	Pressed at 400 psi	65	0.61	Flash
M26	Pressed at 400 psi	54	0.53	No reaction
M26	Pressed at 400 psi	51	0.56	Flash, burnt
M26	Pressed at 400 psi	24	0.61	Burnt spot and smell, no flash
M30	Thin slice	31	1.52	No reaction
M30	Thin slice	74	1.52	No reaction
M30	Thin slice, pressed at 500 psi	39	1.52	No reaction
M30	Thin slice, pressed at 500 psi	16	1.52	No reaction
M30	Pressed at 500 psi	45	0.91	Burnt, no flash, smoke
M30	Pressed at 500 psi	70	0.91	Burnt, no flash, smoke
M30	Pressed at 500 psi	48	0.38	No reaction
M30	Pressed at 500 psi	46	1.22	Burnt, no flash, smoke
M30	Pressed at 500 psi	53	0.61	Burnt, no flash, smoke
M30	Pressed at 500 psi	62	0.53	No reaction
M30	Pressed at 500 psi	57	0.53	Burnt, no flash
M30	Pressed at 500 psi	17	0.46	No reaction
M30	Pressed at 500 psi	75	0.46	No reaction
M30	Pressed at 500 psi	37	0.61	No reaction
M30	Pressed at 2000 psi	35	1.45	Flash, burnt

if the sample was tested as received in the actual in-process state. The materials had to be pressed to a uniform condition before reactions were achieved. Thus it is questionable if the test results represent the behavior of the actual in-process material. The lowest height at which reactions were observed for each material are listed:

RDX (dried)	30 cm
M1 strands	46 cm
M26 paste	56 cm
M30 grains	53 cm

At the time these data were collected, no force-time measurements were made. Therefore, no actual energy, energy density, or rate of energy transfer calculations could be made. However, maximums of these quantities can be calculated based on the potential energy of the drop weight at the heights indicated. The equation for energy is

$$E = W \times H \tag{23}$$

where W is the drop weight and H is the height above the sample from which the weight was released. The energy density also can be calculated knowing the striker pin diameter. These calculations were made and are listed below.

Material	Calculated Energy (J)	Calculated energy density (J/m^2)
RDX	13.2	4.17×10^5
M1	19.8	6.25×10^5
M26	24.2	7.64×10^5
M30	23.1	7.29×10^5

A comparison of these data with the impact sensitivity criteria discussed earlier (Table 15) shows that all the materials required greater stimuli than those given by the criteria. This indicates that these materials are less sensitive than the criteria set for them. Therefore no penalty would be levied on these materials in the classification procedures. Obviously the method for calculating energy density based on potential energy will give sensitivities much too low. It may be that only a fraction of the energy available is transferred to the explosive and this depends on the materials used in constructing the apparatus and on their hardness. More accurate results should be based on the measurement of the actual forces felt by the explosive sample.

Recommendations

It is recommended that further tests be done on this impact test apparatus or other fixtures to prove the validity of the impact procedures for hazards classification. In many situations the test sample must be significantly altered to conduct the small-scale impact test. In these cases there is serious doubt if the test results are meaningful. Whenever the sample must be physically or chemically altered from its in-process form, the small-scale test should not be used and a special test should be designed to allow testing of the in-process form of the material.

Friction Test

The objective of this test is to determine the liklihood that a material will ignite when subjected to friction. According to the analyses of accidents, friction is one of the primary causes. Friction tests are required to determine the friction sensitivity of the material.

The friction tests are designed to simulate frictional forces between moving components as, for example, in a sigma blade mixer, or during machining and material handling. The Thiokol strip friction tester for powders and propellants was designed to duplicate friction environments occurring in processes such as scraping of a drum on a floor contaminated with scraps of oxidizer and propellant. The friction testers also serve a second purpose. They can be used for evaluating a material's sensitivity to materials of construction, surface finishes, slide length, contact area, velocity, and in some cases the coefficient of friction.

The friction sensitivity criterion for hazards classification was determined from the analysis of accidents. This criterion depends on the process operation:

Mixing	4.45×10^8 N/m^2 at 2.4 m/s
Pressing	5.66×10^8 N/m^2 at 2.4 m/s
Drying, Screening, Filling, Conveying	5×10^8 N/m^2 at 2.4 m/s

If the sensitivity value of [illegible] is below the critical value, a penalty factor is levied on the classification procedure. If the friction sensitivity value is greater than the criterion, no penalty is assessed.

The friction test should be conducted on all materials. For the four materials tested in this program the sensitivity criteria are:

1) M1 extruded strands; pressing operation — 5.66×10^8 N/m^2 at 2.4 m/s
2) M26 premixed paste; mixing operation — 4.54×10^8 N/m^2 at 2.4 m/s
3) M30 air dried pellets; drying operation — 2.55×10^8 N/m^2 at 2.4 m/s
4) RDX slurry; conveying operation — 2.55×10^8 N/m^2 at 2.4 m/s

Test Description

As discussed in Appendix B, the strip friction-type test best fits the requirements for hazards classification. A test apparatus was therefore built based on this concept.

The basic friction test apparatus is shown in Fig 31. Its operation is as follows: The metal friction slip slides in a 1.27 cm wide slot in the friction block. The normal force is applied through the stationary force wheel by a hydraulic press. The fluid pressure exerted on the press ram is calibrated to give the force. The linear motion of the friction strip is initiated by a drop weight impacting the arm attached to the rotating wheel. A potentiometer is mounted to the rotating wheel which provides an electrical signal for determining the linear velocity of the friction strip. The area is based on the total area of the friction strip pressed beneath the force wheel.

The sliding strip friction machine was primarily designed to accomodate samples in the powdered, granulated, paste, or solid state. Liquids are a separate issue requiring a friction apparatus for producing viscous effects.

Test Procedure

The friction block temperature should be 25 $\pm$ 5°C. Recommended sample weights should be of the order of these values:

Secondary explosives	25 mg
Propellants	100 mg

The force wheel, friction strip, and friction block should be

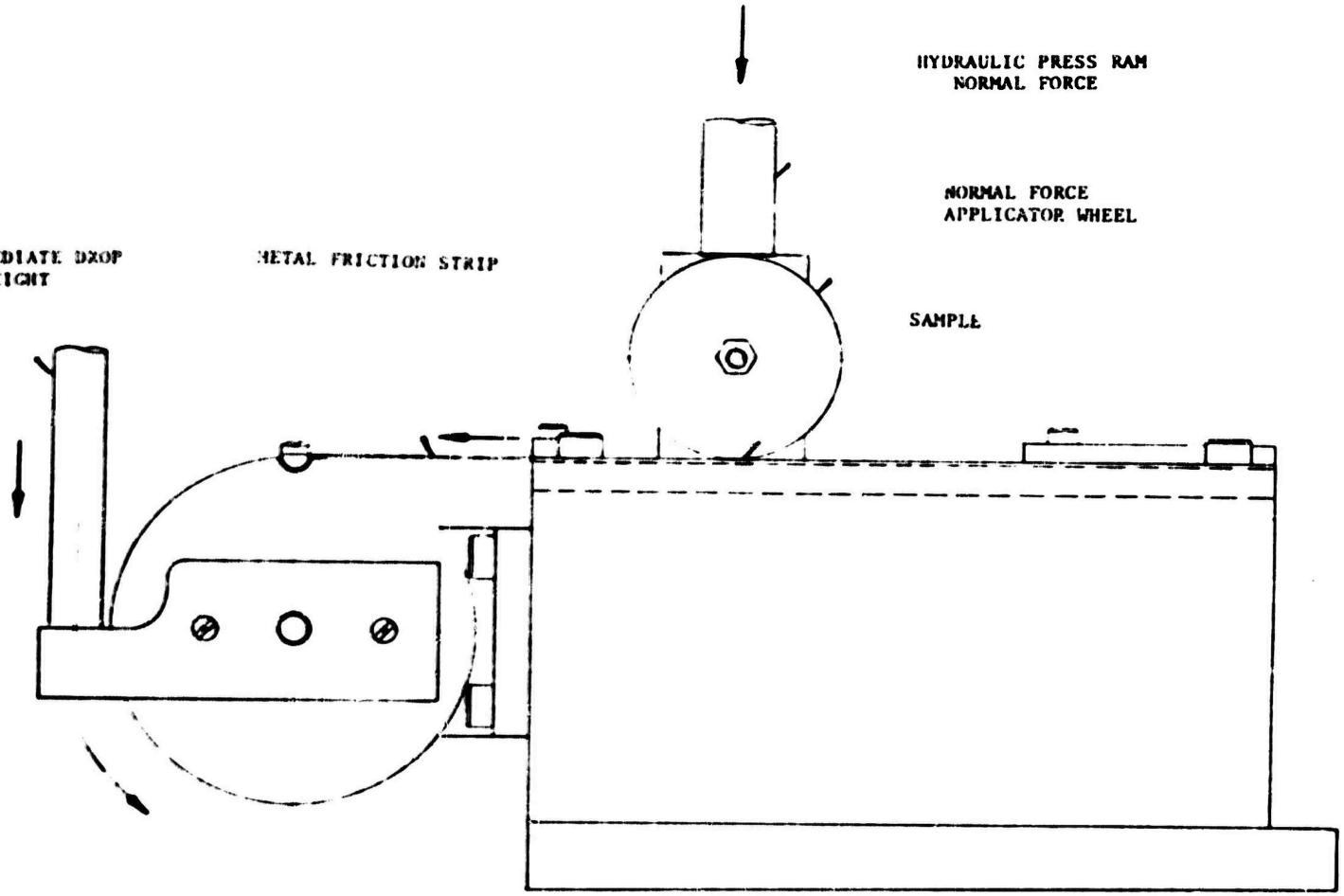


Fig 31 Friction test machine

thoroughly cleaned with solvent. Testing should begin at a high drop-weight height and normal force. The normal force should be maintained constant while varying the drop-weight height and resultant sliding velocity. A Bruceton or "up-down" test sequence should be used to find the 50 percent ignition velocity at a set normal force. Once this is completed, the normal force should be increased or decreased and the Bruceton test sequence repeated.

The data that should be recorded for each test are the maximum velocity, normal force, and the area swept out by the sliding friction strip. The area is simply the total linear travel of the strip times its width. Sample size and method of preparation also must be documented.

Initiation of the sample is detected by a burnt smell, loud sound, crackling sound, or by visually observing a flash or charred friction surface.

Test Results

Preliminary test data have been collected on the M30 and M26 propellants, and the RDX explosive. These data are presented in Table 27. The M30 pellets were cylindrical, with a pattern of small axial holes in each pellet. This form did not lend itself to the small-scale testing, so a slice of a pellet was taken and pressed at various pressures. The pressed material was then cut to the desired shape, 1.27 cm wide by either 0.64 or 1.27 cm long. Pressing the samples between 10.34×10^6 and 13.79×10^6 Pa was adequate. The sample weight varied depending on its length and the pressing force. The maximum drop height of 1.43 m and weight of 4.54 kg were used as starting points to maximize the probability of ignition. No initiations, however, could be attained for the M30 propellant with normal forces up to 9238 N. Generally, the sample was merely smeared under the force wheel. Similar results were obtained for the M26 paste propellant with normal forces up to 7917 N.

The RDX samples were dried before testing to increase the sensitivity. They were tested in this manner merely to try to get an ignition to assure that the apparatus was operating properly. Again no initiations were achieved with normal forces up to 7917 N. The RDX was merely pulverized into a fine powder after passing under the normal force wheel. Sufficient normal forces to cause initiation in the present friction apparatus were probably not achieved.

Table 27

Friction test data

Material	Pressure for pelletizing sample ($P_a \times 10^6$)	Sample length (cm)	Sample width (mg)	Force on sample (N)	Test results
M26 paste	3.45	0.64	61	5278	Smeared
M26 paste	3.45	0.64	69	6599	Smeared
M26 paste	3.45	0.64	63	7917	Smeared
M30 pellet	10.34	1.27	85	3959	Smeared
M30 pellet	11.72	1.27	75	2638	Smeared (~1.27 cm)
M30 pellet	13.79	1.27	75	2638	Smeared (~1.27 cm)
M30 pellet	13.79	1.27	62	5278	Smeared (~1.27 cm)
M30 pellet	13.79	0.64	42	5278	Smeared
M30 pellet	13.79	0.64	30	7917	Strip broke loose
M30 pellet	13.79	0.64	33	9238	Smeared
M30 pellet	13.79	0.64	30	6599	Smeared
RDX slurry	-	0.64	24	530	Pulverized
RDX slurry	-	0.64	27	2639	Pulverized
RDX slurry	-	0.64	28	5278	Pulverized
RDX slurry	-	0.64	26	7917	Pulverized

Constant Parameters: Drop weight = 4 54 kg
Drop height = 1.43 m
Sample width = 1.27 cm

Recommendations

It is recommended that further effort be allocated to investigate and refine the friction sensitivity test apparatus. The normal force achievable on this apparatus is approaching its limit with the values reported herein. Some additional work is necessary to extend this limitation.

For situations where the physical or chemical state of the test material must be significantly altered to utilize the small-scale test apparatus, the results would be highly questionable, if meaningful at all. For such cases a special test, probably much larger in size, should be designed to adequately simulate the actual in-process situation.

Dust Test

The objective of the dust test is to determine if a material has the propensity to create dust in the particular in-process condition being studied. Dust may be defined as a suspension, in air or other gases, of small solid particles ranging in size from 0.05 to 1000 micrometers (Ref 44). The dust, when mixed in the proper proportion with air, can form a combustible or explosive mixture. It is not, however, the objective of the dust test to determine the conditions under which an explosion will occur. The dust test is used to determine if a material will form dust in the first place, and the range of concentrations to be expected.

Dusty atmospheres could occur in process plants, for example, in grinding or conveying operations, particularly with the failure of dust collection systems. The dust test should provide a yes or no indication as to whether or not dusting is a potential problem. The test is not necessary with moist or liquid materials which obviously would not produce dust, or with light fluffy materials where dusting is definitely a problem. However, between these extremes, some dusting criteria are necessary. The dust test is intended to provide a basis for deciding if dust is a problem, and if the dust explosibility tests should be conducted.

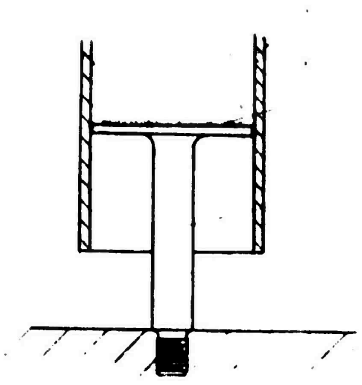
Upon inspection of the four pilot materials received for testing, it was immediately obvious that dusting was not of concern for these materials. The solvent contained in all four materials prevented them from forming any dust.

Test Description

Since no tests are available for determining a material's propensity for creating dust, a new concept was developed. It is desirable that the dust test provide quantitative data on which to base the "dustability" criteria. The concentration of dust in some defined volume will be the indicator of dustability. Two important considerations in designing the dust test are the means used to generate the dust and the volume upon which to base the determination of concentration. These points should in some way be correlated to the actual in-process condition.

The chosen concept was to create the dust either by an acoustically induced vibration or an electromagnetic exciter. The sample would be enclosed in a glass tube and light measuring instrumentation would be used to correlate reductions in light transmission to dust concentration. An apparatus was constructed based on this concept and is shown in Fig 32. An electromagnetic vibration table is used in this case. A piston screws into the vibrating mounting plate and slides freely in a glass or plastic tube. The sample is placed on the top surface of this piston. The piston imparts a uniform velocity to the sample particles. A photometer is used to measure the decrease in light transmission caused by the suspended dust particles. Measurements are to be made at various heights above the average location of the piston surface. The photometer output corresponding to the input vibration amplitude and frequency will characterize the sample dustability.

The mass of dust at a particular height may be assessed by using a light scattering photometer. A near forward, dark field instrument called a Sinclair-Phoenix photometer is commonly used for measuring a wide range of particle sizes. Light from a tungsten filament lamp is focused in a hollow cone. The light is then scattered by dust particles in the tube. Only the scattered light is transmitted to the photomultiplier and is measured. The signal from the photometer can be related to the mass of dust in the viewing volume by calibration. Calibration must be against a material with a known particle size distribution. This is necessary since the particle size distribution of the test material is not known. Figure 33 shows the details of the photometer, which was slightly modified for this application. A special adaptor was constructed so two different tube diameters could be tested (22 and 11 mm) The electromagnetic vibration exciter has limitations of $\pm$ 1.27 cm amplitude and 2 to 3000 Hz frequency range. For higher frequencies an acoustical driver could be used.



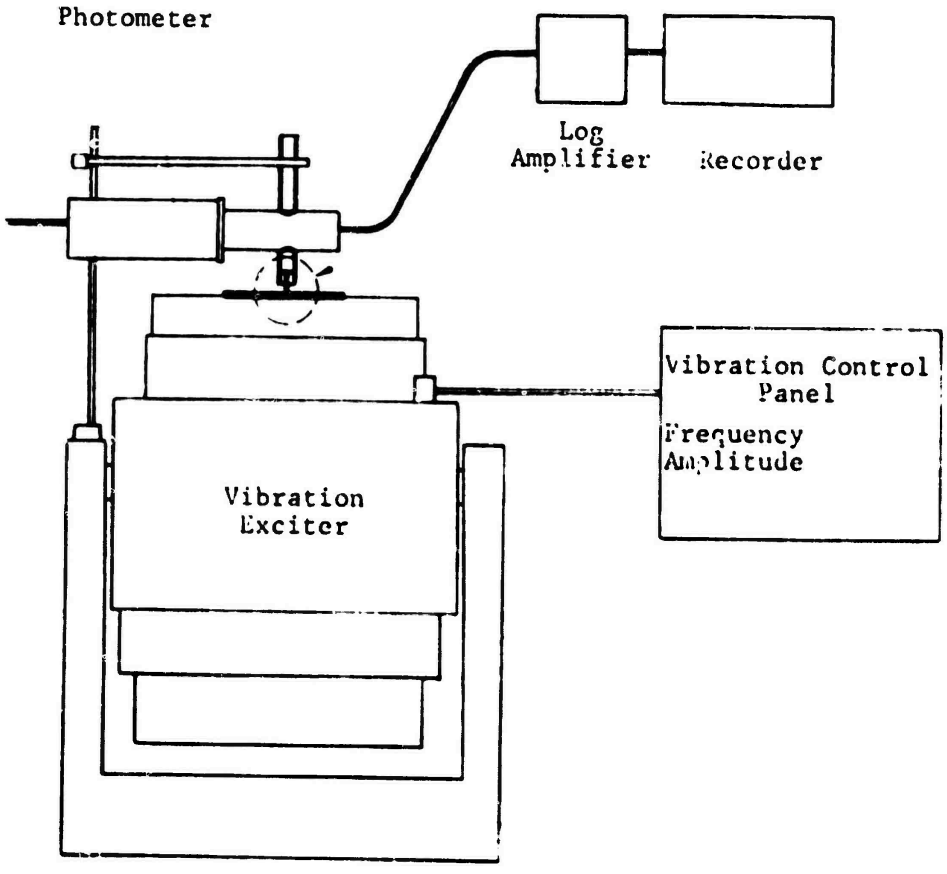


Fig 32 Dust test apparatus

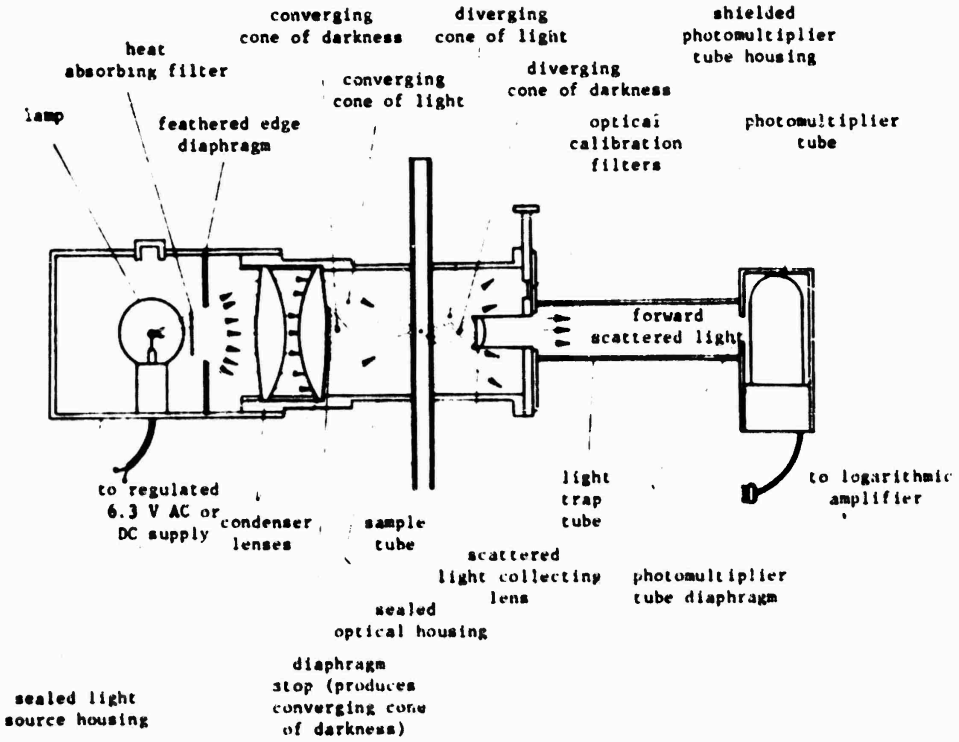


Fig 33 Schematic diagram of Sinclair-Phoenix forward light scattering photometer

The calibration of the instrument should be in four ranges of particle size distribution:

1 - 10 micrometer median size
10 - 30 micrometer median size
30 - 50 micrometer median size
50 - 100 micrometer median size

Candidate materials for the calibration are aluminum spheres or glass beads. It is not necessary to use the vibration table to generate the dust for calibrating purposes. A flow-through arrangement can be used where the dust is pulled or blown through the tube for a known period of time and at various volumetric flow rates. The sample dust should then be collected on a preweighed 47 mm Millipore filter and weighed to determine the total mass. The concentration (or bulk density) is then calculated by the following equation.

$$\text{Concentration} = \frac{M}{Q\Delta t} \tag{24}$$

where M is the total mass, Q the volume flow rate, and Δt the sampling time. The correlation between the photometer signal and dust concentration is then established. For other dust materials, all that is necessary is to measure the signal from the photometer at various heights along the sample tube and then consult the calibration curve to find the dust concentration versus height. The calibration signals, however, are likely to depend somewhat on the particle size distribution used. That is why four different particle size ranges are recommended. It is then necessary to examine by microscopy the actual dust samples to be tested to determine what particle size calibration curve to use.

With the determination of the concentration versus height curves at various vibration frequencies and amplitudes, "dustability" criteria could be established. Some possible approaches for determining these criteria are outlined below.

For a range of defined frequencies and amplitudes of vibration, the minimum 50 percent signal intensity height should be determined. This is shown in Fig 34. For the schematic curves in Fig 34, the minimum 50 percent signal intensity height would be the height corresponding to point one. If the height does not appear to be an important factor, the actual concentration at the 50 percent level could be the basis for the criteria. The 50 percent level was chosen arbitrarily. A lesser or greater level may prove to be more accurate.

$$\frac{I}{I_o} = \frac{\text{Photometer signal with vibrating dust}}{\text{Photometer base signal no dust vibration}}$$

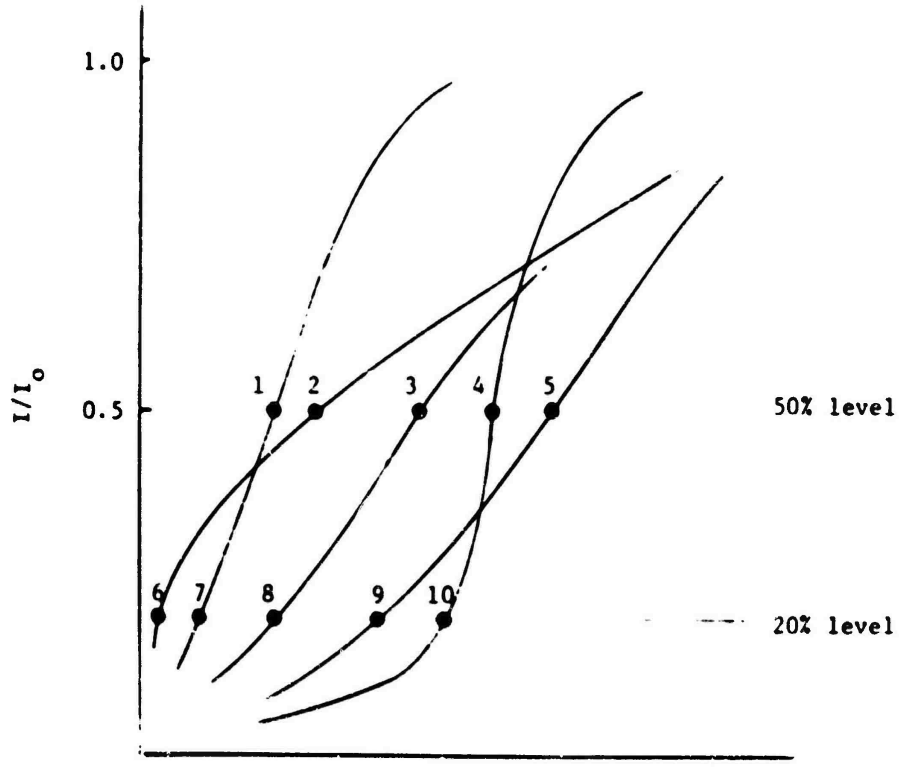


Fig 34 Schematic diagram of dustability test curves

Another possibility is to select a specific height. If the material generates dust above a certain minimum concentration at that height for any realistic frequency and amplitude of vibration, then it is termed "dustable". These are two of many possible methods for determining the dust criteria. The guide behind the method should be that:

(1) The materials at the extremes of dustability correlate with the criteria; i.e., light fluffy materials show a high dustability; wet or moist materials show low dustability.

(2) The dustability criteria be based on, or be correlated to actual in-process conditions. For example, if the laboratory dust test uses the dust concentration as a criterion of pass/fail, it would be desirable if the concentration in the test correspond to the concentrations experienced in the process plant for that material.

Recommendations

The apparatus for the dust test proposed here has been constructed. No testing on propellants or explosives, however, has been accomplished. It is recommended that further development effort be allocated to prove the concepts proposed here. Calibration and testing of a range of materials should be considered. Also, to establish reliability of the dustability criteria and to place them on a reasonable basis, a method of correlating the dust test data to in-process dust conditions should be investigated.

Dust Explosibility Test

The objective of the dust explosibility test as used for hazards classification is to determine the minimum explosive dust concentration in air, and the minimum ignition energy. Dusts can burn, deflagrate, or even detonate depending on the ignition, concentration, and confinement. This test is designed to determine the minimum concentration of dust at which ignition will occur, and the ignition energy required to ignite a dust cloud that is above the minimum concentration. Both data are important for assessing the hazards associated with dusts.

This test is conducted only when the dust test has shown that dusting is a potential problem. The data derived from the test will indicate whether a dust collection system is needed to reduce the dust concentrations, and to determine electrostatic discharge (ESD) energy that would be required to ignite the dust. The data should also be used to establish a criterion for dust explosibility.

The four materials received for preliminary testing were not "dustable". Therefore, the dust explosibility test was not required for these materials in the process state they represent. The materials, however, do contain various percentages of volatile solvents which may create a vapor cloud. This hazard is not within the scope of the dust explosibility test.

Test Description

The Bureau of Mines "Hartmann" Dust Explosibility Test Apparatus (Ref 45) is used for this test. Basically, a suspended dust is created by dispersing the material into air with a gust of gas. A continuous spark is used for the tests to determine flammable concentrations; then, using flammable concentrations, the electrostatic energy stored in a capacitor is used to determine the ignition energy. The apparatus is shown in Fig 35. A momentary dust cloud is produced within the chamber by directing a blast of air on the sample. In one series of tests, the dust concentration is varied while using a continuous high-voltage high energy spark. This allows the determination of the minimum concentration at which the cloud will ignite. In the other series of tests, a set concentration greater than the threshold ignition concentration is used. Different levels of energy stored in the capacitor are used to determine the minimum energy needed to ignite

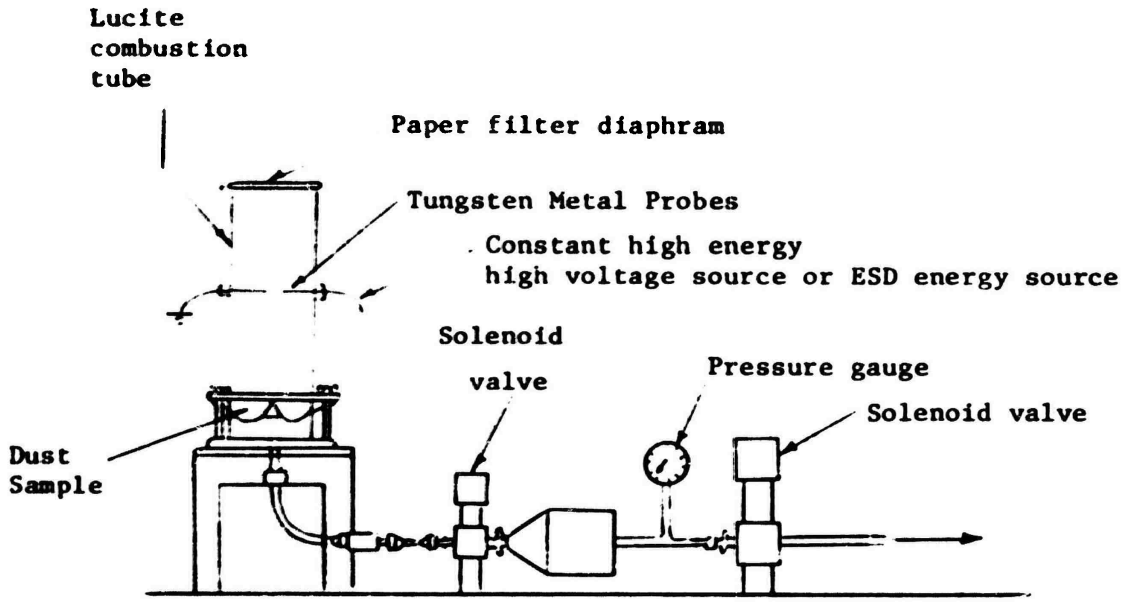


Fig 35 Hartmann dust explosibility test apparatus

the dusts. The gas used in this test can be varied in order to simulate an inert gas operation. This apparatus also has the capability of measuring the rate of pressure rise which gives an indication of the severity of the dust explosion. Some data that have been collected on explosive compounds by other researchers are shown in Table 28.

As pointed out in Ref 47, the data obtained by this test apparatus and method are specific to the materials as tested and are not to be considered "inherent , fundamental properties". Correlation with other tests has not been established. The results are therefore only relative indicators of dust explosibility. This may make it difficult to correlate the data from the test to actual in-process conditions. However, a hazards classification criterion based on relative data may still be of value though it may not be used for hazards analysis.

The explosibility index may provide a basis for hazards classification. This relative index is defined to be unity for Pittsburgh coal. Descriptive objectives for other index values are listed below (ref 46).

Description	Explosibility Index
weak	<0.1
moderate	0.1 - 1.0
strong	1.0 - 10
severe	>10

Recommendations

As a preliminary measure it is recommended that the apparatus and procedures developed for the Hartmann Dust Explosibility Test be adopted and the Explosibility Index be incorporated into the hazards classification procedures.

It is recommended that the cutoff criterion for the explosibility index be at 0.1. This means that if the test data show that the explosibility index is greater than or equal to 0.1, the material in that process operation should be penalized as outlined in the Preliminary Hazards Classification Procedures previously described.

The explosibility index criterion of 0.1 is a reasonably safe cutoff point. However, the criterion may be placed on a more rational basis if a correlation can be shown between the explosibility index and the accident data. This analysis

Table 20

Ignition and explosion data for explosives and related compounds

Material description	Sample number	Moisture content (%)	Particle size* (μ)	Minimum ignition energy of cloud (joules)	Minimum explosive concentration (kg/m^3)	Explosibility index
Aluminum 35%, atomized (average 30 microns) and barium nitrate 65% (average 15 microns)	1594	-	44	0.120	0.200	9.2
Aluminum grade B, 40% (average 34 microns); barium nitrate 30% (average 50 microns); potassium perchlorate (average 10 microns)	1595	-	74	0.375	0.270	< 0.1
Zirconium-magnesium alloy, 65% (60% finer than 74 microns); barium nitrate, 35% (95% finer than 74 microns)	1596	-	74	-	0.430	-
Ammonium nitrate-fuel oil mixture (94% ammonium nitrate, from 10 to 2000 microns; 6% No. 2 fuel oil)	23018	5.6	840	-	2.000	-
Ammonium nitrate-fuel oil mixture (90% ammonium nitrate, from 10 to 2000 microns, 10% No. 2 fuel oil)	23018C	0.1	840	1.600	0.370	-
Ammonium nitrate No. 3, 78%; sodium nitrate, 20%; starch, 2%, wood pulp, 2%.	1777	-	740	0.304	-	-
Ammonium picrate, 60%, sodium nitrate, 40%, resin binder, 5%	900	-	74	0.160	0.200	-
Black powder mixture (91% No. 4 grade & black powder, 9% aromex)	1099	-	149	0.320	0.120	-
Dinitrobenzamide	2296	0.2	74	0.065	0.060	7.2
Dinitrobenzamide acid	2295	0.1	74	0.065	0.040	4.0

* Particles pass given sieve size

Table 28 (cont'd)
Ignition and explosion data for explosives and related compounds

Material description	Sample number	Moisture content (%)	Particle size* (μm)	Minimum ignition energy of cloud (joules)	Minimum explosive concentration (kg/m³)	Explosibility index
Diphenylamine-formaldehyde condensation product	1064	0.3	74	0.060	0.095	0.7
Dinitrotoluamide (3,5 dinitro-o-toluamide)	2159	0.1	74	0.015	0.050	> 10.0
Napalm (70% ferro-napalm, 16% gelled napalm, 6% caustic caliche magnesia)	2121	0.0	74	0.060	0.020	-
Nitroguanidine	1037	-	840	>7.200	-	-
Nitroguanidine	1036	-	250	>7.200	-	-
Nitrostarch	962	-	840	0.060	0.070	-
Pentaerythritol tetranitrate, 90%; antimony sulfide, 10%, dextrin, 2%	916	-	840		0.070	
Lithium, 20%; on binder; magnesium grade B, 20%; lead chromate, 60%	1061	-	840	8.000	0.265	-
Silicon, 30%; lead oxide, (red lead) 90%	1016	-	840	0.360	>3.000	< 0.1
Silicon, 6%; manganese, 40%; lead oxide (red lead), 54%	1036	-	74	0.350	>3.000	-
Silicon, 26%; aluminum, 13%; ferric oxide, 22%; potassium nitrate, 35%; carbon, 4%	914	-	840		0.650	-
Thermite (26% aluminum, 72% iron oxide), 14%; pine igniter--barium nitrate, 20%; aluminum fine grain, 17%, sulfur, 2%, castor o l, 0.2%	1006	-	840	0.260	0.760	-
Tetrazotetralene	662	-	840	0.075	0.070	-

* Particles pass given sieve size

could follow a similar course as was done for the development of the sensitivity criteria for friction, impact, and ESD. Data on explosives using the Hartmann apparatus, however, are not abundant.

[illegible] also recommended that consideration be given to a "Vapor Explosibility Test". The evolution of an ignitable solvent vapor cloud is not within the scope of the Dust Explosibility Test and presents a separate, very serious hazard.

Electrical Properties Tests

The objective of the electrical properties tests was to measure the conductivity, the permittivity, and the breakdown electric field strength of the test sample. Ignition by electrostatic discharge was identified as one of the major causes of accidents. The electrical properties tests relate to the propensity of a material to store electrostatic charges and to the maximum electric field that can be developed before a discharge occurs.

A person can pick up an electric charge by interaction with the floor as he walks, by putting on or taking off clothing, or by rubbing a hand across a table. If the person's clothing is a dielectric or of low conductivity, the surface of the clothing can store a charge. This applies to any dielectric surface such as a nylon jacket or a plastic dust cover on a piece of equipment. High permittivity, low conductivity materials hold a stored charge for a long time, and this ability to hold a charge is one of the main indicators of a potential electrostatic problem.

The ability of a material to store electrostatic charges is related to the relaxation time. If the relaxation time is short, charges will be given up rapidly. If it is long, more charges will be acquired than lost, and the electric charge will build up. The relaxation time constant, τ of a material can be calculated from

$$\tau = \varepsilon/\sigma \tag{25}$$

where ε is the permittivity and σ is the conductivity.

The charge density (q) is proportional to the quantity

$$(\varepsilon/\sigma) \cdot \left[1-\exp(\sigma t/\varepsilon)\right] \tag{26}$$

where t is time (Ref 48). The maximum electric field strength is proportional to q/ε. Thus, the conductivity and permittivity are very important properties in assessing electrostatic hazards.

Electrical Properties

To aid the discussion which follows, the electrical properties of interest are discussed in greater depth first.

The conductivity of a material, σ, is the reciprocal of resistivity, ρ. The test procedure for determining conductivity of a homogeneous material is to measure the sample's resistance, R. Using the measured value of R and the physical dimensions of the sample, the conductivity can be determined:

$$\sigma = \frac{\ell}{AR} \quad (S/cm) \tag{27}$$

where A and ℓ are the cross-sectional area, in cm^2, and the length, in cm, of the sample, respectively.

The permittivity of a material is a measure of its susceptibility to electrification. The permittivity, ε, is expressed as the relative permittivity, ε_r, with respect to the permittivity of free space, ε_o. The relative permittivity $\varepsilon_r = \frac{\varepsilon}{\varepsilon_o}$ is referred to as the dielectric constant. The permittivity of free space has a value:

$$\varepsilon_o = \frac{1}{36\pi x 10^9} = 8.85 \times 10^{-12} \ C^2/Nm^2$$

The dielectric constant of a material can be determined by measuring the influence of the sample material on the capacitance of a parallel plate capacitor. The capacitance, C_o in units of pF, of two flat parallel conductors separated from one another by a distance, ℓ centimeters, of free space or air* is:

$$C_o = \varepsilon_o \frac{A}{\ell} = 0.0885 \frac{A}{\ell} \ (pF) \tag{28}$$

*Relative permittivity of air at one atmosphere, 0^oC is 1.00058

With a dielectric other than air:

$$C = \varepsilon_o \varepsilon_r \frac{A}{\ell} \tag{29}$$

the relative permittivity, or dielectric constant, which is also given by the symbol K, is:

$$K = \frac{C}{C_o} = \varepsilon_r \tag{30}$$

The dielectric strength is a measure of the ability of the material to withstand electrostatic potentials without material breakdown. The dielectric strength of the material can be measured by applying an increasing potential across the material and observing the voltage level at which breakdown (i.e., high current flow) occurs. The dielectric strength, or field strength, is a ratio of the breakdown voltage to the sample thickness.

The relaxation time constant, τ, of the material can be determined from the conductivity and relative permittivity measurements previously described. Using Equations 27 and 29, the RC time constant τ can be written:

$$\tau = RC = \frac{\ell}{\sigma A} \times \varepsilon_o \varepsilon_r \frac{A}{\ell} = \frac{\varepsilon}{\sigma} \tag{31}$$

The relaxation time constant is a measure of the ability of the material to accumulate electrostatic charges.

There are no standard test procedures specified for obtaining the electrical properties of the material. In general, conventional laboratory test equipment is used to measure quantities that are related to the electrical property according to the basic definition. Sample preparation is an important consideration. Ideally, the test sample should be homogeneous, dimensionally stable and should not be unduly influenced by the test procedures. Additionally, it is important that external factors such as relative humidity and termperature do not influence the test data.

The following sections contain a description of the experimental evaluations performed on the propellants during the test program. The procedures used and the results obtained are discussed. The basic equations defining the electrical

properties as previously presented are included in the appropriate section for reading continuity. Finally, recommendations for testing the material in its in-process form are presented.

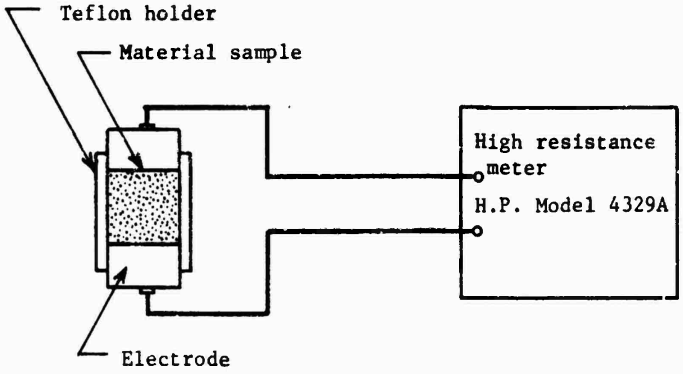
Test Description

It is important to mention at the outset that the experimental data obtained in this test program are not considered adequate for characterizing the in-process electrical properties of the propellant materials which were evaluated. These small-scale tests are suitable for homogeneous materials such as fine powders, uniform solids, and liquids. The materials should also be in a stable physical and chemical state. Sample preparation was a major problem for the propellant materials evaluated. The materials were prepressed to form solid disks, suitable for insertion into the existing test fixture. These test samples were not homogeneous; the solvent content was variable and the sample lacked the dimensional precision required for an acceptable determination of the electrical properties. Loss of solvent after pressing and prior to testing caused surface distortions in the test sample. In addition, the composition was changing due to the loss of solvent. This, in turn, influenced the dimension measurements.

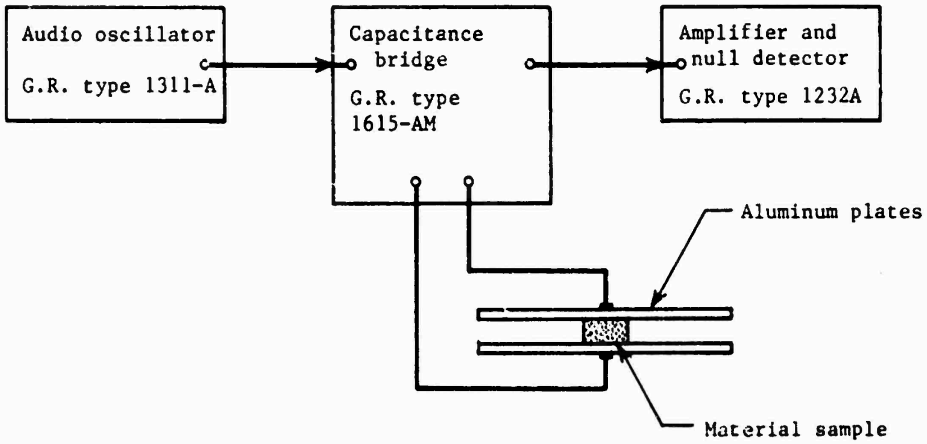
The experimental evaluation effort was useful in identifying problem areas that will be faced in implementing test procedures for in-process materials. Increasing the sample size and simulating on-line test conditions does not appear to present formidable problems for the conductivity and permittivity tests. The dielectric strength test, however, may present problems that are related to sample size and solvent content. These potential problem areas are discussed in the "Dielectric Strength" subsection.

Conductivity

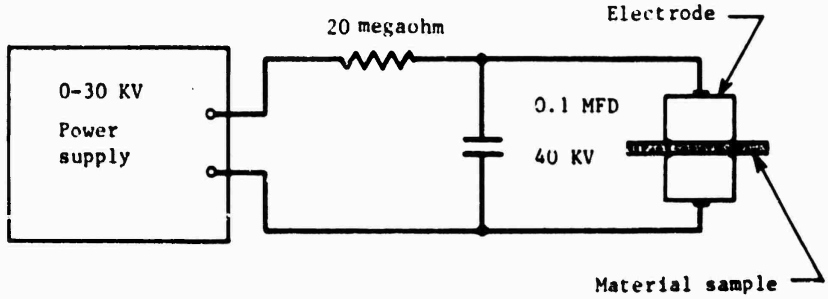
Figure 36a shows the test configuration for the conductivity tests. The sample holder consisted of two aluminum electrodes, 1.91cm long by 1.91 cm diameter and a teflon tube, 5lcm long. The teflon tube had a 2.54cm O.D. and 1.96 cm I.D. The test samples were pressed to form disks with diameters of 1.91cm and various lengths. These samples were placed in the holder and the resistance measurements then were made using a high resistance meter, Hewlett Packard Model 4329A. The resistance of the sample holder was about 2×10^{15} ohms.



(a) Conductivity



(b) Permittivity



(c) Dielectric strength

Fig 36 Test configuration for electrical properties

The high-resistance meter has a range 5×10^5 to 2×10^{16} ohms with seven test voltages in the range 10 to 1000 volts.

A few of the samples fabricated for the permittivity measurements also were used for conductivity measurements. The test fixture designed for permittivity measurements was used for these tests, and the resistance was measured with the high-resistance meter.

The conductance, S, of the material is the reciprocal of the resistance, R:

$$S = \frac{1}{R} = \frac{1}{\rho}\frac{A}{\ell} = \sigma\frac{A}{\ell} \quad \text{(siemens)} \tag{32}$$

where $\sigma = \frac{1}{\rho}$ is the conductivity of the material. The conductivity was calculated from a measure of the resistance and physical dimensions of the sample. The average thickness was used for these calculations. Volume resistance measuring techniques were employed; accordingly, the data, presented in Table 29 represent the bulk conductivity.

As noted earlier, the data presented in Table 29 do not show the required precision of measurement to adequately determine the conductivity for prepressed samples of the propellant materials. For a test voltage of 10 volts, the range of conductivities is:

M1	1.69 to 11.8 x 10^{-11}	S/cm
M26	1.15 to 7.46 x 10^{-9}	S/cm
M30	0.82 to 1.02 x 10^{-9}	S/cm

The materials tested do not appear to have a significant voltage coefficient. M1 shows a nominal increase in conductivity for increasing test voltage and M30 shows a slight decrease. No clear trend is indicated for M26.

The M30 pellets tested have a conductivity range between 2.05 and 3.01 x 10^{-7} S/cm. This increased conductivity is attributed to the solvent content of the samples. The sample pellets tested are representative of the in-process material, thus indicating that the solvent content may be an overriding factor in determining the electrical properties of the material in its in-process form.

Table 29

Conductivity of propellant materials

Material and sample	Diameter (cm)	Length range (cm)	Conductivity, (σ) (S/cm) Test voltage (volts) 10	25	50	100	250	500	1000	Remarks
M1-1	1.910	0.556-0.576	3.21×10^{-11}	3.16×10^{-11}	3.32×10^{-11}	3.49×10^{-11}	3.92×10^{-11}	4.25×10^{-11}	4.44×10^{-11}	Pressed at 5000 psi
M1-2	1.910	0.612-0.653	1.64×10^{-11}	1.71×10^{-11}	1.71×10^{-11}	1.90×10^{-11}	2.02×10^{-11}	2.22×10^{-11}	2.67×10^{-11}	Pressed at 5000 psi
M1-3	1.910	0.478-0.531	1.18×10^{-10}	1.46×10^{-10}	1.67×10^{-10}	1.75×10^{-10}	1.94×10^{-10}	1.96×10^{-10}	2.19×10^{-10}	Pressed at 5000 psi
M1-4	1.910	0.226-0.236	4.06×10^{-11}	5.40×10^{-11}	5.08×10^{-11}	5.41×10^{-11}				
M26-1A	5.874	0.097-0.135	1.66×10^{-9}	1.73×10^{-9}						Pressed at 600 psi
M26-1	1.910	1.04	7.46×10^{-9}	7.46×10^{-9}	7.31×10^{-9}	7.31×10^{-9}	7.04×10^{-9}	6.89×10^{-9}	6.90×10^{-9}	Pressed at 500 psi
M26-2	1.910	1.07	3.60×10^{-9}	3.12×10^{-9}	3.40×10^{-9}	3.16×10^{-9}	2.88×10^{-9}	3.46×10^{-9}	4.16×10^{-9}	Pressed at 600 psi
M26-3	1.910	0.99	1.15×10^{-9}	1.39×10^{-9}	1.39×10^{-9}	1.45×10^{-9}	1.51×10^{-9}	1.58×10^{-9}	1.93×10^{-9}	
M30-1	5.874	0.124-0.155	1.02×10^{-9}	1.02×10^{-9}	7.30×10^{-10}	5.70×10^{-10}				
M30-2	5.874	0.127-0.147	1.01×10^{-9}	1.01×10^{-9}	1.00×10^{-9}	8.38×10^{-10}				
M30-3	5.874	0.149-0.162	8.22×10^{-10}	8.22×10^{-10}	8.22×10^{-10}	8.22×10^{-10}				
M30-1A	0.714	0.238	2.05×10^{-7}							M-30 pellet
M30-2A	0.714	1.113	2.77×10^{-7}							M-30 pellet
M30-3A	0.714	1.191	2.37×10^{-7}							M-30 pellet
M30-4A	0.714	1.548	3.01×10^{-7}							M-30 pellet

Measuring the conductivity of the materials in their in-process form does not appear to present unusual problems. Larger samples and a sample holder (resistivity cell) will be required so that bulk and surface conductivity can be determined.

Surface conductivity may be the important factor for determining the relaxation time constant. The dielectric properties are a measure of the ability of the material to hold a charge. The charge is distributed over a very thin surface layer with the bulk of the material remaining electrically neutral. Accordingly, surface conductivity would have major importance in determining discharge time. Surface conductivity (resistivity) can be attributed to current flow through surface deposits, moisture, oxides and other semiconducting materials on insulator surfaces. With reference to the present application, the solvent may provide the conducting path directly or as a carrier. Accordingly, it may be desirable to determine the electrical properties of the solvent(s) used in the process.

Permittivity Measurements

The dielectric constant, K, of the material can be estimated by measuring the effect of the sample material on the capacitance of a capacitor. Figure 36b is a block diagram of the test setup used for these determinations.

The sample holder consists of two aluminum disks 12.70 cm diameter by 1.27 cm thick. The disks were used to form a parallel plate capacitor. Copper leads, 0.0794 cm by 0.635 cm approximately 46 cm long, were used to connect the parallel plate capacitor to the measuring equipment. The measuring equipment consisted of a General Radio Company Type 1620-A capacitance measuring assembly: a Type 1311-A audio oscillator, a Type 1615-AM capacitance bridge, and a Type 1232-A tuned amplifier and null detector.

The technique for determining the dielectric constant of the materials was patterned after those described by E. E. Walbrecht (Ref 49). With no sample in the fixture, the capacitance was measured by separating the plates a known distance. Three teflon disks, 0.318 cm diameter by 0.105 cm thick, were used for this purpose. The measured capacitance was 116.1 pF, as compared with the calculated value of 106.4 pF. This difference is attributed to stray capacitance and fringing.

The influence of this capacitance was accounted for by calculating on effective plate area, A_e. By rearranging Equation 28 and substituting the measured values, one gets:

$$A_e = 116.1 \frac{0.105}{0.0885} = 137.75 \text{ cm}^2.$$

The value of K as determined from the measured capacitors, is obtained by considering the system as consisting of two capacitors in parallel where the measured capacitance, C, is

$$C = C_a + C_m \tag{33}$$

and C_a and C_m are the capacitive contributions from the air and material areas respectively. Substituting Equations 28 and 29 into Equation 33 gives us:

$$C = \varepsilon_o \frac{A_a}{\ell} + \varepsilon_o K \frac{A_m}{\ell} \tag{34}$$

and

$$K = \frac{\ell C}{\varepsilon_o A_m} - \frac{A_a}{A_m} \tag{35}$$

where

K = dielectric constant

C = measured capacitance (pF)

ℓ = sample length (cm)

A_a = cross-sectional area for air dielectric
= 137.75 - A_m (cm^2)

A_m = material cross section area (cm^2).

The measured capacitance as a function of frequency is presented in Table 30. The first entry shows that the capacitance of the parallel plate test fixture is independent of frequency in the range tested.

Table 31 shows the computed dielectric constants for the propellants tested. The dielectric constants were calculated by using the mean value for sample length. The deviation in

Table 30

Measured capacitance versus frequency

Material and sample	Diameter (cm)	Length range (cm)	100 Hz C(pF)	500 Hz C(pF)	1000 Hz C(pF)	5000 Hz C(pF)	10,000 Hz C(pF)	
Test fixture	12.70	0.1054	116.1		116.02		116.0	Test fixture
M1-4	1.910	0.226-0.236		66.0	65.4	64.5	64.3	
M26-1A	5.874	0.987-0.135	210.0	191.6	182.8	163.6	159.0	
M26-4	6.350	0.127	296.0	234.0	221.7	199.1	137.5	
M30-1	5.874	0.124-0.155	267.1	214.1	199.3	175.2	165.1	
M30-2	5.874	0.127-0.147	290.3	237.1	218.3	188.6	177.6	
M30-3	5.874	0.149-0.162	282.1	253.1	240.2	202.8	184.8	

Table 31

Computed dielectric constants

Material and sample	Diameter (cm)	Length range (cm)	100 Hz K	500 Hz K	1000 Hz K	5000 Hz K	10,000 Hz K
M1-4	1.910	0.226-0.236		13.01	12.46	11.64	11.45
M26-1A	5.874	0.097-0.135	6.03	5.24	4.71	3.79	3.56
M26-4	6.350	0.127	10.03	7.22	6.66	5.64	5.11
M30-1	5.874	0.124-0.155	11.46	8.37	7.51	6.11	5.52
M30-2	5.874	0.127-0.147	12.51	9.47	8.39	6.69	6.06
M30-3	5.874	0.149-0.162	14.28	12.38	11.55	9.11	7.94

calculated values for different samples of the same material is attributed to inconsistency of the samples--particularly the density, physical dimensions, and solvent content. The decreasing dielectric constant of a given sample for increasing frequency, indicates the propellant material behaves as a polar dielectric.

Adapting this measuring technique to in-process materials appears feasible. It would be necessary to increase the size of the parallel plates and to provide a sample container. The influence of the sample container on the measured capacity could be taken into account easily.

Dielectric Strength

Dielectric strength measurements were eliminated from the test program. Their importance in characterizing the materials is recognized; however, this decision was based on priorities within the funding and time frame of the program.

A block diagram of the existing system for implementing the dielectric strength measurements is shown in Fig 36c. It appears that the existing system will have to be modified to provide a system suitable for testing in-process materials. Two factors--increased sample size and the relatively high conductivity anticipated for in-process materials--require consideration. Increased sample size will require a higher source potential to determine the dielectric strength. It will probably be necessary to consider the sample holder an expendable item when testing the larger samples. Also, the relatively high conductivity may cause changes in the test sample (i.e., E^2/R heating) prior to breakdown.

In the event a higher source potential is required, the existing power supply should be replaced. The influence on sample heating can be reduced to an acceptable level by pulse loading the sample. Pulse loading, as used here, means to subject the sample to a voltage pulse of short-duration. This can be accomplished by charging the capacitor to a known voltage level and then, by using a switching circuit, discharge the capacitor through the test sample. This technique would probably require the use of new samples for each test level in order to eliminate residual effects.

Breakdown of the test material is accompanied by ionization and high current flow. As a practical matter, breakdown is usually detected as a loud sound during the test or as

damage to the sample as detected in a posttest inspection. The use of a current sampling resistor should be considered for a more quantitative indication of breakdown.

Recommendations

The basic measurement techniques described above for determing the electrical properties of a material are realistic and appropriate for hazards classification. It must be strongly emphasized, however, that for the test to be valid, the physical and chemical characteristics of the sample must be the same as those existing in the process. In addition, the tests are valid only if the sample is homogeneous inside the test holder. Thus, altering the test material to fit the apparatus or allowing a solvent to evaporate can yield drastically different property values than exist in the process.

A material with relatively large basic dimensions, for example pellets, cannot be evaluated realistically using a small scale fixture. A valid test could be done by increasing the scale of the apparatus so that the pellets appear to be small (like a powder) relative to the inside dimensions of the test container.

Electrostatic Discharge Test

The objective of the electrostatic discharge (ESD) test is to determine the energy in the form of an electrical spark required to ignite material samples. Materials can gather and store electrostatic energy simply by flowing over a surface such as the wall of a hopper. If a metal component in a system becomes ungrounded, the metal can store a large charge. Because of the metal's high conductivity, a simple discharge could contain the total stored energy and thus constitute a significant hazard. The electric charges stored by a piece of equipment can be developed by interaction with particles flowing in a bed or cloud through it. For example, the flow of powder from a hopper or through a pneumatic duct could leave the hopper or duct at a high voltage if these items were electrically isolated (ungrounded). Similarly, flow of a charged powder into an ungrounded item, such as a tote bin, would raise the voltage of that piece of equipment. An extension of this concept is an ungrounded person. Humans can likewise generate and store charges. These stored charges can be discharged into in-process materials under the proper conditions. The ESD test determines the sensitivity of the material to such discharges.

The ESD test is one of the core tests recommended for all materials. The analysis of accident data resulted in the ESD sensitivity criterion of 0.17 joules. If the ESD test shows a material to have an ignition energy less than the criterion, then a penalty factor will be assessed in the hazard classification scheme. A material with an ESD sensitivity greater than the criterion will not be penalized.

Test Description

The basic concept of the ESD test is to store energy in a charged capacitor and then release the energy into a layer of the sample material. It is necessary to measure the voltage and current through the sample as a function of time to determine the energy. A typical test apparatus is shown in Fig 37. A schematic circuit diagram of the test is shown in Fig 38. The discharge path is affected by the physical shape of the electrodes and the electrode spacing.

The test procedure is to place the sample in or on the sample holder. The sample holder may be in various forms to accommodate materials in various states (solid, liquid, or aerosol). The capacitor is then charged by a high voltage power supply (in the range of 5000 V). The capacitor is discharged across the sample in one of two ways. Either the discharge needle electrode is lowered in increments until a spark is drawn through the material, or the discharge needle electrode is set at a predetermined height above the material and a switch is closed allowing the spark to jump the gap. Obviously, the needle must be set at a height at which discharge can occur.

To determine the energy delivered to the sample, a very high impedance electrostatic voltmeter must be used (0 to 50,000 volts). The capacitor is typically 0.01 to 0.04 microfarads. Two voltages must be measured to calculate the energy. These are shown in Fig 38. The time histories of the voltage across the sample, $V_{sample}(t)$, and the current through the sample, $I(t)$, are needed. By referring to Fig 38, the current can be obtained by dividing the voltage across the sample and resistor by the resistance R_i :

$$I(t) = \frac{V_i(t)}{R_i} \qquad (36)$$

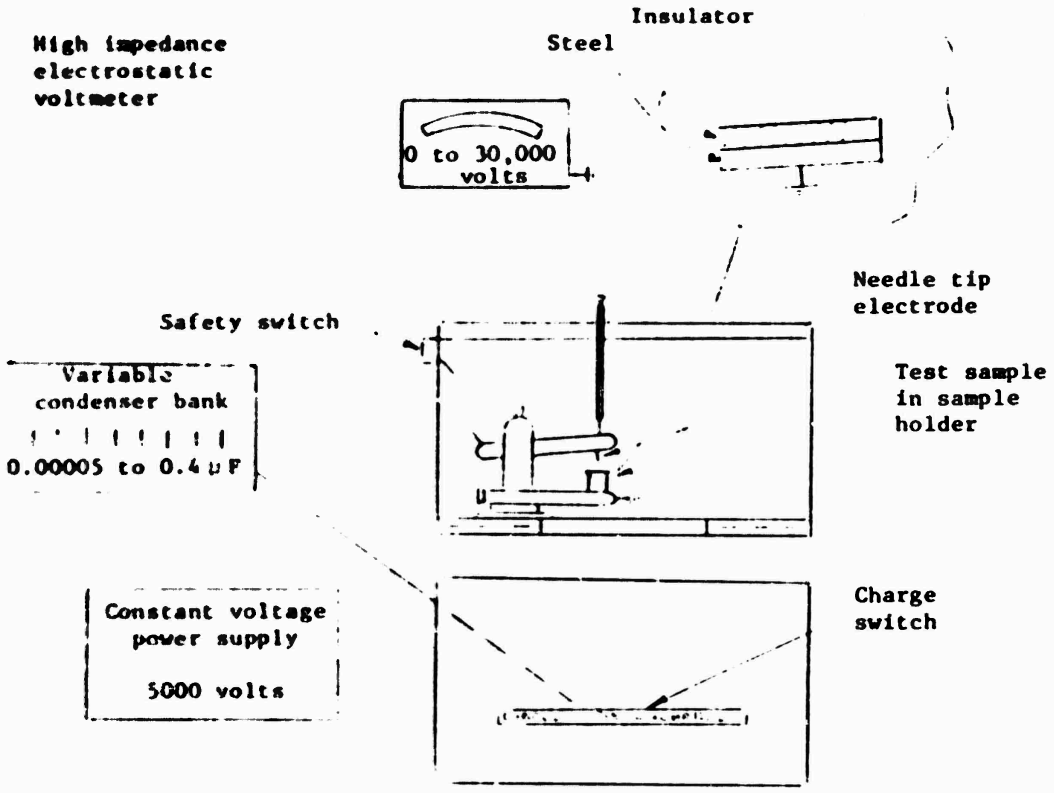


Fig 37 Typical electrostatic discharge test

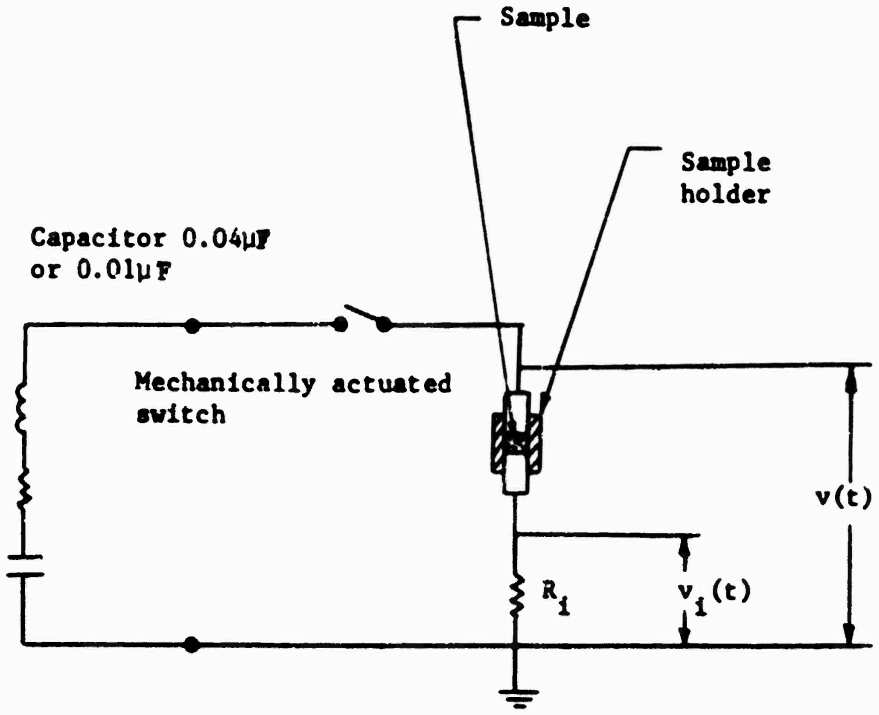


Fig 38 Schematic circuit diagram for electrostatic discharge test

The time history of the voltage across the sample is the difference between V(t) and $V_i(t)$.

$$V_{sample}(t) = V(t) - V_i(t) \tag{37}$$

From these two measurements the energy is calculated as:

$$E = \int_0^t V_{sample}(t) \tag{38}$$

$$E = \int_0^t \frac{V_i(t)}{R_i} \cdot \left[V(t) - V_i(t)\right] dt \tag{39}$$

This method for calculating energy is more accurate than the standard formula $E = 1/2\ CV^2$ where C is the capacitance. This formula overestimates the energy losses in the conductors, switches, discharge gap, and resistivity of the test specimen. It is therefore preferable to measure the energy in each test, or for the discharges to be calibrated for each series of tests.

A test procedure similar to that in NAVORD OD44811 (Ref 50) is recommended.

Test Procedure

The test apparatus is built to specifications listed in PL/DL16804 (Ref 51). Electrode spacing should be adjusted before each test. The relative humidity should not exceed 40 percent, and ambient air temperatures should be between 10 and 32°C. The upper needle electrode should be replaced after 10 trials, after any test where a reaction occurs, at the beginning of a test series with a new explosive, or whenever the operator observes a change in its condition. The apparatus should be checked periodically with lead azide (35mg) at 7500 volts. The apparatus is considered to be performing satisfactorily if 10 specimens explode in 10 trials.

For booster explosives, a sample size of about 30mg is recommended. For main charge explosives and propellants, the maximum sample size should be:

Solid propellants	50mg
Powders or granules	50mg
Casting powders	150mg
Liquids	25mg

The test sample is first placed in the sample holder and mounted in the test fixture. The electrode spacing is then adjusted to a convenient starting point. The spacing is typically between 0.041 cm and 0.58 cm. This should be checked for each test and adjusted as necessary. The capacitor should then be charged to the desired voltage (0 to 50,000 volts) as indicated by the electrostatic voltmeter. The charge switch is then released and the results are observed. An audible report or visible smoke or flame is considered evidence of a reaction. This should be distinguished from the noise of the spark only. The two voltage records may also indicate evidence of a reaction. The voltage records should be preserved as the energy can be calculated at a later time. Holding the electrode spacing fairly constant, the charging voltage should be varied $\pm$ 1kV in Bruceton fashion over the full range of voltages. The electrode spacing should then be changed and tests conducted again over the full range of charging voltages using a Bruceton approach. The different electrode spacings will produce different pulse shapes.

All the energies should be calculated from the accumulated voltage records for this one value of capacitance. A tabulation should be made at each energy level and charging voltage as to the number of reactions, number of no reactions, and total number of trials. A statistical analysis of the data will reveal the 50 percent probability of ignition energy level. Reference 52 describes in detail the statistical method used to analyze the data. The method is summarized below.

Defining the parameters:

p = probability of a reaction
q = probability of a no reaction
n = total number of trials

The probability deviations are calculated as:

$$\pm \left(\frac{pq}{n} \right)^{1/2} \text{ or } \pm 0.5n^{-1/2} \text{ as a maximum.}$$

By plotting the energy on arithmetic probability paper, the 50 percent point can be estimated.

To clarify the data analysis up to this point, an example is described next. The data collected for black powder using a 0.01μF capacitor are shown in Fig 39. The data show the range of energies calculated for each voltage level. The curve is drawn through the points where the Bruceton method predicted the boundary to be. For example, at 6 kV the transition between no reaction and reaction was at 0.10 joules. From this curve and the data, Table 32 was prepared. This provided the information for calculating the statistical parameters summarized in Table 33. The energy is then plotted against the probability of a reaction, (p), plus or minus the deviation, on arithmetic probability paper (Fig 40). A straight line drawn through the points or their band will determine the 50 percent probability level. In this example the 50 percent probability of ignition is estimated to be 0.068 joules.

The above test procedure and analysis should be conducted for a range of capacitances. The minimum 50 percent probability energy level then should be selected as the hazard limit. This is shown in Fig 41 taken from Ref 53. This hazard limit is then compared to the 0.17 joule ESD criterion for hazards classification. For the above example, the figure indicates a hazard limit of 0.12 joule, which is less than 0.17, and the material would be penalized in the hazard classification procedure.

Recommendations

No electrostatic discharge testing has been conducted in this program. Therefore the validity of the ESD sensitivity criterion has not been established. It is recommended that tests be conducted on some pilot materials to establish the validity of the criterion. Some additional effort should be allocated toward developing a test fixture and sample holder which will accommodate any sample state (solid, liquid or gas).

Thermal Test

The objective of this test is to determine the activation energy for in-process materials and to determine the auto-ignition temperature corresponding to a given heating rate. Numerous process operations require heat addition to the

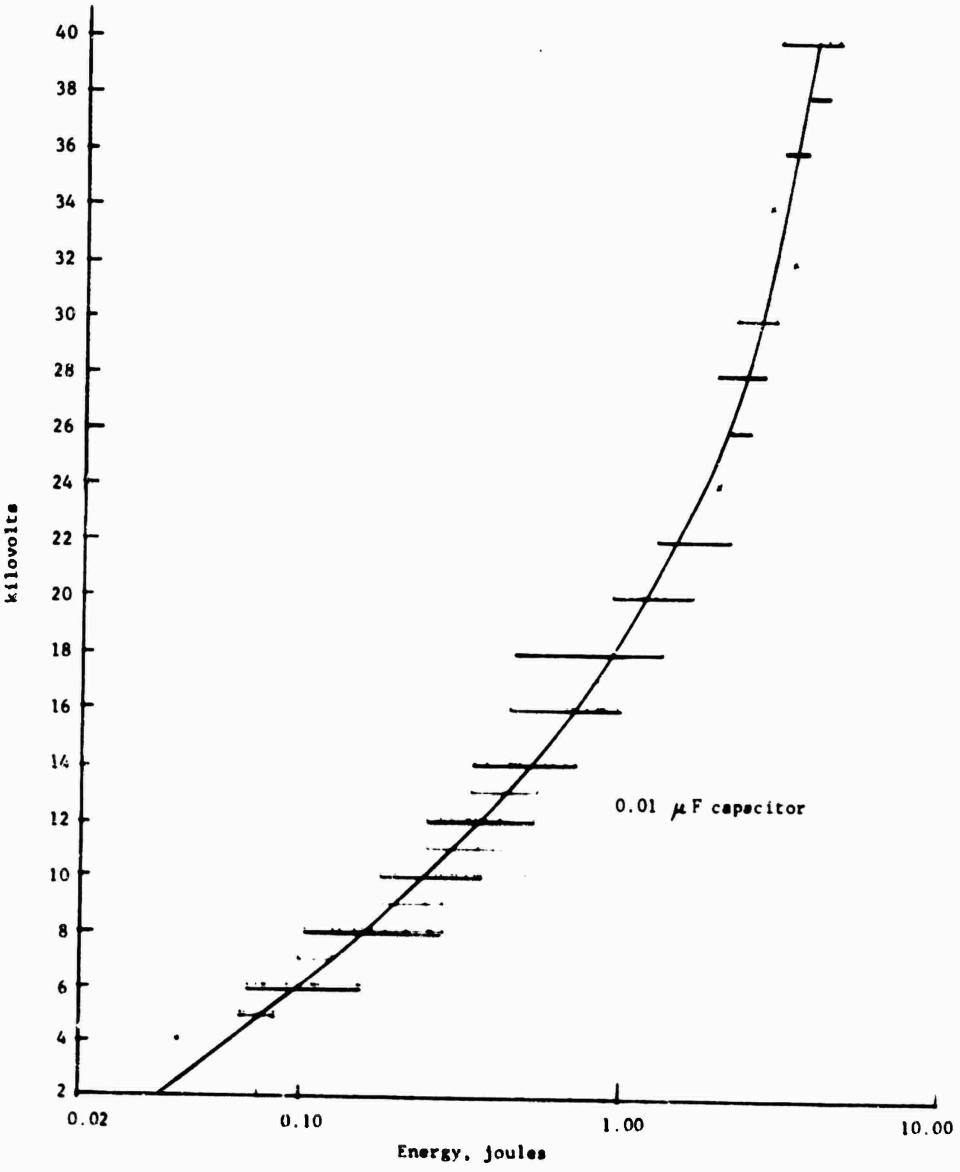


Fig 39 Determination of 50 percent probability of ignition energy level, for ESD

Table 32

ESD test data for black powder

Charging Voltage (kV)	Number of Reactions	Number of No Reactions	Total Number of Trials	Energy (joules)
3	0	1	1	0.046
4	1	4	5	0.060
5	2	1	3	0.076
6	6	0	6	0.100
7	1	0	1	0.130
8	2	0	2	0.160
9	1	0	1	0.200
10	2	0	2	0.245
11	1	0	1	0.300
12	2	0	2	0.365
13	1	0	1	0.440
14	3	0	3	0.520

Capacitor = 0.01 μF

Table 33

Statistical summary of ESD test data for black powder

E(joules)	n	p	q	$\pm 0.5\ n^{\frac{1}{2}}$	$\pm(pq/n)^{\frac{1}{2}}$
0.046	1	0	1.0	±0.50	-
0.060	5	0.2	0.8	-	±0.180
0.076	3	0.66	0.33	-	±0.272
0.100	6	1.0	0	±0.21	-
0.130	1	1.0	0	±0.50	-
0.160	2	1.0	0	-	-

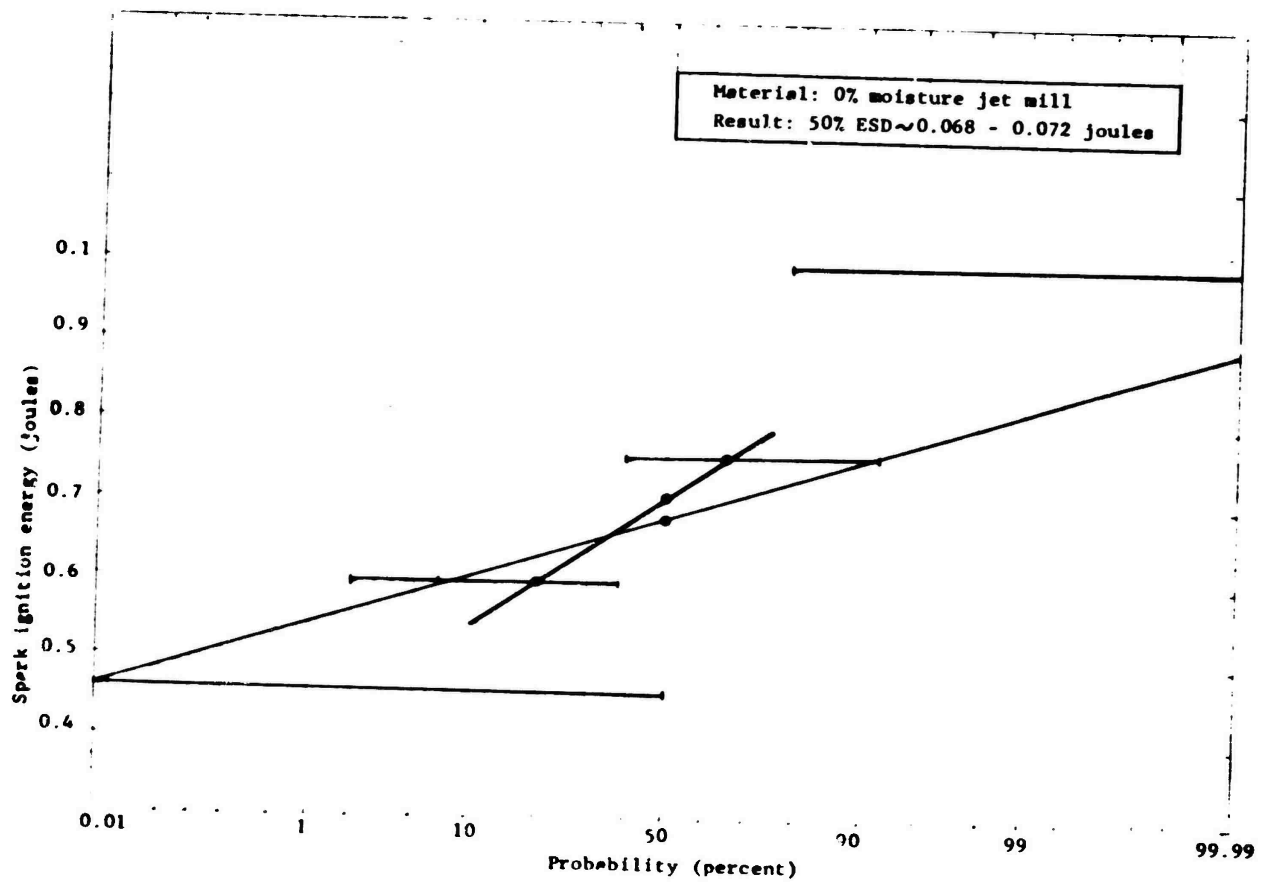
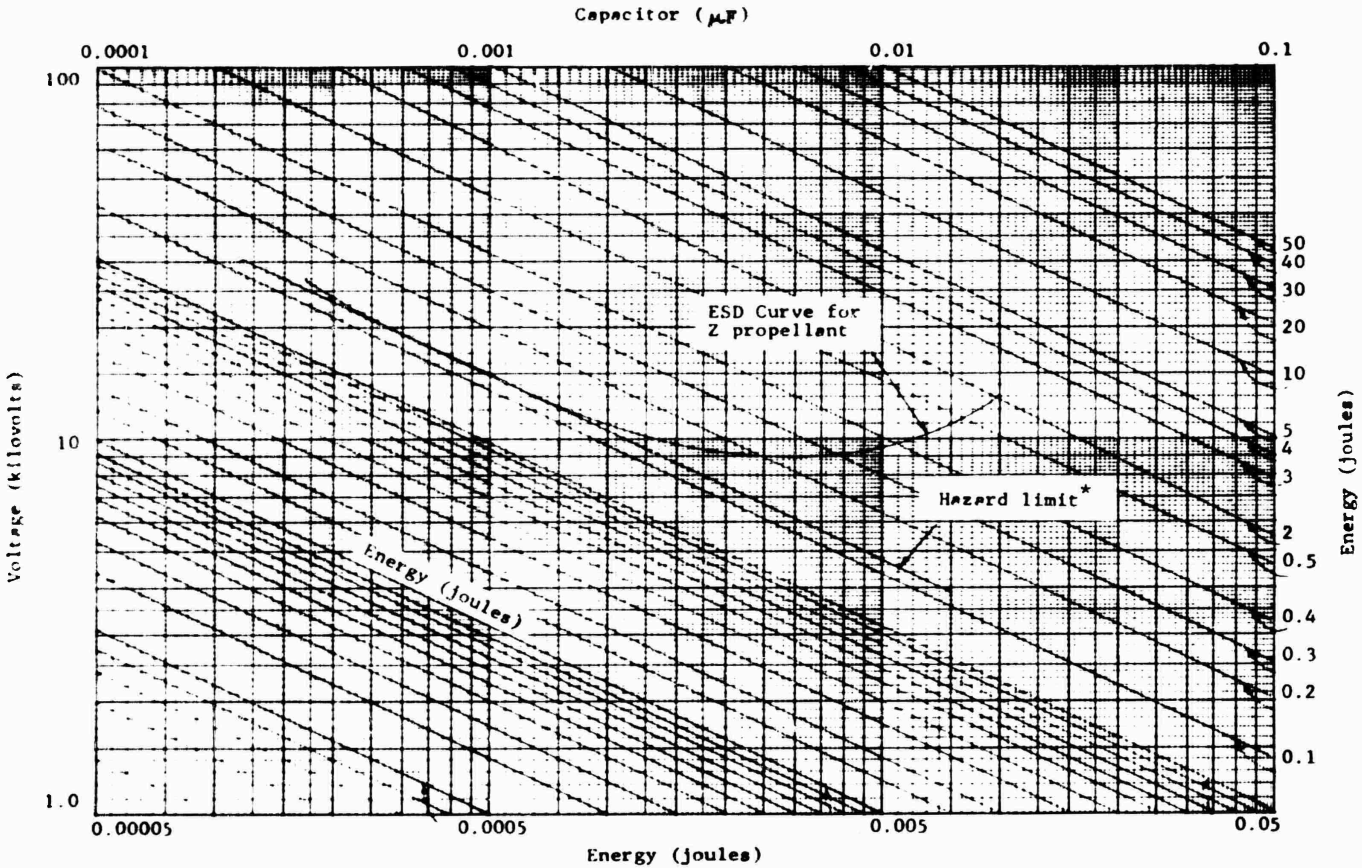


Fig 40 Determination of 50 percent ignition energy from experimental data (Example)



*Hazard limit is the minimum 50% fire energy in joules.

Fig 41 Typical electrostatic 50 percent fire curve for "Z" propellant (from Ref. 53)

working material, e.g. reactors, dryers, and melt-pour operations. This results in normal operating temperatures greater than ambient. Abnormal heat additions can occur due to failure of cooling equipment or steady frictional heating. The activation energy is a measure of a material's susceptibility to chemical decomposition at any temperature. A high activation energy indicates slow decomposition. The autoignition temperature places an upper bound for the safe operation of any process.

Of the four materials to be tested, only one (M30 pellets) represented an operation where the heating test would be necessary. The M30 air dried pellets are exposed to a drying operation which normally requires heat addition.

Test Description

The differential scanning calorimeter (DSC) was chosen as the best method to achieve the desired objectives. During the test, the sample material and a reference material are heated simultaneously at the same rate. The sample and reference are contained in separate cups but placed in a common holder. Both cups are instrumented with thermocouples. The difference in electric power required to keep both sample and reference pan at the same temperature is recorded and is called a thermogram. An endothermic process (heat absorption) will require power to the sample pan and results in a downward deflection of the recorder pen. An exothermic process (heat release) will require less power to the sample and the recorder pen will deflect in the opposite direction. The temperature at which ignition occurs is clearly evident on the thermogram. The DSC analyses permit interpretation of phase changes, decomposition, melting points and thermal stability.

The test procedure is relatively easy. The tests were performed in atmospheric air. The instrument can handle other gaseous environments. A heating rate of 10°C per min was selected for these tests. The M30 sample was sliced to a size of 1mm x 1mm x 0.1mm thick. A small lid was placed over the sample after it was placed in the sample cup. The reference used in these tests was merely an empty sample cup.

The thermogram for M30 triple base propellants (lot number RAD 77F0015012) is shown in Fig 42. Curve B is the M30 thermogram. Curve A is a blank run under the same conditions as the sample curve but with both pans empty. This

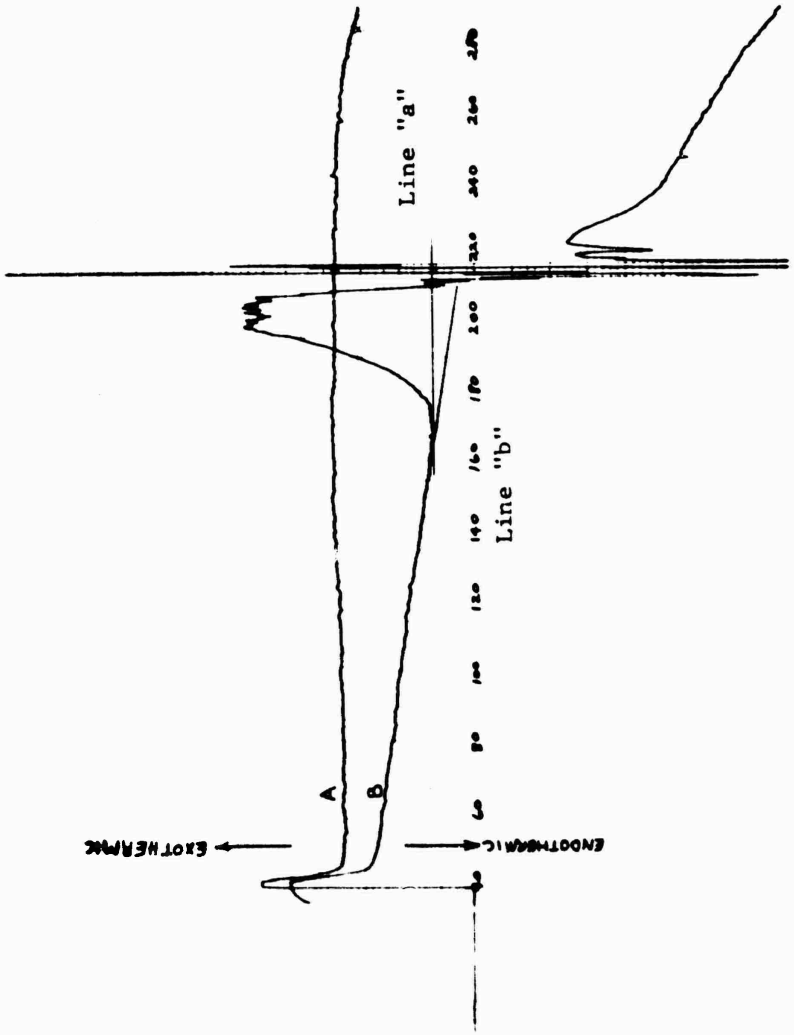


Fig 42 Example thermogram

was merely to confirm that a straight, horizontal base line was achieved. The M30 thermogram is seen to decrease monotonically as temperature increased between 40 and 170°C. This behavior is probably due to evaporation of the solvents that were present in the sample. At 170°C the M30 clearly begins to decompose thermally. This onset of an exothermic reaction leads to the autoignition of this material. Safe handling of the material should be limited to below 170°C.

For larger quantities of M30, the limiting temperature may be different. Because of limited heat dissipating qualities of more massive quantities, self-heating may result which may lead to an explosion. The self-heating temperature depends on the amount of material, its environment, and the time duration over which the material will be held at the elevated temperature (Ref 54).

Further analysis of the thermogram will produce the activation energy. The rate of energy evolution is proportional to the amount of pen deflection on the thermogram. In this case only the exothermic reaction is of interest. To measure the amount of deflection, it is necessary to select a base line. Two possibilities exist: choose a horizontal line, tangent to the peak of the endothermic reaction, or draw a line on an angle tangent to the endothermic curve. Both lines are drawn in Fig 42, identified respectively as line 'a' and line 'b', and the deflection was measured in arbitrary units from each base line up to the exothermic curve. The data are presented in Table 34.

Following the derivation and analysis methods of Ref 55, the activation energy can be calculated from the following equation:

$$E^* = \frac{-19.16 \log_{10} (d_1/d_2)}{1/T_1 - 1/T_2} \quad \text{(J/mole)}$$

where d is the pen deflection at a temperature T. This also can be written:

$$E^* = -19.16m$$

where m is the slope of a straight line for log d versus 1/T. The data from Table 34 were plotted in Fig 43 to determine m. A low activation energy indicates rapid decomposition. Therefore, in analyzing the experimental data, it is safer to choose

Table 34

Thermogram data analyses for determining activation energy of M30 pellets

For straight horizontal baseline:

	d	T(°C)	T(°K)	$\log_{10} d$	$1/T \times 10^3$ (1/°K)
1	0.5	175	448	-0.3010	2.2321
2	2.0	180	453	0.3010	2.2075
3	5.5	185	458	0.7404	2.1834
4	10.0	190	463	1.0000	2.1598
5	17.5	195	468	1.2430	2.1367

$$m = \frac{\log_{10} (d_2/d_4)}{1/T_2 - 1/T_4} = \frac{-0.699}{0.0477} = -14.654$$

$$E^* = (-19.16)(-14.654) = 2.81 \times 10^5 \text{ J/mole}$$

For decreasing baseline (tangent to curve):

	d	T(°C)	T(°K)	$\log_{10} d$
1	1.2	175	448	0.0792
2	3.0	180	453	0.4771
3	7.0	185	458	0.8451
4	11.8	190	463	1.0719
5	19.2	195	468	1.2833

$$m = \frac{-0.9927}{0.0723} = -13.73$$

$$E^* = 2.63 \times 10^5 \text{ J/mole}$$

Horizontal baseline: $E^* = 2.81$ J/mole

Sloping tangent baseline: $E^* = 2.63 \times 10^5$ J/mole

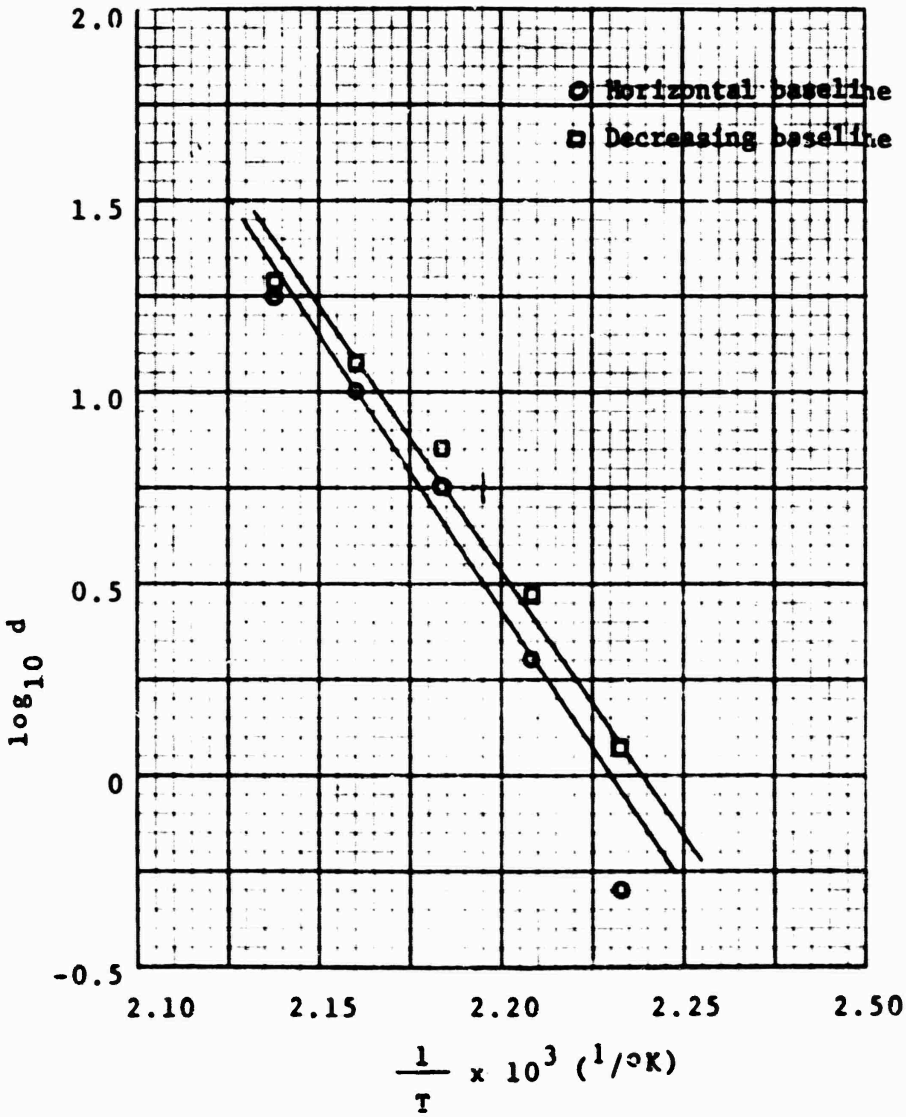


Fig 43 Determination of slope n for data in Table 34

the method giving the lowest value of the activation energy. This indicates then, for M-30 triple-base propellant pellets, the activation energy is 2.63×10^5 J/mole.

Recommendations

It is recommended that the differential scanning calorimeter test be incorporated into the hazards classification procedures for in-process materials. In conducting the test, sample sizes should be kept less than 20 mg and a heating rate of 10°C per min be adopted. The sample cups for the DSC should be used with the lids. This is not essential for the operation of the instrument, but it does make a difference in the results. Consideration should be given to using sealed cups. These were not tried in this investigation.

To incorporate the activation energy and autoignition temperature into the classification procedures, the following criteria should be considered.

For any process operation where the heating test is required, first the autoignition temperature should be compared to the maximum process operating temperature plus 20 percent. If the autoignition temperature is less than this value (i.e., $T_{ai} < T_p + 0.2\ T_p$) then a penalty is assessed according to the methods outlined previously. Secondly, the activation energy should be compared to the total heat input per unit mass of material (Q/M) for the process operation under study. The 20 percent factor is again recommended to define the cut-off point. The criteria can be stated as:

$E^*_* > Q/M + 0.2\ Q/M$ No penalty
$E^*_* < Q/M + 0.2\ Q/M$ Penalized

where Q is the total heat addition to the material in the operation considered and M is the total mass in that operation.

Further clarification of these criteria is recommended. The 20 percent safety factor may not be realistic and the total heat input to the material (Q) may not be an easy quantity to obtain. Additional tests and comparisons to actual processes are recommended.

CONCLUSIONS AND RECOMMENDATIONS

Based on the results of the investigation described in the preceding sections, the following conclusions and recommendations have been formulated.

Conclusions

(1) There are many deficiencies in the current hazard classification schemes. These include the definition of the current hazards classes, the test procedures and interpretation of results, and the lack of effects data.

(2) The current quantity-distance requirements are inadequate as they do not account for the threats posed by asymmetric fireball, glass breakage, thermal radiation or fragments.

(3) The most common causes of an accident are: friction, impact, electrostatic discharge, and thermal heating. The frequency of these causes varies with the materials and process operation.

(4) Correlations were made between the sensitivities of materials involved in accidents and the sensitivities of materials not involved in accidents. Statistically significant differences in the sensitivities of the two populations were identified for friction, impact, and electrostatic discharge. These differences were used to formulate sensitivity criteria.

(5) A preliminary hazards classification procedure was developed which utilizes data from material properties, sensitivity and effects tests. A basic set of tests are required in the procedure.

(6) Selected small-scale tests were evaluated critically for their application in hazards classification of in-process propellant and explosive materials. Care must be taken to ensure that the physical and chemical state of the material is not altered in preparation for the test. This invalidates small-scale test results for many materials. In such cases a large-scale version of the test or a special test would be required.

Recommendations

Preliminary recommendations were formulated based on the findings to date. These recommendations are:

(1) Revise the quantity-distance standards to allow the use of the threat equivalency which is calculated in the classification procedure described in this report. This revision should also change the DoD hazard classes to the UN hazard classes.

(2) Reevaluate the sensitivity criteria given in this report by using the entire DDESB data base. This should include a more thorough review of each accident. Concurrently, obtain the raw Radford AAP sensitivity data and compare Radford's results with the results of other organizations.

(3) Conduct the large-scale tests called for in the classification procedure to ensure that the required data can indeed be obtained. The possibility of obtaining secondary fragment data with the test should also be explored.

(4) Develop a procedure to be followed for the many situations where the test material is not a homogeneous solid, liquid or fine powder. For these cases some small-scale tests are no longer valid, and a large-scale version or a special test should be used.

CONCLUSIONS AND RECOMMENDATIONS

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APPENDIX A

SUMMARIES OF SELECTED ACCIDENT REPORTS

Table A-1

Summary of selected accidents which occurred during pressing operations

ASESB no.	Agent	Amount (lb)	Fatalities/ injuries	Component or operation	Type of output	Max. distance missile/glass breakage (ft)	Probable causes
1185	ASROC (Cruciform grain -rework)	--	0/0	Press-extrusion operation	Explosion-Fire	660/-	1) Powder contamination 2) Friction (powder-powder) 3) Heat of impact 4) Trapped air compression within press bucket
677	TNT	1/2	0/3	Plunger-die matrix	Explosion	-/-	Die-plunger misalignment causing frictional initiation
679	Tetryl pellet	--	-/-	KUX pelleting press	Explosion	-/-	Friction/impact: misalignment of ram; mechanical malfunction
687	Tetryl pellet	--	-/-	KUX pelleting press	Explosion	-/-	Impact, friction
711	TNT	--	--	Press	Explosion-Fire	125/-	Mechanical malfunction: metal-to-metal contact
754	Nitroguanadine green powder area (solvent vapor shot)	--	0/0	Pre-blocker press	Explosion	--	1) Autoignition of vapors 2) Impact
946	Flake TNT	--	0/0	300 ton transfer press	Explosion	--	Friction: 1) Extrusion-side of ram & die wall 2) Impact-ram & fractured die 3) Foreign material 4) Misalignment
1506	Composition A-3	Single pellet	0/2	Stokes pelleting press	Explosion	--	1) Foreign material 2) Failure of punch 3) Excessive pressure due to excessive pellet buildup on-ram
1261	M-1 propellant		0/1	Farguhar vertical blocking press	Fire-Explosion	--	Compression of entrapped air and solvent vapor causing autoiggition (ethyl ether, ethyl alcohol)

Table A-1 (contd)

Summary of selected accidents which occurred during pressing operations

ASESB no.	Agent	Amount (lb)	Fatalities/ injuries	Component or operation	Type of output	Max. distance missile/glass breakage (ft)	Probable causes
1345	M30-multi-perforated triple base propellant	30 lbs	0/0	Extrusion press	Explosion	Damage in press bay	--
1376	Multibase casting powder	35/80	0/0	Extrusion press	Fire	--	--
835	Double base solventless rocket propellant Navy X-8	--	0/0	Extrusion press	Explosion	--	--
773	M5	--	0/0	Extrusion press	Fire-Explosion	Bay area	Heat generated by fire near press
802	M7 (solvent-less)	38 lb	--	15" R.D. wood	Explosion	141/--	--
913	Ammonium perchlorate	--	--	Lombard horizontal extruder	Explosion	2 cells completely demolished	1) Friction of moving parts 2) Adiabatic compression of trapped air 3) Heat rise during normal operation 4) Oxidizer entrapped between moving parts
1292	P&R pellets	7 lbs	0/1 (lacerations, burns)	Rotary Cherry Burrell press	Explosion		Foreign particle, metal-metal interface (ram-wall)
1146	RDX	½ lb	0/0	Arthur Colton single action pelleting press	Explosion	30/-	Unknown
?	C E pellets	--	0/1	Porter press	Explosion-Fire	--	Mechanical failure (core rod) - metal-metal and explosive interface - caused by misalignment
617	Lead Azide	--	0/1	Pellet press	Explosion	--	Impact, friction
937	N-5 formula (solventless double base slurry)	--	0/0	Expeller/ extruder	Explosion-Fire	~860 ft/--	1) Friction generating heat near torpedo head 2) Foreign material causing friction, pinching, or impact

Table A-1 (contd)

Summary of selected accidents which occurred during pressing operations

ASESB no.	Agent	Amount (lb)	Fatalities/ injuries	Component or operation	Type of output	Max. distance missile/glass breakage (ft)	Probable causes
1217	Double-base casting powder	140/180	0/0	Finishing press	Explosion-Fire	Immediate area within building	At dia and bottom of press basket surface)lateral motion-friction
1367	RDX pellets	.1 lb	1/1	Press	Explosion	--	--
901	M-16 & wax	30 (M-16) 40 (wax)	--	Horizontal press	Explosion fire	--	1) Adiabatic compression of air trapped in wax or interface of wax and propellant-hot spot 2) Contaminated area-interface (propellant leakage) causing heat of friction initiation
1193	M6 (solventless) double base composition	100 lb (press) 707 lb (stored)	--	Farquhar 15" horizontal press	Explosion	Immediate building	Friction initiation - due to foreign metal inclusion between ram and basket
1377	C-3 prop	60 lb	0/0	Blocking press	Fire	Damage in immediate building	--
1419	Triple base propellant	10 lb	0/0	Extrusion press	Explosion	Damage to press, building	Foreign metal inclusion
1068	Butadiene-N-Ammonium Nitrate	75	0/0	1070 ton extrusion press	Fire	Immediate area of press	Autoignition-adiabatic compression due to breakdown of limit switch controlling initial ram
1083	AML 2056-D propellant	--	0/0	Watson-Stillman finishing press	Explosion	Immediate bay area	Ignition of solvent vapors - adiabatic compression
833	ARP double base casting powder	"2 blocks"	1/0	Finishing press	Explosion	Immediate bay area	Adiabatic compression of solvent vapor (ether alcohol) due to blockage of vent
901	M-7	--	--	15" R.D wood press	Explosion-Fire	625/--	--
1226	M-30 propellant	--	--	Farquhar 12"	Explosion-Fire	Immediate bay area	Adiabatic compression entrapped vapor
690	M-10	--	0/2	Extrusion press	Explosion	Immediate area	1) Adiabatic compression 2) Rapid extrusion-localized overheating

Table A-1 (contd)

Summary of selected accidents which occurred during pressing operations

ASESB no.	Agent	Amount (lb)	Fatalities/ injuries	Component or operation	Type of output	Max. distance missile/glass breakage (ft)	Probable causes
718	Matador re-work powder (rejected grains)	7 grains	--	Press	Explosion	20/-	Contamination of seal-line interface in die assy. Ram was stationary, press-gate and die-assy as the focal point of det-onation; compression of heel on 7 grain charge-initiation possibility
653	C E perfor-ated pellet	2 oz pellet	0/0	Porter press	Explosion		Foreign particle inside press
377	TNT	--	--	Press	Explosion-Fire	--	Expl. dust and pulley slipping-friction initia-tion
757	Flake TNT	16 grains	0/0	Stokes pellet press	Explosion	Damage to im-mediate area	Foreign metal initiated (friction) during oper-ation of press
10	Gunpowder	2 pellets + 60 lbs (hopper)	0/2	Press	Explosion	--	Loose powder ignited by friction; pressure be-tween extracting shoe and mold
963	M17 Prop mix	90 lbs available (top of block ini-tiated)	0/0	13-3/4 vertical blocking press	Explosion	--	Ignition of solvent-vapor by adiabatic com-pression
1127	Black powder	(6) 30 mm pellets	0/1 burn	Press	Explosion-burn	--	Unknown
975	Black powder	100 lbs + 1500 lbs charge house	0/0	Pellet press	Explosion-fire	Immediate area	Unknown (possibly friction)
961	Boron - P nitrate ig-niter comp	24 pellets (2 lbs) + 7 lbs on table	0/0	Stokes rotary press	Explosion-fire	Immediate area	Friction between die and upper punch
654	Black powder	--	--	Block press	Explosion	--	Rapid compression of sol-vent vapors caused heat build-up to initiate ex-plosive
612	Double base Nitrocellon-91 4 percent Nitroglyc-erin-42 0 percent (solventless)	70 lbs	0/0	15 in R D cordite press	Explosion-Fire	Limited to building 47/-	Compression of powder under flappers when press-ed against room face

Table A-1 (concl)

Summary of selected accidents which occurred during pressing operations

ASESA no.	Agent	Amount (lb)	Fatalities/ injuries	Component or operation	Type of output	Max. distance missile/glass breakage (ft)	Probable causes
64	SF62(4n.4 pellets)	165 grains	0/1 (burn)	(3) Triple punch Horsam presses	Explosion	Immediate area near punch	Perforating needle failure introduced as foreign article
714	Powder	--	2/5 (burn)	Horizontal finishing press	Flash fire	--	During extrusion process
976	Double base solventless	50 lbs	0/0	Watson-Stillman 15 in. horizontal extrusion press	Explosion-Fire	80/-	Friction, failure of Teflon ring seal; metal to metal contact; ram & press basket
1013	AlCle	353 grains	0/1 (burn)	400 ton compaction press	Explosion-Fire	Minor damage	Misalignment of die mandrel & retaining mandrel causing interference (metal-metal)
862	Casting powder O I O	--	0/0	Pawfoot press	Explosion (minor)	Superficial damage	Adiabatic compression - solvent vapor
1040	T36 prop	50 lb (rework)	--	Blocking press	Explosion	--	1) Heat by friction caused by interaction of ram head and cylinder wall and included powder buildup at interface (normal). 2) Friction at cylinder interface caused by mechanical failure at lower end of ram.
1174	H-8	--	1/3	Evenspeed roll	Flash fire	NA	Unknown

Table A-2

Summary of selected accidents which occurred during mixing operations

ASESB no.	Agent	Amount (lb)	Fatalities/ injuries	Component or operation	Type of output	Max. distance missile/glass breakage (ft)	Probable causes
1473	Casting (20% scrap) powder	620	0/0	Talley mixer	Fire	-/-	Friction between blade and lining
920	Dynamite-nitroglycerine	1000 700	2/5 (frag)	Talley machine	Explosion	600 ft/3 miles	Unknown
1309	C-3 casting powder	--	0/0	Mixer	Fire-Explosion	Bldg only/--	Foreign particle/failure or blade; friction/impact
896	Composite propellant	98 lbs	0/2 (flash burn)	20 gal Baker Perkins mixer	Explosion	-/-	Foreign article between blades and liner caused frictional heating
878	Polysulfide base TRX 110C propellant	--	-/-	200 gal Baker Perkins mixer	Explosion-Fire	150 ft/-	1) Blade clearance .088/.109 in. - friction 2) Static spark (gas leakage observed-ammonia perchlorate and fuel)
729	Composite (nitroguanidine, K_2SO_4)	200 3.9	0/2 (fire)	Mixer charging	Fire	NA	Sigmablade-frictional heating
1310	NACO propellant single base	"8 blocks"	2/1 (burn)	Mixer	Fire	NA	1) Foreign material - friction 2) Dehydration of mixture increasing sensitivity for impact 3) Metal-to-metal blade-lining contact caused by initial deflection of resistive force with NACO blocks
472	M7 propellant	450 lb	1/1 (blast) victim thrown 120 yds	Mixer	Explosion	-/-	Friction - blade-lining pressing of dry potassium perchlorate pre-blend (.025 in. clearance)
1000	Polybutudiene ammonium perchlorate + MAPO (solid propellant)	280	0/0	Mixer	Fire	NA	Spontaneous autoignition of MAPO - no blades in mixer

Table A-2 (concl)

Summary of selected accidents which occurred during mixing operations

ASESB no.	Agent	Amount (lb)	Fatalities/ injuries	Component or operation	Type of output	Max. distance missile/glass breakage (ft)	Probable causes
1206	Double base propellant (NC, amm. perchlorate & aluminum)	720	0/0	Mixer	Fire	NA	Foreign objects (metal) - frictional heating of blade-object-lining interface
1261	TP-H1085 propellant	4400	3/2 (fire)	Baker-Perkins mixer 300 gal (scrape down)	Fire	NA	Ignition of ammonium perchlorate/sublimed recrystallized ferrocene by heat of friction. Action: steel spatula/liner during scrape down
1289	Single base multiperforated powder	5000	*/	Mixer - (discharge into buggy)	Explosion	-/-	Ignition of dust granules by ESD
811	Propellant composite	500	2/0	Mixer	Explosion-Fire	(900/2000)/-	Foreign article-scraping tool inside mixer caused frictional heat initiation between blade and liner
964	Polysulfide perchlorate propellant	--	--	Extruder	Explosion	--	Friction: 1) foreign object; 2) blade & liner contact; 3) solid buildup
657	Propellant Carbon black Nitrocellulose Ammonium perchlorate	--	0/0	Mixer	Explosion	400/-	Too low a content of solvent in mixture
382	Nitroglycerine Nitrocotton	3578 936	6/1	Washing	Explosion	--	Friction-wooden clip and rubber tube
1189	Lead Azide (alcohol & freon)	2	1/-	Filling flasks Washing-Aspiration	Explosion	--	Friction due to bumping of flask and funnel
741	Ball powder WC870	1060	1/- (Burn)	Salt coat and glazing Sweetie barrel	Explosion	Immediate area	Slurry spillage-alcohol fumes ignited by metal-metal contact (bucket & object)

Table A-3

Summary of selected accidents which occurred during casting operations

ASESB no.	Agent	Amount (lb)	Fatalities/ injuries	Component or operation	Type of output	Max. distance missile/glass breakage (ft)	Probable causes
906	TNT	Residual TNT contaminate	0/0	Melt reservoir	Fire	NA	Impact of contaminated bottom of tank; residual TNT on bottom exposed to 220°F for 9 hours
1066	1) 4-Polaris A3 second stage 2) Nitroglycerine 3) Casting powder 4) Scrap casting solvent 5) Aspirator	-- 4800 865 250 30	3/11	Prop. motor casting	Explosion	1500/2000	Solvent handling - vapor initiation
989	Pentolite (50/50)	/20	0/0	Melt tank	Explosion & fire	100/0	1) Instability of Pentolite under prolonged heating 2) (Air) pneumatic transfer system provided ignition; impingement of functional initiation of explosive dust 3) ESD
868	TNT	Residual TNT on floor	0/1	Melt chamber	Fire	NA	Friction initiation due to scraping of dry TNT residual on concrete floor with steel spatula
1230	Cyclotol	--	6/4	Melt and pour operation	Explosion-Fire	--	1) Spark initiated - tool droppage 2) Riser scrap causing friction between agitator and kettle 3) Foreign material present-friction 4) Contamination of electrical controls with explosive dust, etc.

Table A-3 (concl)

Summary of selected accidents which occurred during casting operations

ASESB no.	Agent	Amount (lb)	Fatalities/ Injuries	Component or operation	Type of output	Max. distance missile/glass breakage (ft)	Probable causes
1275	TNT	--	0/0	Melt tank	Fire	--	Friction/spark initiation during removal of old (TNT) contaminated insulation (maintenance)
1222	RDX slurry	--	0/1	Valve for recrystallization still	Explosion	Equipment area	Impact; unplugging of valve with non-sparking screwdriver
518	RDX/TNT (6) torpedoes		11/	Melting process	1) Bomb 2) Fire-Explosion	500 yd proximity completely destroyed	Foreign material inclusion; cardboard soaked with oil caught fire during melting
41	Black powder	5000	0/0	Glazing	Explosion	850 ft/1½ miles	Lightning
1330	1) Semi-finished black powder	7000	1/1	Glazing	Explosion	¼ mile/2 mile	Unknown
	2) Finished black powder	1300					
601	Lead azide	1) 10 grain detonator	0/1	Unloading moulds from extraction unit	Explosion	5-10'/--	Invested mold was brought in contact with surplus explosion on top of the extraction machine

Table A-4

Summary of selected accidents which occurred in a reactor

ASESB no.	Agent	Amount (lb)	Fatalities/ injuries	Component or operation	Type of output	Max. distance missile/glass breakage (ft)	Probable causes
1476	TNT	10,000	0/6 major 100 minor	Nitrator-separator	Explosion	3000/ 70 ft crater	Thermal/exothermic instability-inadequate mixing due to hose obstruction with agitator
1267	Nitroglycerine	--	--	Nitrator	Explosion	--	Chemical decomposition of old spent acid within reactor-exothermic
502	TNT	--	0/0	Tri-nitrator	Fire	--	Thermal instability - caused by operator procedure
1117	Hexogene	--	0/0	Nitrator	Reaction chemical	--	Thermal instability - caused by mach failure
1191	TNT	--	0/3	Dinitrator	Reaction chemical violent	--	Exothermic reaction
1811	Nitroglycerine	Small		Nitrator	Explosion	--	Temperature increase
982	Tetranitromethane	--	--	Reactor	Explosion	Building destroyed	Poor agitation caused a runaway chemical reaction with excessive heat build-up, mechanical failure of agitator
1119	Nitroglycerine	1100	2/-	Nitrator-separator	Explosion	<500 ft building destroyed	Block cock - friction oriented
380	Nitroglycerine	5500	--	Nitrator-separator	Flame-explosion	--	1) Decomposition of impurities adhering to reactor shell 2) Decomposition of flacculent matter on surface of separated nitroglycerine
908	Nitroglycerine	?	0/0	Dinitrator waste acid	Explosion	--	Contamination during cleaning exercise
312	Nitroglycerine	450x3 1350 lb	2/0	Reactor	Explosion	Building destroyed <500'	Unknown
603	Nitroglycerine	--	0/1	Nitrator (Biazzi system)	Explosion	1000' plant destroyed	Decomposition of nitroglycerine-runaway reaction due to excessive acid addition, heat generated and fast temperature rise
907	PETN Petrin acrylate (rocket propellant)	1 lb	1/3	Nitrator	Explosion	<100 ft	Instability of impure PETN included with acid at ambient temperature and further decomposition of explosion with the addition or a water-acid base (accidental addition of water)

Table A-5

Summary of selected accidents which occurred during conveying operations

ASESB no.	Agent	Amount (lb)	Fatalities/ injuries	Component or operation	Type of output	Max. distance missile/glass breakage (ft)	Probable causes
593	Nitroglycerine	--	--		Explosion	- /750	Friction: wood scraper and compound
1304	RDX/TNT Swsrf	3		Vacuum pump	Fire	NA	Pump overheating
1032	HMX Slurry	--	0/0	Pump	Explosion		a) HMX caking within pump cavity caused friction b) Presence of super-sensitive Alpha fumes of HMX
1052	DNT/TNT (70-30%)		0/0	"Downie" pump	Fire	NA	1) Localized overheating within pipe casing 2) Foreign objects
1280	Propellant single base slurry		0/0	Slurry deliv-ery pipeline	Explosion-Fire	250/-	Thermal decomposition due to heat application from steam tracer line and propellant accumu-lation within pipe
1314	Commercial powder	Total 8210 (12,000 lbs/hr)	0/6	Air-conveying of powder for reblender	Explosion	Immediate building 82/-	1) Friction heat build-up of particles inside rubber hose (deterio-ration of hose) 2) Extreme velocities of transfer

Table A-6

Summary of selected accidents which occurred during drying operations

ASESB no.	Agent	Amount (lb)	Fatalities/ injuries	Component or operation	Type of output	Max. distance missile/glass breakage (ft)	Probable causes
1287	Ball powder WC852	6000 (init) 13,780	0/0	Dryer	Fire-L O. Det-onation	Building area	Excessive heat build-up
1467	Black powder boron pot nitrate tungsten delay mix	25 20 1000	0/0	Dryhouse and oven	Explosion	--	Unknown
910	Dry Nitro-starch	850	0/0	Dryer	Explosion	75/	Overheated motor in fanhouse
758	Nitrocotton	700	0/0	Dryhouse	Explosion	100/	Unknown
712	Nitrocotton	400	1/0	Dryhouse	Explosion	1200/200	Unknown
1279	NC		0/6	Dehydration press	Explosion	--	Friction-caused by misalignment of ram and NC block with liner
394	TNT	300 stove 1000 drying 150 hopper 5-6 tons stored	0/0	Dehydration unit	Explosion	--	1) Decomposition accel-erated by pressing of ammonium nitrate due to contamination in drying stove
393(a)	Cordite RDB		0/0	Stove	Fire	--	--
393(b)	Cordite RDB		1/0	Stove	Fire	--	Ignition of solvent va-pors
393(c)	Cordite RDB		2/2	Stove	Fire	--	Ignition of inflammable vapor in the vapor piping
393(d)	Cordite MD		1/5	Recovery stove	Fire	--	Vapor ignition of acetone -air mixtures which were to be recovered
393(e)	Cordite RDB		0/1	Stove	Fire	--	Ignition of vapors
376	Di-nitro-phenol-picric acid		7/69	Drying room	Fire-Explosion	15 yd crater 12 ft deep 500 yd missile	Initiation-smoking
383	Cordite RDB	47,332	0/0	Recovery stove	Explosion	--	Vapor ignition

Table A-6 (concl)

Summary of selected accidents which occurred during drying operations

ASESB no.	Agent	Amount (lbs)	Fatalities/ injuries	Component or operation	Type of output	Max. distance missile/glass breakage (ft)	Probable causes
1045	Mercury fulminate	5 lbs	0/0	Drying operation	Explosion	60/50	Unknown
1125	Lead Azide Lead trinitro resorsinate	27	1/0	Dry house	Explosion	chamber/building	Impact (?)
1129	Lead Azide Lead Styphnate	22 lbs	1/0	Drying operation	Explosion-Fire	-/60	Impact
1202	MP-2 Experimental prop	10 oz	--	Cure oven	Explosion	immediate oven area	Thermal instability between MP-2 and other ingredients
1107	EM-27 prop.	--	--	Curing facility	Explosion	--	a) High temp. b) Equip. failure c) Impact (slippage)
1056	FPC experimental motor mold	--		Curing house	Explosion-Fire	immediate bay area	Impact and sympathetic reaction
349	Fire gunpowder	2800	5/2+	Drywhouse (handling)	Explosion	350/	ESD-explosive dust explosion
798	Nitrocellulose colloid		1/4	Hardening still (drying operation) cleaning scraping)	Flash Fire	NA	1) Impact from brass scraper 2) Friction-employee standing on residue material 3) Decomposition of remains within still
534	Fulminate and tetraethylic substance	--		Hot air stove	Explosion	--	Overheating
1123	Mercury fulminate	55 lbs	2/0	Dehydration	Explosion	Building area	1) Initial ignition of dry fulmate by friction (metal-metal) with subsequent ignition of alcohol-air mixture in collector
821	Single base prop (shock gel process)		0/1	Rotating drum dryer	Explosion	--	1) Loose metal supports within dryer initiated pellets by friction

Table A-7

Summary of selected accidents which occurred during filling operations

ASESB no.	Agent	Amount (lb)	Fatalities/ injuries	Component or operation	Type of output	Max. distance missile/glass breakage (ft)	Probable causes
786	Ammonium Dichromate	150	0/0	Aluminum drum collector	Fire	NA	1) Spark-impingement of particle against metal valve on receiving can (ungrounded) 2) Heat of friction - V belt rubbing against pully contaminated with ammonium dirchromate dust 3) Air lock valve blade became overheated due to friction
755	Multi-perforated single base M10 cannon powder and graphite dust	3000 and additional 2000	2/3	Loading pre-blender hopper	Explosion-Fire	900/4500	1) Static discarge - due to powder impingement 2) Potential difference between accumulated static charge of powder in buggy and the powder in metal barral 3) Potential difference between operator and powder 4) Friction between hopper edge and buggy
615	Composition A	--	0/0	Filling unit	Explosion	--	Friction-metal-metal contact
802	Nitroglycerine, TNT, Nitrocotton, Dynamite, Dope	350/300/150/2700/760	0/0	Dope handling unit	Fire-Explosion	400/1500	Unknown
569	Smokeless powder	130,000	9/0	Filling bin	Fire	--	1) Friction-metal-metal contact by opening slide gate on car 2) ESD
807	Black powder potassium nitrate	3500	2/0	Pack house	Explosion	100/-	--
1138	Powder dynamite	4620	2/4	Packing house	Explosion	900/-	Friction? conteiner floor spark

Table A-7 (concl)

Summary of selected accidents which occurred during filling operations

ASESB no.	Agent	Amount (lbs)	Fatalities/ injuries	Component or operation	Type of output	Max distance missile/glass breakage (ft)	Probable causes
1218	Lead Azide	15	1/13	Weighing filling operation	Explosion	Immediate building	1) Impact initiation (dropping freon spray nozzle) 2) ESD 3) Friction initiation (Spatula and compound)
777	Photoflash powder	16 and add 40		Loading machine	Explosion	475/470	Impact or friction initiation
887	Hi-speed	1100	2/2	Cartridging machine	Explosion	600/600	Unknown
1339	Nitroglycerine	7000	1/13	Loading operation	Explosion	1200/3200	Unknown
1319	Dextrinated lead azide	--	0/1	Charging unit	Explosion	--	1) Mechanical misalignment causing heat of friction 2) Impact due to misalignment of punches
1033	Dry nitrostarch	100	1/0	Unloading dryer	Explosion	75/0	Unknown
776	Photoflash powder	25	0/3	Loading/ filling machine	Explosion-Fire	Immediate area	Static discharge
994	Petrogel # 1		0/0	Filling/ packing	Fire flash	--	Sliding catch box over contaminated propellant
1272	M9 propellant	--	2/32	Loading machine	Fire-Explosion	--	Mechanical malfunction
1448	M9 propellant		0/3	Loading perry accofil machine	Flash fire	NA	Friction of exposed sensitive explosive
3	Detonators	1) No. 13 detonator plus 190 grains	1/0	Breaking down detonators (dismantling)	Explosion	10/-	Striking detonator with a sharp tool and hammer
952	Ammonium perchlorate developmental	--	3/1	Removing oxidizer slurry from tank	Explosion	--	Probing of packed sludge with a rod (friction)

Table A-8

Summary of selected accidents which occurred during screening operations

ASESB no.	Agent	Amount (lb)	Fatalities/ injuries	Component or operation	Type of output	Max. distance missile/glass breakage (ft)	Probable causes
721	Mercury fulminate	9 oz.	0/1	Sieving	Explosion	--	Impact/friction initiated striking of funnel with brush handle
785	Lead styphnate	1-1/2 lbs.	0/0	Jelly bag screener	Explosion	50/	1) Impingement-during pouring of L.S. over screener 2) ESD
744	None-dynamite dope ingredients	--	0/0	Screener	Fire	NA	Friction caused by foreign material in screen
751	None-dynamite dope ingredients	--	0/0	Screener	Fire	NA	Electrical wiring
581	T9 powder pot. nitrate, ammonium picrate, aceton, ethylcellulose, zinc stearate, tri-calcium phosphate	1700 lbs. total in area	2/0	Blender/ screener unit and filling drums	Explosion	--	1) Frictional-metal-metal contact 2) Static discharge-all during operation
1184	Green smoke powder	--	--	Sifting	Explosion	--	Friction

Table A-9

Summary of selected accidents which occurred during machining

ASESB no.	Agent	Amount (lb)	Fatalities/ injuries	Component or operation	Type of output	Max. distance missile/glass breakage (ft)	Probable causes
649	Black powder	500	0/0	Corning mill	Explosion-Fire	1300/1 mile	Friction-foreign metal object between mill rolls ignited dust
781	Sodium nitrate black powder	650	0/0	Wheel mill	Explosion	--	Wheel slippage-Friction
782	Black fuse powder	3700	0/0	Corning mill	Explosion	350/1300 building demolished	Unknown
633	Rocket propellant powder	56 (initial) 548 (total)	1/0	Cutting machine	Explosion		1) Friction-steel blade on machine ignited nitroglycerine fumes or powder dust 2) Impact knife on machine guide causing shock initiation of nitro condensate
665	Mercury fulminate	--	1/2	Rebowling operation	Explosion	--	Inadvertent impact
1038	TPH 8126 composite propellant	total 7 lbs	1/2	Bandsaw	Explosion-Fire	12 ft/-	Ignition of propellant fines in lower guide blocks of band saw friction
1490	Nitrocellulose slurry	contaminated pipe section	0/1	Hacksaw blade	Explosion	10/-	Friction due to metal-metal contact
1499	Black powder (block)	41	0/1	Bandsaw	Fire	NA	Friction between metal and black powder
1205	Propellant grain		0/0	Saw	Fire	NA	Friction
1108	Reinforced grain (Minuteman rocket motor)	10 lbs	0/0	Vertical radial saw (hydraulic)	Fire	NA	Inherent friction in machining of reinforced grain

Table A-9 (contd)

Summary of selected accidents which occurred during maching operations

ASESB no.	Agent	Amount (lbs)	Fatalities/ injuries	Component or operation	Type of output	Max. distance missile/glass breakage (ft)	Probable causes
735	Rocket grain	--	0/0	Dowel Rod machine	Explosion	Immediate bay area	Friction initiation during machining
1277	M13	50 lb	0/0	Stokes granulator	Fire	NA	1) Friction between agitator and screen 2) Abrasion nature of binding agent due to natural evaporation
1254	Sparrow MK 38 mod 0 solid grain prop	91 lb	0/1	1/32 Drill into prop	Fire	NA	Friction heat buildup
1231	Ammonium perchlorate		0/0	Sweco vibro-energy gringing mill	Explosion	--	a) Adiabatic air compression b) Localized buildup near flanges c) Foreign particle
504	Green charge mix (Pot Mix charcoal sulphur)	312	0/0	Wheel mill #2	Explosion-Fire	310/intra-plant	Unknown
959	Composition prop experimental Emerson-Cummings Epoxy resin catalyst alum powder pot perchlorate	(cast) 3/4 lb	(cuts) 0/1	Machining lathe	Explosion	--	Unknown (Decomposition of experimental chem
1295	Ammonium perchlorate	--	0/0	Raymond grinder	Explosion	Nav area/ building	Friction initiation due to contamination of bearing hub oil (hydrocarbon)
646	Mark 16 solid prop		0/1	Rotary saw	Fire	NA	Spark initiation and heat buildup
691	Sodium Nitrate black powder composite	--	--	Wheel Mill	Explosion	220.-	Friction-two dry powder

Table A-9 (concl)

Summary of selected accidents which occurred during machining operations

ASESB no.	Agent	Amount (lb)	Fatalities/ injuries	Component or operation	Type of output	Max. distance missile/glass breakage (ft)	Probable cause
699	Benite powder	41 strands	0/2	Bandsaw	Fire	NA	1) Ignition of vapors due to friction caused by: a) Excesive saw speed b) Insufficient coolant flow c) Adherence of powder to saw and revolving under wheel of saw 2) Static spark ignition of alcohol vapors
6024	JOM extruded MK22 grains	65	0/0	Saw	Fire-Detonation	--	Friction originated, drip vacuum enhanced spread of fire
1114	M-5 paste slurry	--	--	Expeller mill	Explosion	--	Mechanical or chemical initiation(?) Friction

Table A-10

Summary of selected accidents which occurred during maintenance operations

ASESB no.	Agent	Amount (lb)	Fatalities/ injuries	Component or operation	Type of output	Max. distance missile/glass breakage (ft)	Probable causes
611	"Explosive"	3/4	1/5	Maintenance-conveyor system	Explosion	Immediate area	Cutting torch-localized heat on contaminated vacuum pipe
626	"Dry" Nitrocellulose	--	1/2	Repair and maintenance	Explosion	--	Cigarette/match ignition of contaminated underground pipe
410	Contaminated waste water	--	1/0	Maintenance	Fire-Explosion	--	Waste-water residue contaminated in basin. Fire initiated by friction from metal friction subsequent explosion of pipe caused by heating due to fire
356	Residual dry Nitrocellulose	5-10	0/5	Cleaning	Explosion	--	Swab initiated Nitrocellulose within pipe by friction
1234	Composition A-5	17-25 lbs + 25 + 5		Cleaning-maintenance	Fire-Explosion	--	Caused by striking vacuum kettle against garbage can to provide ignition by 1) Impact 2) Friction 3) ESD
1180	Double base triple base propellant	--	1/0	Maintenance-welding of bride block	Explosion	--	Welding of contaminated bridge block sufficient to allow localized heat initiation
1227	Lead azide residual crystals	--	1/0	Cleaning	Explosion		Impingement of contaminated residual sludge by hose stream caused foreign objects within to sbrade LA crystals

Table A-11

Summary of selected accidents which occurred during storage

ASESB no.	Agent	Amount (lb)	Fatalities/ injuries	Component or operation	Type of output	Max. distance missile/glass breakage (ft)	Probable causes
945	Nitroglycerine	3000	0/0	Storehouse	Explosion	1000/plant area	Initiation by range fire
1079	Nitroglycerine	7540	1/1	Storage Tanks	Explosion	1000/1 mile	Unknown (friction possibility) deceased entered area with pail
528	Gun powder	36 tons	15/25	Storage	Fire-Explosion	1/4 mile 1-3/4 mile	Fire initiated
597	Powder	--	--	Storage	Explosion	--	Cigarette initiated
661	Nitrates a) Soda b) Ammonia	--	12/-	Storage	Fire-Explosion	Crater 22 ft Depth 6 ft	Unknown

APPENDIX B

EVALUATION OF SMALL-SCALE TESTS

FRICTION SENSITIVITY TEST

Objective: To estimate the likelihood that a sample will ignite when subjected to friction (normal force at a given velocity).

Operating Procedure: Force is applied to a sample which is in motion relative to another sample.

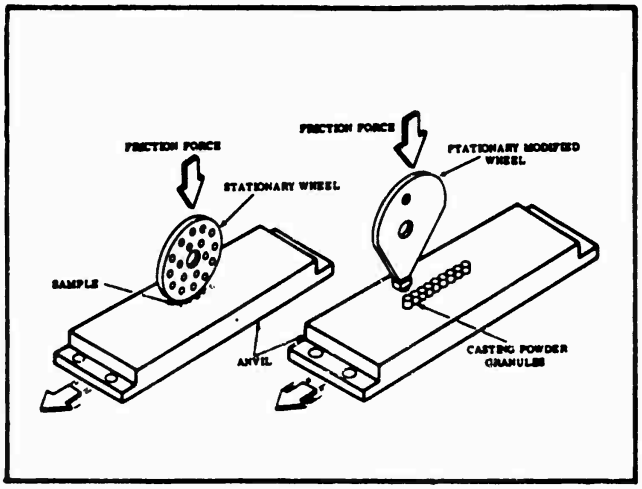


Fig B-1* ABL Friction tester

Test Description:
There are four basic types of friction testers. These can be characterized as:

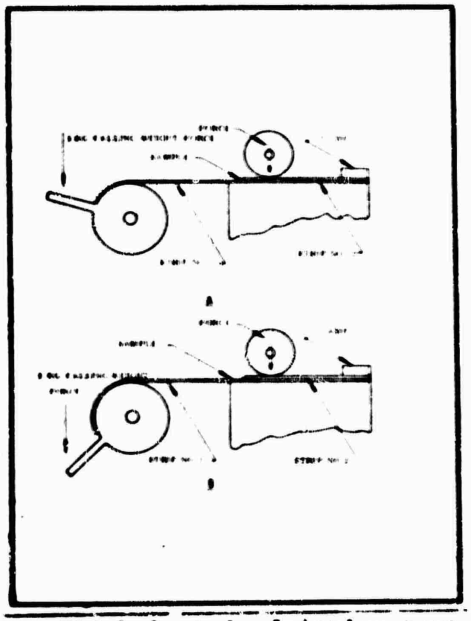
- sliding block or strip friction testers
- pendulum friction testers
- rotary friction testers
- bulk material friction testers.

These friction tests differ in the types of sample materials that can be tested and the means of applying the friction. Unless otherwise cited these tests are described in Ref B-1.

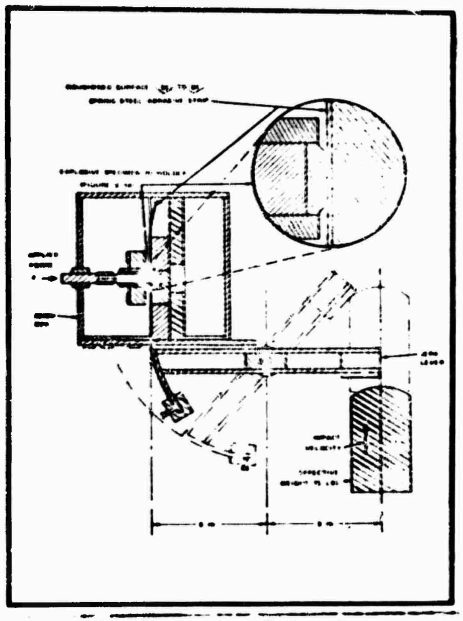
The sliding block or strip friction testers consist of a movable block, anvil, or strip; a device to force the sample against the anvil; and a device to move the anvil. Friction testers in this category include:

- the ABL friction test (Fig B-1)
- the Thiokol strip friction test (Fig B-2a)
- the OD44811 strip friction test (Fig B-2b)
- the ERDE emery paper friction test, and
- the ERDE sliding block friction test.

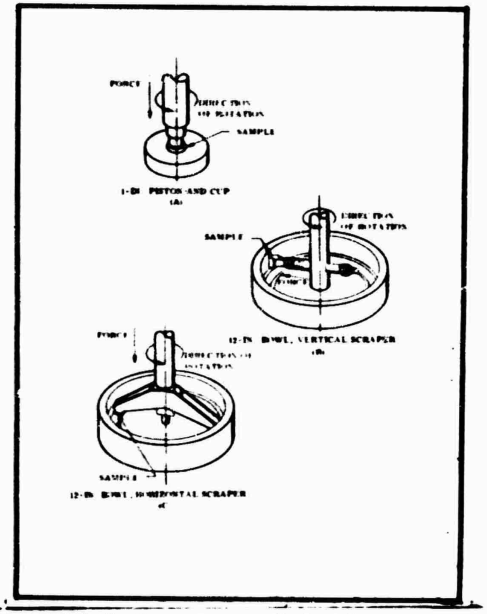
To conduct the test, a sample is placed on the anvil or friction strip and preloaded to the desired pressure using weights or hydraulic means. The anvil or friction



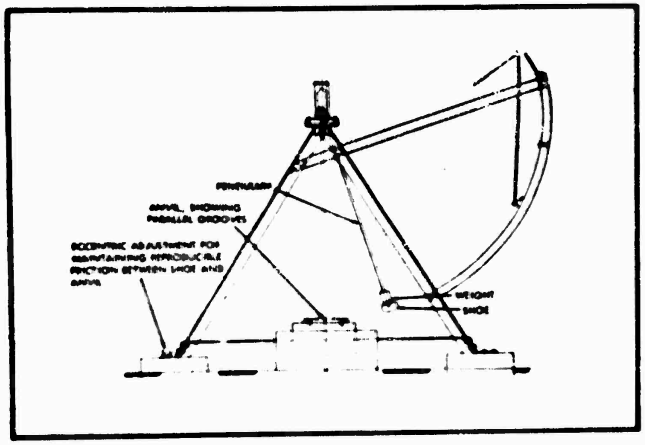
a. Thiokol strip friction test



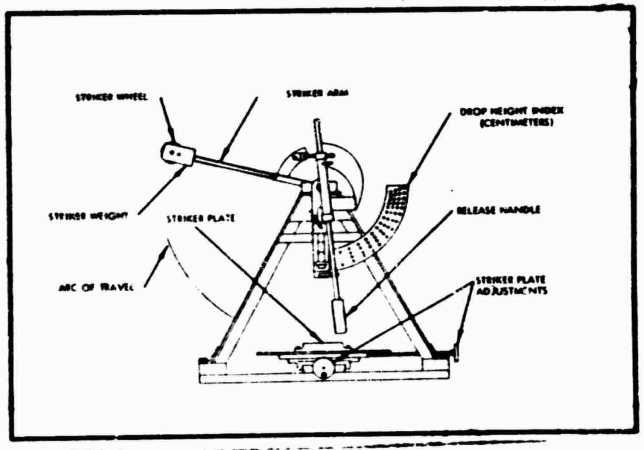
b. OD44811 strip friction test



c. Thiokol rotary friction test



d. Bu Mines pendulum friction test



e. NWC pendulum friction test

Fig B-2 Friction testers

strip is then set in motion by the direct impact of a pendulum weight, the impact through linkages of a dropweight, or the action of a motor. The preload pressure, the velocity of the anvil or strip, and whether or not an ignition occurred are recorded. The OD44811 strip tester (Ref B-2) handles only cast or pelletized samples. The others handle liquids, slurries and granular solids as well as cast or pelletized samples.

The pendulum friction testers consist of a fixed anvil and a pendulum arm fitted with a "shoe" which slides across the test material. Friction tests in this category include:

- the Bureau of Mines friction tester (Fig B-2d)
- the NWC China Lake friction tester (Fig B-2e)
- the Picatinny Arsenal friction tester, and
- the AWRE mallet friction test.

To conduct these tests, a sample is placed on the anvil. Then the pendulum is raised to the desired height and released. The pendulum will swing across the anvil several times before coming to a stop, with the shoe impacting and sliding across the sample several times. The material of the shoe and whether or not an ignition occurred are recorded. These testers handle liquids, slurries, and granular solids. They may or may not handle cast blocks or pellets.

The rotary friction testers consist of a bowl or cup and a moving (rotary) slider. The primary tester of this type is the Thiokol rotary friction tester (Fig B-2c). To conduct these tests, a sample is placed in the container. The slider is loaded and the motor turned on. The slider will continuously wipe across the container, pushing material between it and the container. Liquid, slurry or granular solids can be tested. It may be possible to replace the slider with a sample of cast or pelletized material.

Bulk material friction testers consist of a hemispherical sample with or without inert fillers and a stationary anvil. Tests in this category include:

- the Los Alamos large-scale skid test
- the Pantex large-scale skid test, and
- the AWRE charge oblique impact test.

To conduct these tests, the sample is formed into the proper shape. The sample is then allowed to impact and slide across the test surface. Due to the nature of the tests, only castable or formable solid materials can be used.

Applicability to Hazards Classification: According to our accident analyses, friction is one of the primary causes of accidents. Friction test(s) are required to gain a reasonable indication of the friction sensitivity of the material.

Discussion: There are many potentially useful friction tests, and for purposes of hazards classification it is necessary to reduce the number of choices. The criteria which can be applied are:

- the factors which are important in the frictional initiation of explosives
- the applicability of the tester to the various material states found in a process, and
- the ability of the tester to simulate actual friction forces in the process.

J. A. Brown (Ref B-3) conducted a study of friction fundamentals in explosives for Picatinny Arsenal in 1970. He discussed in some detail what causes friction and frictional heating. In his study, he considered three possibilities related to frictional characteristics which could lead to the frictional initiation of explosives. These are:

- Possibility 1: The important friction characteristics are those of the solid explosive surface itself.
- Possibility 2: The important friction characteristics are those of the container surface, not the explosive itself.
- Possibility 3: The important factor is not sliding friction at all, but rather heating from viscous or plastic flow of the explosive.

Possibility 1 says that there is a thick layer of explosive between the sliding surfaces such that the characteristics of the explosive are the controlling factor. This is pretty much simulated by the bulk material friction tests and the OD44811 friction test where the material-surface contact predominates. However, this type of friction is unlikely to occur within the process as it occurs primarily with solid materials. There are very few friction sources where solids are involved within a process. Its occurrence with liquids, slurries or pastes would be more akin to flow of the material. Also, the tests which were mentioned can be conducted only with materials which can be cast or pressed into a solid shape. Thus, this possibility and the tests can be ignored.

Possibility 2 says that there is a thin layer of material between the sliding surfaces such that the explosive acts only as a lubricant or as an acceptor of heat generated from the rubbing of the two surfaces. This type of friction can occur in the process such as in the pinching of a sample between the blade and the wall of a sigma blade mixer. The sliding block, rotary, pendulum,and to some extent the strip friction testers simulate this condition. The rotary friction testers allow continuous friction which may or may not be desirable, but they have difficulty in handling pressed or cast materials, so such tests will be eliminated. Pendulum friction testers yield no quantitative data, so they too are eliminated. This leaves just some of the sliding block and strip friction testers.

Possibility 3 says that initiation is caused by heating from viscous or plastic flow of the explosive, such as that which occurs during the extrusion of propellants. None of the current friction tests are applicable. Brown does suggest that the Susan and some of the impact tests would be applicable. Unfortunately, it would be difficult to isolate the friction data from the impact data in those tests. Brown does suggest a new test which is shown in Fig B-3.

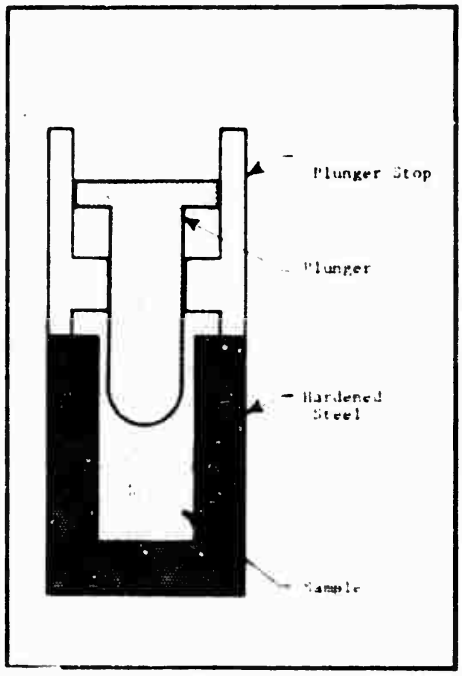


Fig B-3 Brown's suggested test for viscous friction

In his suggested test, a plunger would be used to force the material through an annular orifice formed by the plunger and the hardened steel cup. The plunger's stroke would be controlled to avoid impacting the bottom of the cup and would be guided so that it wouldn't contact the cup's walls. The test would be workable with liquids, slurries or pastes. However, it is not known how it would work with granular or solid materials. The test does not give details on the clearances or sizes that would be required or any indication as to the speed of the plunger or what quantities could be measured.

Best Standard Tests: The sliding anvil or strip friction tests seem to be the best because they can handle all material forms and because they simulate the process environment. Of these tests, the ABL friction tester and the Thiokol strip friction tester appear to be the most versatile.

Need for New Tests: Brown may be correct in claiming that heating due to viscous flow may be a problem. His test or a workable variation would be necessary to develop data to prove or disprove his hypothesis.

SMALL-SCALE IMPACT TEST
AND ADIABATIC COMPRESSION TEST

Objective: To estimate the energy density required to initiate a small sample of material by impact.

Operating Principle: A small sample of material is crushed between a fixed anvil and a movable hammer or striker. The impact energy is supplied by a falling dropweight.

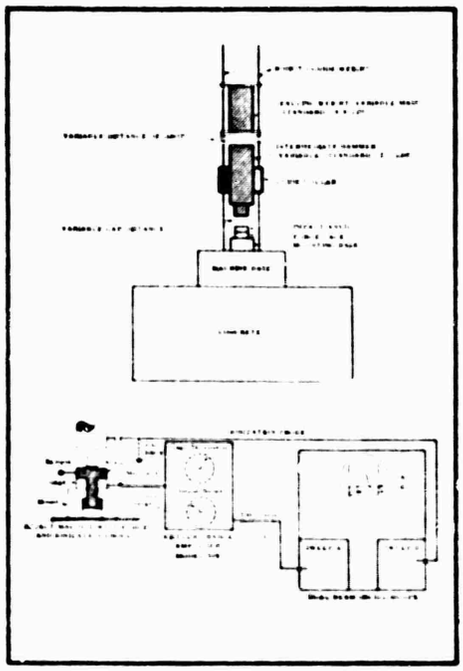


Fig B-4 Laboratory impact tests

Test Description: A small sample of material (usually 35 mg) is placed either on an anvil or in a cup on an anvil. A hammer of known weight is raised to a predetermined height above the sample and is released. The falling hammer or dropweight either impacts the sample directly (uncommon) or indirectly through one or more strikers. Ignition of the sample is detected by audio (sound), visual (smoke, char, flame) or infrared analysis (decomposition products). A typical tester is shown in Fig B-4. Impact testing is conducted in a sequence such that either the 50 percent sensitivity level or threshold initiation level is determined. This level is presented either in terms of the dropheight (for a fixed weight), the dropweight (for a fixed height), or energy if an energy measurement system is used.

There are many different impact machines in current use. In fact, each laboratory involved in explosives testing has a machine that's not exactly like anyone else's. However, there are a number of generically similar machines in use. Unless otherwise cited, these machines are described in Ref B-1. A number of laboratories use machines based on a design by the Explosives Research Laboratory and known as ERL machines. Variations of such machines are used at the U.S. Naval Ordnance Laboratory, Los Alamos Scientific Laboratory, Naval Weapons Center (China Lake) and the Lawrence Livermore Laboratories. Variations of the Rotter Machine are used at AWRE (GB) and CERL (Canada). Variations of the

Bureau of Mines machine are used at the Bureau of Mines (Fig B-5a), and ABL. Picatinny Arsenal (Fig B-5c and 5d) and Bureau of Explosives (Fig B-5b) use their own machines.

Applicability to Hazards Classification: According to our accident analyses, impact is one of the primary causes of in-process accidents. As such, a knowledge of impact sensitivity is important. The small-scale impact tests, while not totally accurate, do yield reasonable indications of impact sensitivity.

Discussion: The apparatus and procedures for small-scale impact testing vary from laboratory to laboratory. No two are precisely the same, although tests conducted using the ERL machines yield results that are quite similar. The results obtained on other machines differ from the ERL results. This is shown in Table B-1 which gives some of the operating characteristics and test results for the different machines.

Some of the test results from Table B-1 are plotted in Fig B-6. The figure shows that:

- there are considerable differences in the results of tests conducted on different machines
- ERL machines yield similar results
- the presence of grit (sandpaper) increases the observed impact sensitivity
- pelletized materials often display different impact sensitivities than granular samples
- there is no apparent correlation of results.

The last comment may be true only because of the scales used on the graph. One axis is the TIL results obtained by Radford AAP in energy units while the other axis shows the 50 percent heights.

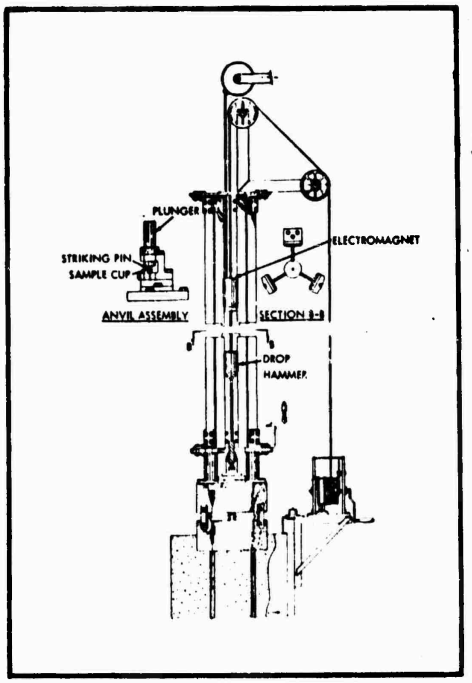
A tentative method of test for dropweight impact sensitivity of solid-phase hazardous materials has been developed by ASTM Committee E27, subcommittee 03 (Ref B-4). This method claims that the dropheight at which there is a 50 percent probability of ignition, h_{50}, can be normalized to account for different dropweight masses, m, and different striker surface areas, A, by: $(m/A)h_{50}$ = constant. This equation has not been applied to the data from Table B-1, so no claims to its validity will be made.

In assessing the impact data, it is important to point

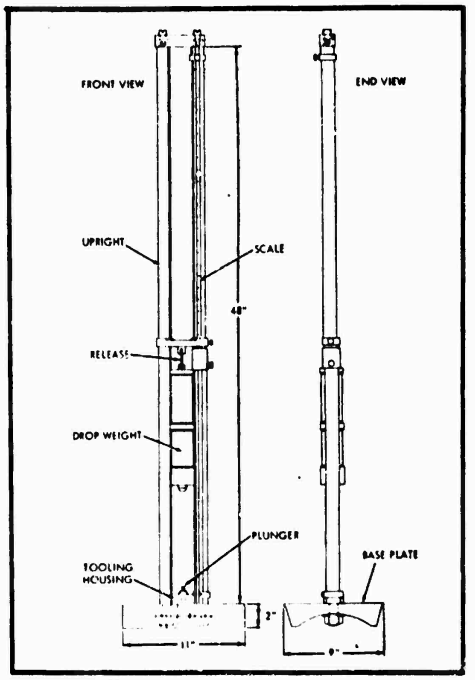
Table B1

Comparison of impact apparatus and test results

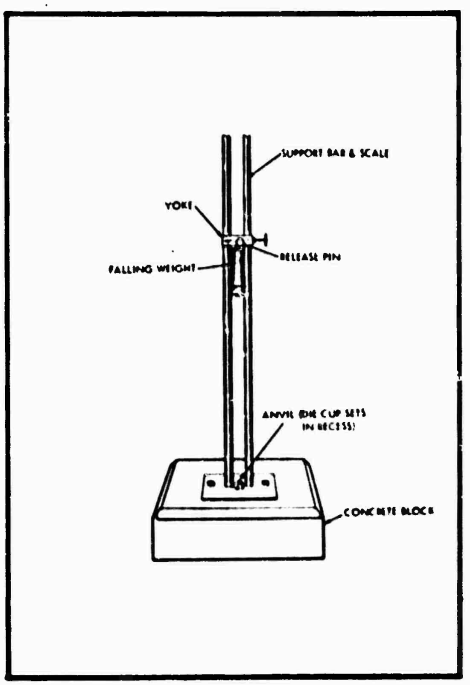
Laboratory	BuG	LOS ALAMOS		NWC CHINA LAKE	LAWRENCE LIVERMORE LABORATORIES			BuMINES	PICATINNY ARSENAL		BuEXPL	RAAP
Type Machine	ERL	ERL	ERL	ERL	ERL	ERL	ERL	BM	PA	Old BM	BE	BM
Drop weight (kg)	2.5,5	2.5	2.5	2.5	2.5,5	2.5,5	2.5,5	5	0.5,1,1.5, 2,2.5,3	0.5,1,2	3.63	2
Max Drop Height (cm)	320	320	320	33	177	177	177	330	---	100	76.2	---
Striker (cm)	1.58φx0.95	1.9φx1.02	1.19φx1.52	1.58φx15.9φ	1.19φx17.78	1.19φx17.78	1.19φx17.78	1.27φx1.9?	---	1?x?cm	---	---
Cup (cm)	---	---	---	---	---	---	---	1.27φx.64	?	1?x?cm	?	?
Type Device	1? sand-paper	1? sand-paper	12B rough-ened	1? sand-paper	1? sand-paper	12A sand-paper	12B rough-ened	Filter paper (liq)	---	---	---	---
Sample	35mg	40 mg	40 mg	35 mg pellet	35 mg pellet	35 mg pellet	35 mg pellet	35 mg	Full cup	20mg sol. 7 mg liq.	10 mg	---
Type Test	50%	50%	50%	50%	50%	50%	50%	50%	10%	10%	---	TIL
TYPICAL RESULTS												
PETN	12 cm	2.5 cm	2.5 cm	2.5 cm	11 cm	11 cm	20 cm	43 cm	15.2 cm	17 cm	---	---
RDX	2? cm	? cm	32 cm	19 cm	---	?? cm	---	79 cm	20.3 cm	32 cm	---	$3.05x?0^4$ j
Tetryl	?? cm	?? cm	---	?? cm	?? cm	30 cm	?? cm	94 cm	20.3 cm	26 cm	---	---
TNT	1?? cm	??? cm	>320 cm	?? cm	8? cm	?? cm	---	183 cm	38.1 cm	>100 cm	---	$4.45x10^4$ j
HMX	?? cm	?? cm	?? cm	1? cm	?? cm	?? cm	39 cm	---	22.9 cm	32 cm	---	$3.0x10^4$ j
Comp B	??? cm	?? cm	---	?? cm	---	---	---	---	15.6 cm	75 cm	---	$2.8x10^4$ j
Lead Azide	? cm	---	---	---	---	---	---	---	12.7 cm	17 cm	---	$2.2x10^4$ j



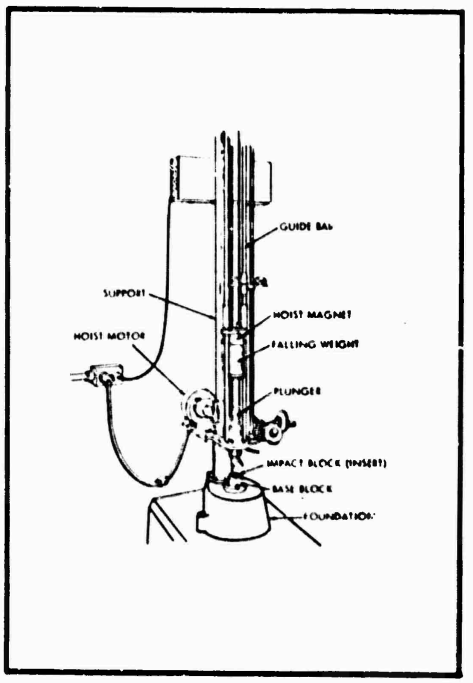
a. Bureau of Mines



b. Bureau of Explosives



c. Picatinny Arsenal



d. Picatinny Arsenal

Fig B-5 Different impact testing machines

out that the usual way of calculating kinetic energy, KE = mgh (m = mass of dropweight, h = dropheight) is in error when one or more intermediate strikers are used. This is due to the fact that all of the energy in the dropweight is not transferred to the striker and the sample. The kinetic energy is really given by:

$$KE = \left[\frac{M^2}{(M + m)^2} (1 + e)^2\right] mgh \qquad (1)$$

where M is the mass of the intermediate striker and e is the coefficient of restitution of the two impacting surfaces (Ref B-5). For similar machines, such as the ERL machines, the masses of the dropweight and striker are identical. What is different are the materials of construction and hardness,which yield different coefficients of restitution.

These coefficients are relatively close so that the impact energies are quite similar, yielding similar test results. However, when different machines are used, the masses may vary in addition to the coefficients of restitution so that different dropheights are required to produce the same energies, even when using the same dropweight. In addition, the sample size varies, so that the area over which the energy is applied changes, giving different energy densities. Therefore, if more than one machine is to be used in the impact tests, some means is required for corelating the energies and energy densities.

The sample condition and the tools utilized in the impact tests also are important. Tests conducted on ERL machines using pelletized samples yield different results, in some cases, from tests conducted on granular samples. For the most part, when differences are apparent, the pelletized samples are more sensitive. This could be due to the extreme deformation that occurs in the pellet as compared to the granular sample. This deformation and the resultant viscous flow could be a frictional type ignition. The tests conducted in a cup are quite different from those where the sample is merely placed on the anvil. The cup provides much more confinement and, if the cup and plunger are well sealed, could include adiabatic compression as an ignition mechanism. The presence or absence of grit also affects the test results significantly. Lawrence Livermore Laboratory and Los Alamos Scientific Laboratory ran tests using either sandpaper (a source of grit) or a roughened anvil. The tests using the sandpaper showed a significantly higher sensitivity than those with the roughened anvil, both with granular and pelletized samples.

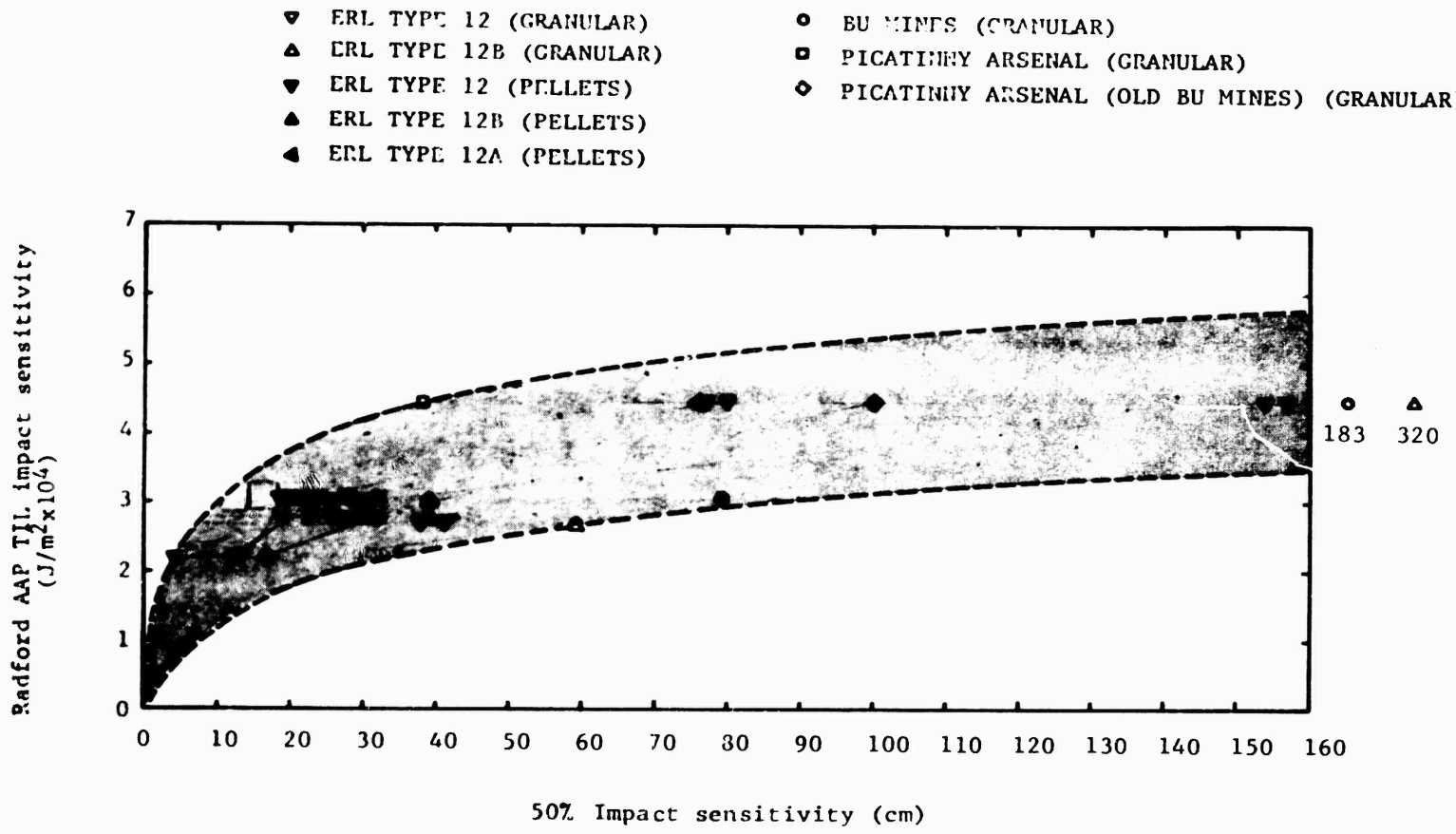


Fig B-6 Comparison of impact sensitivity test results from different impact machines and material forms

Ignition of propellants and other materials has occurred as a result of the adiabatic compression of gaseous bubbles. This compression can occur as a result of an impact. If a gas bubble is compressed very rapidly, so that no heat enters or leaves the system, very high temperatures can be attained in the compressed gas. The theoretical temperature (Ref B-6) which can be attained by adiabatic compression is

$$T_2 = \left(\frac{P_2}{P_1}\right)^{\frac{n-1}{n}} \cdot T_1 \qquad (2)$$

where T is temperature (°K), P is pressure, n is the ratio of specific heats and the subscripts 1 and 2 designate initial and final conditions. If a bubble is rapidly compressed to about 1.4×10^7 Pa, a bubble temperature of almost 900°C can be attained. This temperature may be sufficient to ignite the material in question. However, studies by IITRI for AFOSR (Ref B-7) have shown that the material outside the bubble does not heat very rapidly and that time is also an important factor.

OD44811 (Ref B-2) purports to have a test for adiabatic compression. This test basically is a small-scale impact test which utilizes a specially designed and sealed cup and plunger arrangement. This is shown as Fig B-7. Basically, a dropweight is released from a given height and is allowed to directly impact a pressure ram. The pressure ram has an "O" ring seal and builds up pressure in an air gap and the explosive sample. The ram is such that direct impact of the sample is impossible. Ignition is said to occur if the pressure relief hole is blown open. It is not known whether this or similar tests actually simulate adiabatic compression.

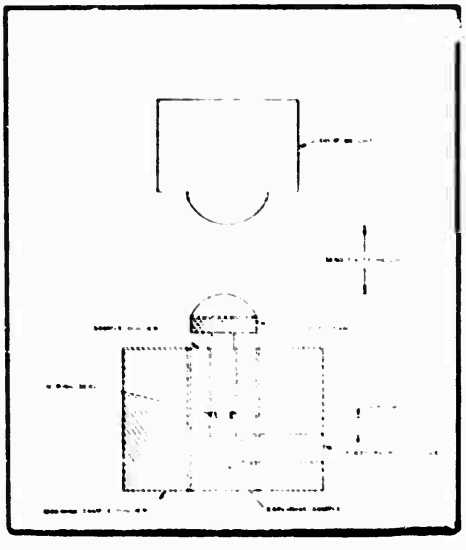


Fig B-7 OD44811 adiabatic compression test (Ref B-1)

Best Standard Tests: No best standard tests can be chosen. The only decision on the standard tests is to eliminate the pelletized sample except in cases where a pellet is what actually exists in the process. It would be best, however, if an impact gage system were used to measure the energy actually delivered, and to standardize and mechanize

the go-no go criteria.

Need for New Tests: As adiabatic compression may be measurable in special drop tests, it would be desirable to develop a standard adiabatic compression test.

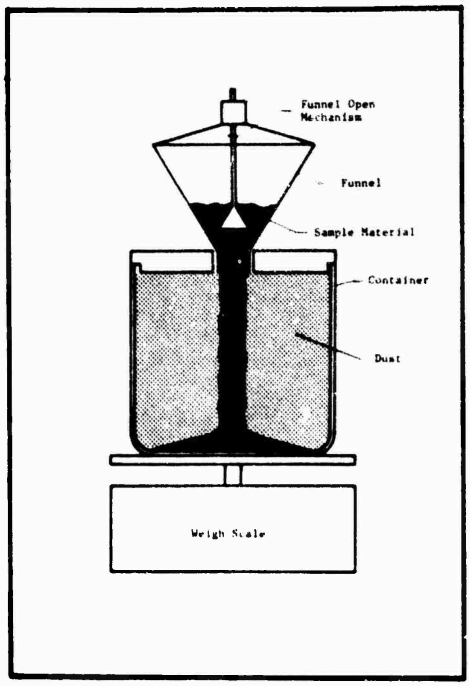
DUSTING TESTS

Objectives:

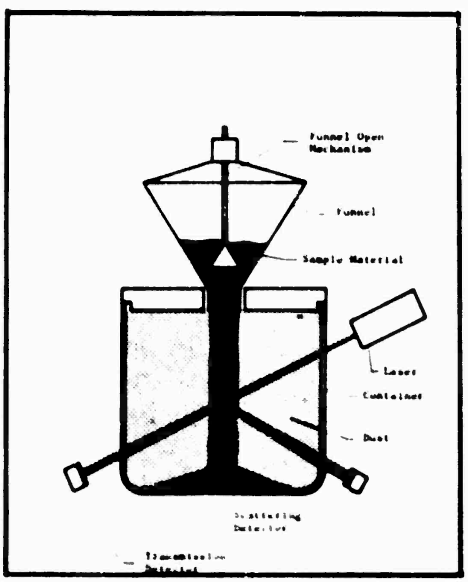
1. (required) To determine if dusting is likely, and
2. (optional) To determine the most likely dust concentrations.

Operating Principle: The sample is transferred from one container (funnel) to another container (jar) as is shown in Figs B-8 a and b. During the free fall transfer process, some of the sample will remain suspended in the air after the majority of the sample has settled. The amount of dusting is determined by the weight of material suspended in the air in the weight method, or by the intensity of scattering or the decrease in transmission of the laser beam in the laser method.

Test Description: A known weight of the sample material is placed in the funnel. After checking out the instrumentation, the material is allowed to flow into the container. 1) In the weight method (Fig B-8a), the weight of the container is recorded as a function of time. A weight vs time trace like that in Fig B-9 is expected. Time, t, is the settling time, and W_1 is the greatest weight of dust remaining in the air. 2) In the laser method, Fig B-8b, either the intensity of the back scattering or the intensity of the transmitted beam is recorded as a



a. Weight method



b. Laser method

Fig B-8 Dusting tests

function of time. Time, t, is the settling time with either S_{max}, $S_{0.50}$ or $t_{0.50}$ from Fig B-10 being taken as an indication of the severity of dusting.

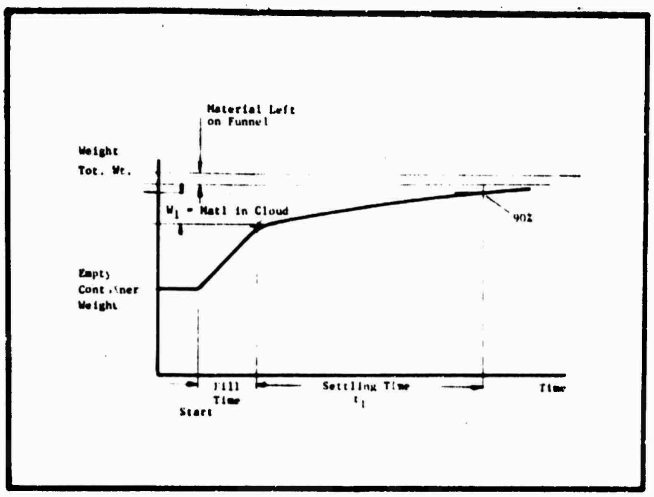
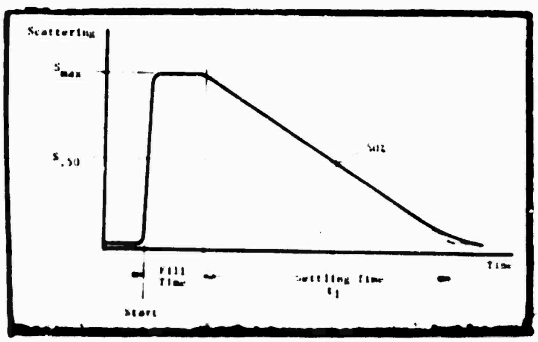
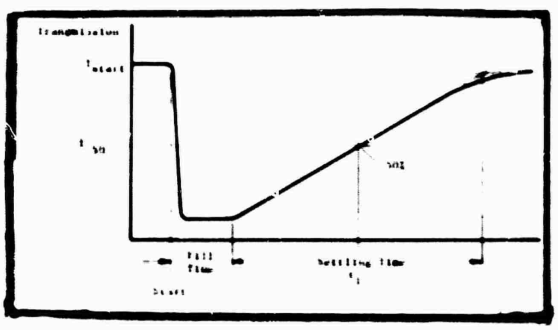


Fig B-9 Weight method



a. Transmission approach



b. Backscattering approach

Fig B-10 Laser method

Application to Hazards Classification: Dusty atmospheres can occur within process plants. This dust test is intended to provide an unambiguous basis for deciding if dust is a problem and if the dust sensitivity tests should be conducted.

Discussion: The dusting test is needed to give a Yes or No indication as to whether dusting is a potential problem. The test is probably unnecessary with large particles or moist materials which would not dust, or with light fluffy materials (i.e., sulfur) where dusting is definitely a problem. However, between these extremes, some dusting criteria are

necessary. The laser method would produce an unambiguous Yes or No indication once general criteria for such a decision are developed. It may also provide data as to the concentration of the dust. However, the backscattering method probably depends upon the reflectivity of the material. The weight method would produce more information. However, due to the small quantity of material that may be suspended as dust, it may be difficult to get sufficiently accurate weight readings.

The Bureau of Mines has been making modifications to the Hartman Dust Apparatus. One of the most recent modifications is the inclusion of a dust concentration sensor. According to Dr. Hartsburg (Ref B-8) the sensor consists of two fibre optic probes spaced a known distance apart with one probe connected to a light source and the other to a detector. Dr. Hartsburg claims that the decrease in transmission of light caused by the dust can be related to the dust concentration if the separation distance between the probes and the size distribution of the dust are known.

Approaching this problem mathematically, the decrease in transmission is proportional to the portion of the light beam being blocked by a dust particle(s). Thus,

$$A_s - nA_p = A_s \left(1 - \frac{\text{act trans}}{100\% \text{ trans}}\right) \qquad (1)$$

where A_s is the area of the light beam, A_p is the average projected area of a particle, n is the number of particles in the beam, act trans is the intensity of the beam in the dust cloud and 100% trans is the intensity of the beam transmitted in the absence of dust. The concentration of dust within the path of the light beam is:

$$\text{Concentration} = \frac{nV_p\rho_p}{A_s \ell} \qquad (2)$$

where V_p is the average particle volume, ρ_p is the average particle density and ℓ is the distance between the two fiber optic probes. Combining Equations 1 and 2 and simplifying yields:

$$\text{Concentration} = \left(\frac{\text{act trans}}{100\% \text{ trans}}\right) \left(\frac{V_p}{A_p}\right) \left(\frac{\rho_p}{\ell}\right) \qquad (3)$$

An assumption that the particles are roughly spherical in shape with an average radius of r_p allows Equation 3 to be further simplified to:

$$\text{Concentration} = \left(\frac{\text{act trans}}{100\% \text{ trans}}\right)\left(\frac{\rho_p}{\ell}\right)\left(\frac{1}{3} r_p\right)$$

This final equation shows that the concentration can be obtained by the probes if the separation distance between the probes (ℓ), the size of the particles (r_p), and the density are known (ρ_p). This shows that Dr. Hartsburg's apparatus works and indicates that, in principle, IITRI's apparatus also should work.

Standard Tests: There are no tests known to IITRI which are applicable. The tests described in the preceding text are concepts devised in this program.

New Tests: A new dust test should be developed, possibly along the lines of the concepts developed in the previous text.

TRANSITION TESTS

Objectives:

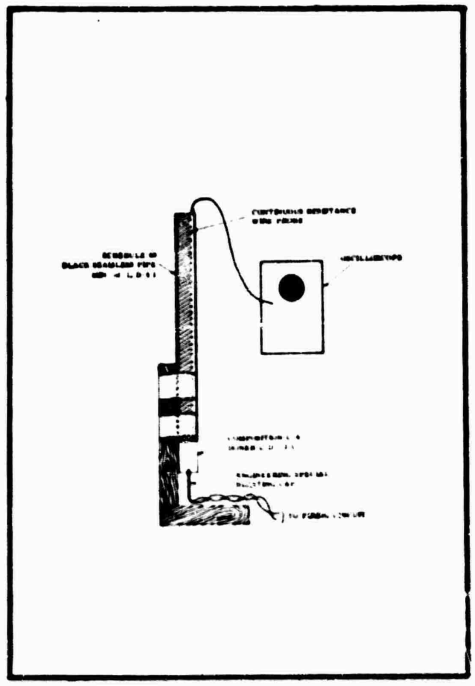
1. To determine if a material will detonate, and to establish the critical diameter for propagation (critical diameter test).

2. To determine if a material will react explosively when initiated by a flame, and to establish the height at which the transition occurs (critical height test).

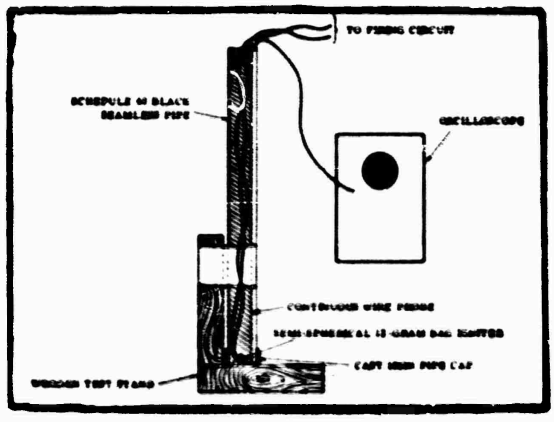
Operating Principles:

1. Critical diameter test: materials are initiated by a detonating high-explosive donor. The material diameter is varied and the ability of the material to propagate the detonation is determined.

2. Critical height test: materials are subjected to flame ignition. The distance (height) is determined at which a transition from burning to deflagration or detonation occurs.



a. Critical diameter test



b. Critical height test

Test Description:
1) A typical test arrangement for determining the critical diameter is shown in Figure B-11a.

Fig B-11 Transition tests (Ref B-9)

Basically, the arrangement consists of a tube filled with the test material, an explosive donor, and a means of measuring the reaction velocity as a function of tube height. The test can simulate various process configurations by choosing the tube material and wall thickness which best simulates the type of confinement in the process. Pressure gages and other instrumentation can be used to estimate the reaction severity. The diameter of the tube is varied, while maintaining the same length to diameter (L/D) ratios for the tube and explosive donor. The critical diameter is the smallest tube inside diameter for the given tube material and wall thickness at which propagation occurs.

2) A typical test arrangement for determining the critical height is shown in Fig B-11b. Basically, the arrangement consists of a tube filled with test material, a flame source, and a means of measuring the reaction velocity as a function of tube height. This test utilizes the same tube materials as the critical diameter tests and begins at a diameter larger than the critical diameter. Additional tests are conducted at still larger diameters so that a relationship can be found between diameter and critical height. The critical height is the distance along the tube at which a transition from slow burning to deflagration or detonation occurs.

Application to Hazards Classification: The critical diameter test determines if the material is detonable and at what diameters it is detonable. The process size can be compared to the critical diameter and used to determine if a detonation is possible. The critical height test indicates if a fire, once started, could progress to a detonation.

Discussion: The detonability of a material is often assessed by using cap sensitivity or card gap type tests. These tests are fine for use on explosives having critical diameters of less than 2 cm. However, many propellants have critical diameters in tens of centimeters. In these cases, the test size is much smaller than the critical diameter so that detonation is never observed. As such, it is necessary to determine detonability in tests in which the size of the sample can be varied. The critical diameter test allows for a variable test size. In addition, it provides information on the critical diameter of the material. If the process never uses a diameter larger than the critical diameter, it should be unrealistic to expect a detonation under any condition.

A flame ignition source is much more likely within a process than a shock wave of the type produced by a high explosive. Therefore it is important to determine the

distance required for the material to make the transition from burning to deflagration or detonation. If the largest process length is less than this transition distance (critical height), it is unrealistic to expect a fire to cause an explosion.

The critical height may vary depending upon the energy content and rate of energy release of the igniter. Therefore, the igniter used in the tests would be controlled so that all such tests would be comparable.

Best Standard Tests: All of the critical diameter, critical height tests are basically similar. These tests differ mostly in the materials of construction. As these tests would utilize materials which closely simulate the actual in-process conditions, this variable is eliminated and the tests become more alike.

ELECTROSTATIC DISCHARGE (LAYER) TESTS

Objective: To determine if an electrostatic discharge will ignite the sample material and to determine the spark energy at which ignition would occur.

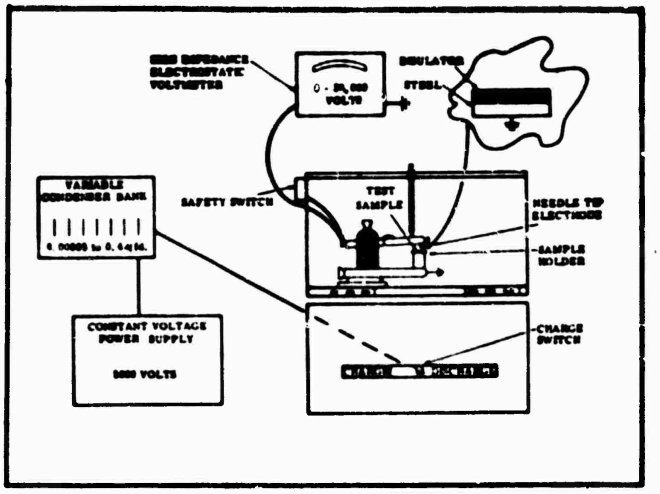


Fig B-12 Typical electrostatic discharge test

Operating Procecure: Electrostatic energy, stored in a charged capacitor, is discharged to a layer of the sample material.

Test Description:
A sample is placed in or on a sample holder. A capacitor is charged by a high-voltage source. To discharge the capacitor, either the discharge needle is lowered until a spark is drawn through the material, or the discharge needle is set at a predetermined height above the material and a switch is closed allowing the spark to jump the gap. Obviously, the needle must be set at a height at which discharge can occur.

Application to Hazards Classification: Materials can gather and store electrostatic energy simply by flowing over a surface such as the wall of a hopper. Humans can likewise generate and store charges. These stored charges, under proper conditions, can be discharged into in-process material. This test determines the sensitivity of the material to such discharges.

Discussion: Most laboratories take the spark energy as that given by the formula $E = 1/2\ CV^2$ where E is the energy in joules, C is the capacitance in farads and V is the potential in volts. However, it has been IITRI's experience that the formula overestimates the energy losses in the conductors, switches, discharge gap, or the resistivity of the test specimen. Thus it is preferable for the energy to be measured in each test, or for the discharges to be calibrated for each series of tests.

Best Standard Tests: All standard electrostatic discharge tests are essentially the same. Any such test can be used provided the energy is measured in each test, or the discharges are calibrated and checked prior to each series of tests.

IMPINGEMENT TESTS

Objective: To determine whether a material, moving at some velocity and striking a target, will initiate.

Operating Principles:

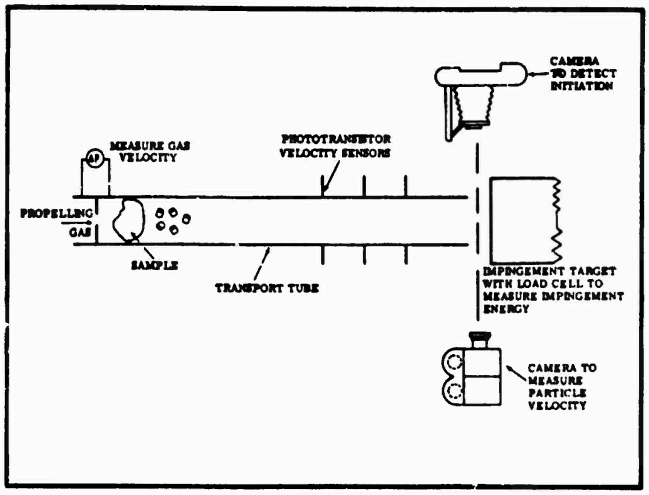
1. Propelled: the sample is propelled by moving air or other suitable fluid at a known velocity and is impinged on a massive target.

2. Free Fall: the sample is dropped and allowed to fall freely against a target.

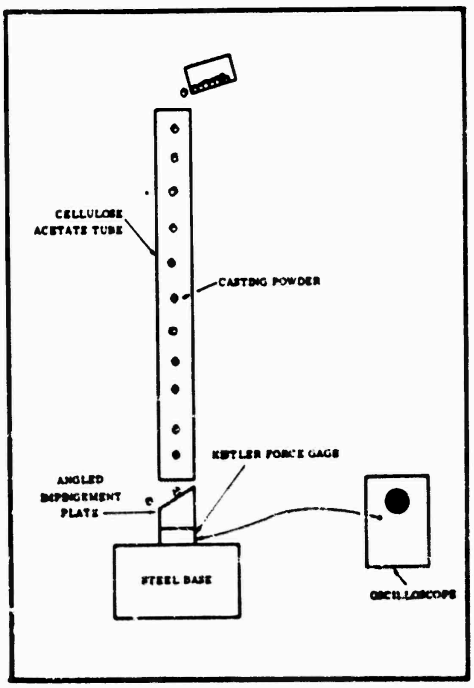
Test Description:

1. Propelled: the sample is injected into a moving stream of air. The air carries the sample at some measured velocity into a target plate.

2. Free Fall: the sample is dropped from some known height and is allowed to fall onto the target plate.



a. Propelled



b. Free fall

Fig B-13 Impingement tests

Application to Hazards Analysis: Impingement is a probable stimulus for accidents occurring during conveying and screening. It is important that the threshold initiation velocity due to impingement be greater than the highest velocity that can be achieved in the process. If this

condition does not exist, accidents caused by impingement are likely to occur.

Discussion: This test simulates the conditions that exist when in-process materials are conveyed by a flowing gas or liquid, or simply are allowed to fall freely, as from a belt conveyor to a hopper bin. The target materials, surface finish and surface angle can be changed to examine the effects of these characteristics. The test results are reported as the impingement velocities that cause ignition. These impingement velocities are measured quantities. The upper limit of impingement velocities is approximately 335 m/s, the sonic speed in air.

Best Standard Tests: The tests described were taken from CPIA 194 (Ref B-9).

ELECTROSTATIC DISCHARGE (DUST)TEST

Objectives:

1. To determine whether a finely divided solid material will ignite when dispersed in air or any other gas.

2. To determine the concentrations at which such ignitions are possible, and

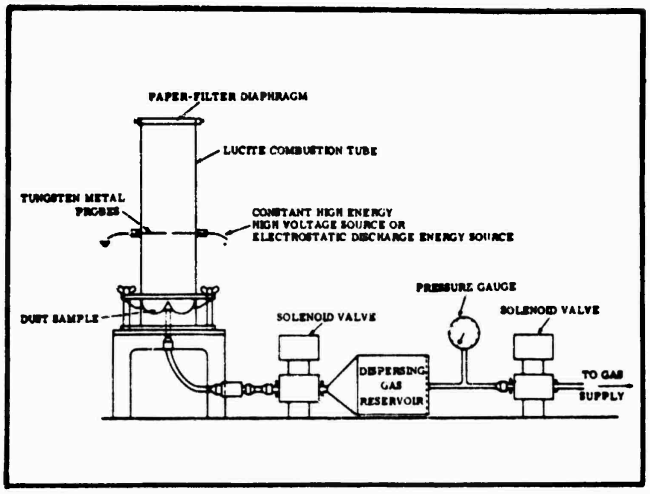


Fig B-14 Electrostatic discharge test

3. To determine the electrostatic discharge energy necessary to ignite the dust.

Operating Principles: A suspended dust is created by dispersing the material into air with a gust of gas. A continuous spark is used to determine ignition concentrations, and the electrostatic energy stored in a capacitor is used to determine ignition energy.

Test Description: The Bureau of Mines "Hartmann" Dust Explosibility Test Apparatus (Ref B-10) is used for this test. Basically, a momentary dust cloud is produced within the chamber by directing a blast of air on the sample. In one series of tests, the dust concentration is varied while applying a continuous high-voltage, high-energy spark. This allows determining the concentration at which the dust will ignite. In the other series of tests, a fixed concentration is used which is greater than the threshold ignition concentration. Different amounts of energy stored in a capacitor are used to determine the energy needed to ignite the dusts.

Applicability to Hazards Classification: Dusts can burn, deflagrate, or even detonate depending on the ignition source and confinement. This test determines the minimum concentration of dust at which ignition will occur, and the level of electrostatic discharge energy required to ignite a cloud if it is above the minimum concentration. Both data are important for assessing the hazards associated with dust clouds.

Discussion: This test is conducted only when the dusting test has shown that dusting is a potential problem. The data derived from the test will indicate whether a dust collection system is needed to reduce the dust concentrations, and the ESD energy that would be required to ignite the dust cloud. The gas used in this test can be varied to simulate an inert gas operation. The rate of pressure rise also can be measured with one variation of this apparatus. This rate indicates the severity of the dust explosion.

Best Standard Tests: The Bureau of Mines test utilizing the "Hartmann" apparatus is the standard test for dusts.

Objectives:

1. To detect the occurrence of excthermic and/or endothermic reactions in the material as the material is heated, and

2. To determine the autoignition temperature at that heating rate.

Operating Principle: The sample and an inert reference material are placed in an environment whose temperature is increased at a given rate. The temperature of both the sample and inert reference are measured, with the difference in temperatures being recorded. The test is continued until autoignition of the material occurs.

Test Description: This test is performed by placing a small sample of the material and a reference material (e.g., alumina or glass beads) in the same container, and heating the container at a uniform rate. Each item is instrumented with thermocouples so that individual measurements of temperature can be made. A prechosen, constant rate of temperature rise is maintained, which continues until the test material ignites. The difference between the temperatures of the test sample and reference material is recorded. This difference shows the exotherms and endotherms of the sample. The temperature at which the sample ignites also is recorded. Different results, especially in the autoignition temperature, can be obtained with different heating rates. This test is, or should be, performed at different heating rates.

Applicability to Hazards Classification: The temperature at the onset of an exotherm represents an upper limit for which a process should be designed to operate. The autoignition temperature gives an indication of the temperature that must be reached before ignition could occur from a steady heat source such as would occur due to failure of cooling equipment or steady frictional heating.

Discussion: There are other thermal tests which could be considered for this application. Among these are the autoignition test, the vacuum stability test, and the differential scanning calirometer test (DSC). In the autoignition test, a sample is placed in an oven, or bath, or on a surface maintained at a constant temperature. The autoignition temperature is the temperature of the oven, bath, or surface at which the sample ignites in 0.1, 5, 10 or ... seconds.

In the vacuum stability test, the sample is placed in

a test tube and is kept at a constant temperature for a number of hours. The volume of gases produced during this time is measured, with the results being given in terms of ml of gas/X hr @ Y°C. The DSC is similar to a DTA in that endotherms and exotherms are detected. Other specialiazed tests such as "cook-off" or bonfire tests also exist.

All of these tests suffer from deficiencies not found in the differential thermal analysis (DTA) test. The autoignition test does not yield any information on the temperatures at which exotherms begin. The vacuum stability test becomes less than meaningful with materials containing volatile solvents. The DSC requires comparatively more expensive equipment.

Best Standard Tests: The most practical type of thermal analysis for this application is differential thermal analysis or DTA. The test is conducted basically in the same manner by all laboratories. The sample sizes and the equipment may differ; however, there is no real difference between the tests.

Objectives: To measure:

1. the conductivity
2. the permittivity, and
3. the breakdown electric field strength of the test sample.

Operating Principle:

1. The conductivity is obtained from the resistivity of the test sample.
2. The permittivity is obtained from the dielectric constant of the material. The dielectric constant can be estimated from the change in capacitance of a parallel plate capacitor with or without an insert of the sample material.
3. The breakdown electric field strength is obtained by applying a high potential across the sample and increasing the potential until breakdown occurs.

Test Description:

1. Conductivity - the conductivity of a material is the inverse of its resistivity. Thus by measuring the resistivity, the conductivity is also known.
2. Permittivity - the permittivity of a material is related to the dielectric constant of the material

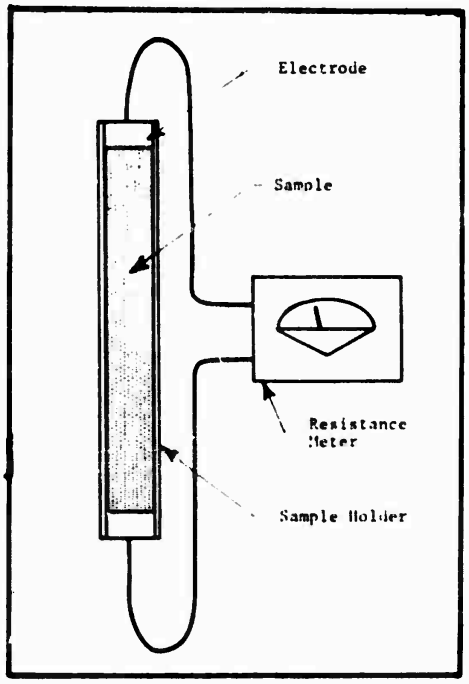


Fig B-15 Conductivity test

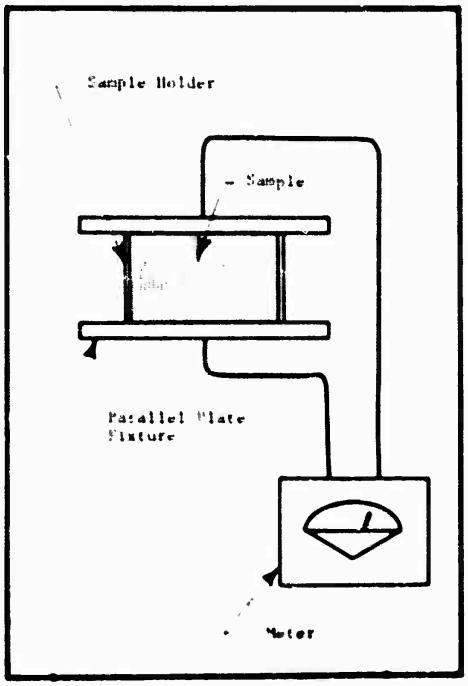


Fig B-16 Permittivity test

The measurement of the permittivity is only slightly more complex. First, one measures the dielectric constant of the material. This can be estimated by making a parallel plate capacitor, then measuring its capacitance both with and without the sample between the plates. The ratio of the capacitances is a good approximation of the dielectric constant. It is best for the sample not to go to the outer edges of the parallel plates as edge effects are easier to account for. Once the capacitances are found, the permittivity is calculated from $\varepsilon = K \varepsilon_o = (C_s/C_o)\varepsilon_o$ where C_s is the capacitance with the sample between the plates and C_o is the capacitance with only air between the plates.

The measurement of the capacitances with the area of the plates larger than that of the sample (a ratio of 4:1 is suggested) simplifies the calculation for fringe capacitance. With the sample material in place, the fixture can be considered essentially as two capacitors in parallel, i.e., the capacitance of the area covered by the sample comprising one, and the remaining plate area separated by air plus the edge, fringe, and other stray capacitors comprising the other. With this arrangement, the parasitic capacitances are essentially constant in both the loaded and unloaded fixtures.

The use of the parallel plate capacitors is relatively simple for cast, machined or pressed samples as solid disks can be made for insertion into the fixture. Liquids, slurries and granular solids are another matter. It should be possible to use s standard circular ring between the plates to hold such sample materials. As the effect of the ring would be the same in both the loaded and unloaded fixtures if the ring is always in place, the effect of the ring can be accounted for.

The breakdown or dielectric strength of the material can be measured by applying a potential across a sample of material through cylindrical electrodes placed on either side of the sample. Care must be taken so that breakdown occurs within rather than around the sample. Also, as energy is applied to the sample, ignition is possible. The potential applied across the sample when breakdown occurs is the breakdown voltage; and the breakdown voltage divided by the electrode spacing is the breakdown electrified strength or breakdown field density.

Best Standard Tests: There are no standard tests for these properties. Rather, these properties are functions of a material's characteristics and the laws of physics, so that any similar tests would be adequate. The tests described above were those described by E. E. Walbrecht in Reference B-12.

through the equation $K = \varepsilon/\varepsilon_o$ where ε is the permittivity of the sample, ε_o is the permittivity of a vacuum and K is the dielectric constant. The dielectric constant is defined and can be determined by measuring the effect of the sample material on the capacitance of a capacitor.

3. Breakdown strength is the electric field strength at which a spark is capable of passing through the sample. It can be determined by varying the field strength across a sample and determining the field strength at which breakdown occurs.

Application to Hazards Classification: Ignition by electrostatic discharge was identified as a major cause of accidents. The quantities measured in this test are electrical properties which relate to the propensity of the material to store electrostatic charges and to the maximum electric field that can be built up (energy before a discharge occurs.)

Discussion: The ability of a material to store electrostatic charges is related to its ability to give up the charges. If the relaxation time is quite fast, charges will be given up as fast as they are acquired. If the relaxation time is slower, more charges will be acquired than are given up, and the electric charge will build up. The relaxation time constant, τ, of a material can be calculated from $\tau = \varepsilon/\sigma$ where ε is the permittivity and σ is the conductivity.

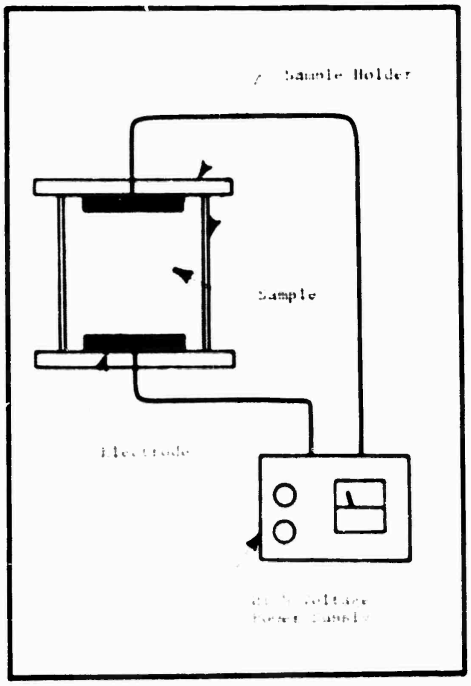


Fig B-17 Breakdown strength test

The charge density q is proportional to $(\varepsilon/\sigma)(1-\exp(1-\sigma t/\varepsilon))$ where t is time (Ref B-11). The maximum electric field strength is proportional to q/ε. Thus, the conductivity and permittivity are very important.

The measurement of the conductivity is relatively simple. Basically, one measures the resistance R of a sample of area A and length ℓ. Then, the restivity is $\rho = AR/\ell$. The conductivity is the reciprocal of the resistivity or $\sigma = r^{-1} = \ell/AR$.

LARGE-SCALE IMPACT TESTS

Objective: To estimate the energy or velocity of impact required to ignite large samples of a material.

Operating Principle: A large sample (grams to kilograms) of the test material is impacted by or against a large metal plate.

Test Description: There are four different types of tests which can be considered large-scale impact tests. These are:

- Large-scale drop tests
- Large-scale skid tests
- SUSAN tests, and
- Flyer plate tests.

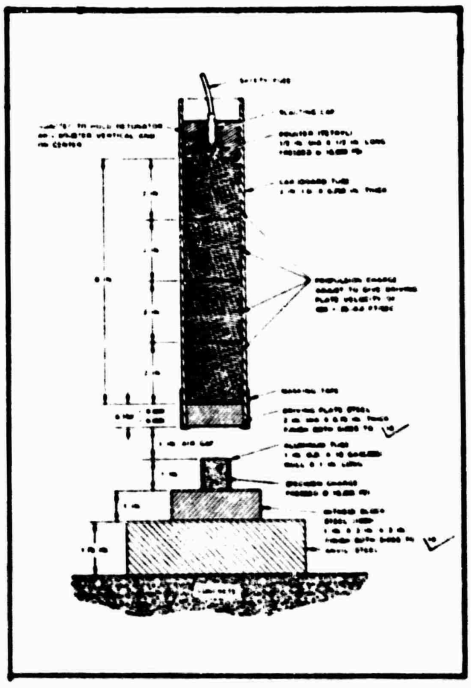


Fig B-18 Flyer plate test (Ref B-13)

The large-scale drop tests consist of dropping large charges of material from a given height (usually 10 to 30 m) onto a steel plate. The plate may or may not have spikes or protrusions. The material is generally packaged in its shipping container for these tests. A variation of this test is to place the sample on a rigid anvil and to drop a weight onto the sample.

The large-scale skid test consists of impacting a hemispherical charge of the sample material on a rigid and often coated steel anvil. The charge is suspended on a pendulum type device so that sliding friction also occurs. This test is described in more detail in the description of friction tests.

The SUSAN test consists of firing a special sample-filled projectile at a rigid target plate (Fig B-19). Pressure gages are used to record the pressure which is the basis for estimating the energy released. This energy release is plotted on a graph versus the projectile velocity. While impact is nominal stimulus, friction caused by slippage across a fracture plane or extrusion and pinching between the projectiles and the target plate also occur.

The flyer plate test, Fig B-18, consists of explosively

driving a metal plate at a sample of material placed on an anvil. Variations of this test have been used where the sample is placed on the "flying plate". Usually, the plate is kept at a constant size and weight with the plate velocity being varied until ignition occurs.

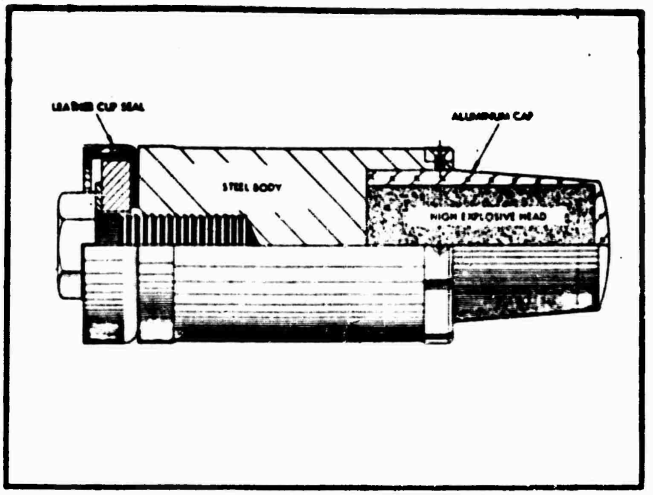


Fig B-19 SUSAN projectile (Ref B-14)

Application to Hazards Classification: Large-scale impacts can occur within a process. An example is the dropping of a container of in-process material being taken from one batch operation to another. This test provides an indication of the susceptibility of the material to such large-scale impacts.

Discussion: It is useful to compare the different types of large-scale tests. In terms of available energy, the SUSAN and flyer plate tests can supply much more energy than the drop or skid tests simply because they are driven by explosives or propellants rather than by gravity. For example, in a series of tests conducted by IRECO for Commercial Solvents (Ref B-15), there were 6670 J available by dropping a 45.4 kg plate from 15.4 m onto a 55 gal drum of nitromethane, 3.4×10^4 J from dropping the drum onto a steel plate and 5.29×10^6 J using an explosively driven flyer plate.

In terms of cost, the drop tests are the least expensive as they utilize the material as is in its shipping container. The skid tests are slightly more expensive as the sample must be formed into a hemisphere. The flyer plate tests are moderately more expensive due to the need for the driver plate and driver explosive. The SUSAN tests are considerably more expensive due to the initial cost of the firing device and the cost of manufacturing and loading the special projectiles.

In terms of being able to handle different material states, the drop and SUSAN tests are the best as they have natural containers for liquids, slurries and granular solids. The flyer plate tests can handle all types of materials if

a container is used. The container will have some effect on the test results. However, this effect should be no greater than the effect of the container in the drop tests or the projectile in the SUSAN tests. The skid test can utilize only formable solid materials and cannot utilize liquids, slurries or granular solids.

In terms of utility of results, the flyer plate test is the best as mathematical models of the test have been developed which are capable of many things, including the estimation of hot spot temperature-time profiles (Ref B-7, B-16). Similar models have not been developed for other tests.

On the basis of this comparison, the flyer plate test appears to be the best overall.

Best Standard Test: The flyer plate test is the best. It will, however, require the establishment of standard sizes for the flyer plate and the charge as well as definite criteria to distinguish between degrees of "GO's".

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