(12) LEVEL III

b.5

CONTRACTOR REPORT ARLCD-CR-80025

RECOMMENDED HAZARD CLASSIFICATION PROCEDURES FOR IN-PROCESS PROPELLANT AND EXPLOSIVE MATERIAL

HYLA NAPADENSKY
RONALD PAPE
IIT RESEARCH INSTITUTE
CHICAGO, ILLINOIS

RICHARD RINDNER, PROJECT LEADER
DANIEL SATRIANA, PROJECT ENGINEER
ARRADCOM

SEPTEMBER 1980

US ARMY ARMAMENT RESEARCH AND DEVELOPMENT COMMAND
LARGE CALIBER
WEAPON SYSTEMS LABORATORY
DOVER, NEW JERSEY

APPROVED FOR PUBLIC RELEASE: DISTRIBUTION UNLIMITED.

UNCLASSIFIED
SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

REPORT DOCUMENTATION PAGE

READ INSTRUCTIONS BEFORE COMPLETING FORM

1. REPORT NUMBER
Contractor Report ARLCD-CR-80025 ✓

2. GOVT ACCESSION NO.
AD-A090 674

3. RECIPIENT'S CATALOG NUMBER

4. TITLE (and Subtitle)
RECOMMENDED HAZARD CLASSIFICATION PROCEDURES
FOR IN-PROCESS PROPELLANT AND EXPLOSIVE MATERIAL

5. TYPE OF REPORT & PERIOD COVERED
Final

6. PERFORMING ORG. REPORT NUMBER
J6432 ✓

7. AUTHOR(s)
Hyla S. Napadensky & R. Pape, IIT Research Institute
Richard Rindner, Project Leader, ARRADCOM
Dan Satriana, Project Engineer, ARRADCOM

8. CONTRACT OR GRANT NUMBER(s)
DAAK10-78-C-0029 ✓

9. PERFORMING ORGANIZATION NAME AND ADDRESS
IIT Research Institute ✓
10 West 35th Street
Chicago, IL 60616

10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS
MMT-5784289

11. CONTROLLING OFFICE NAME AND ADDRESS
ARRADCOM, TSD
STINFO (DRDAR-TSS)
Dover, NJ 07801

12. REPORT DATE
September 1980

13. NUMBER OF PAGES

14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)
ARRADCOM, LCWSL
Energetics Systems Process Division (DRDAR-LCM-SP)
Dover, NJ 07801

15. SECURITY CLASS. (of this report)
Unclassified

15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

Approved for public release; distribution unlimited.

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES

This project was accomplished as part of the U.S. Army's Manufacturing Methods and Technology Program. The primary objective of this program is to develop, on a timely basis, manufacturing processes, techniques, and equipment for use in production of Army materiel.

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

Hazard classification procedures
Process chemicals
Explosives
Sensitivity
Airblast
Fire
MMT-protective technology

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

A hazard classification procedure is described. The procedure is designed to characterize the potential hazards (fire and explosion effects) imposed by in-process chemical mixtures which exist in propellant and explosive manufacturing operations. Classification testing is divided into a sensitivity evaluation and an effects evaluation. Classifications which are assigned are nearly identical to the NATO/UN classes for storage and transport of explosive materials.

DD FORM 1 JAN 73 1473 EDITION OF 1 NOV 65 IS OBSOLETE

UNCLASSIFIED
SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

CONTENTS

FIGURES

TABLES

INTRODUCTION AND BACKGROUND

This document describes a procedure for characterizing the potential hazards imposed by chemical mixtures which exist in propellant and explosive manufacturing operations. Inprocess materials are classified based on their initiation sensitivity and the most likely consequence of an initiation. Thus classification testing is divided into a sensitivity evaluation and an effects evaluation. The hazard classifications which are assigned are nearly identical to the NATO-UN classes for transport of explosive materials. However, since this procedure is for inprocess materials which exist inside process plants, rather than end items in transport or storage, the NATO-UN classifications have been expanded to better represent process plant hazards.

The hazard classification procedures described in the document are preliminary. Intended users and regulatory agencies should critically review this document before the procedure is made a requirement. Modifications indicated by the review should be incorporated, several of the specific test methods should be refined (particularly the friction sensitivity test), and a validation of the overall procedure should be completed by applying it to several process operations with known accident histories.

The Classifications

Materials evaluated using this procedure will be assigned one of the following classifications based on the test results:

Class	Description
1.1A	MASS EXPLOSION HAZARD. An ignition will result in an explosion involving virtually the entire contents of the process vessel practically instantaneously. Damage will occur due to airblast, fragments, and the fireball.
1.1B	CLOUD EXPLOSION HAZARD. An ignition will result in a dust suspension or vapor cloud explosion inside the process vessel producing substantial, quickly increasing pressures.
1.2	PROJECTION HAZARD. Materials which have a projection hazard but not a mass explosion hazard. Any nearly completed items whose final form is classed 1.2 for transport and storage will automatically be classified 1.2. This hazard category is otherwise not applicable for inprocess materials.
1.3A	MASS FIRE HAZARD. An ignition will result in a significant fire. Damage will occur due to radiant heating from the flame, radiant heating from hot combustion products beneath the ceiling (if indoors), flame impingement, and firebrands.
1.3B	FIRESPREAD HAZARD. An ignition will result in a fire which spreads quickly over a layer of the material in a conveyor or trough configuration.
1.4	MINOR HAZARD. The material does not present a significant hazard. The effects are largely confined to the individual process container.
1.5	VERY INSENSITIVE. It is extremely unlikely that an initiation will occur in the material.

What Materials Must Be Tested

This procedure is designed to characterize the major hazards which exist in each area of a process operation. Since inprocess materials continually change as they pass through the process steps, it would be impractical to classify every possible chemical/physical composition* which exists in a process plant. Therefore, the user must select "typical chemical/physical compositions" which are representative of each process area.** When selecting the sample materials to be tested, the following should be considered:

1. Since the selected materials are to represent the entire process area, each material which is chosen should be similar in composition to a substantial quantity of material in the area. Material forms which exist only in small quantities in the area will not be evaluated unless they are suspected of being a particular hazard.
2. The selected sample materials should in total represent nearly all of the potentially hazardous process material in the area.
3. Sample materials can be extracted from process vessels or synthesized in a laboratory. Extraction from process vessels is preferred because trace materials may build up in a real system and could influence sensitivity. Accessibility and safety considerations are likely to dictate where a material can be extracted and thus its composition. In cases where it is not practical to extract a sample from the actual process, synthesized samples are acceptable.

When Should This Procedure Be Applied

This procedure should be applied to all new process operations as early in the development cycle as it is possible to specify the inprocess materials' chemical/physical composition, approximate material quantities, and approximate component geometries. If possible, this should be accomplished during or before the operation of a pilot plant.

---

*Chemical/physical composition. The process material being evaluated must have the same chemical composition as the actual process material (including any additions such as water or solvent). The physical characteristics such as particle size distribution, bulk density, and temperature must also be representative of the actual system.

**Process area. A distinct area in a process plant. The area boundaries are generally walls setting it apart from other areas. A process area is an enclosure containing process vessels and equipment items or a group of process vessels and equipment items located in the plant as a cluster set apart from the other items.

The sensitivity evaluation can be completed at the laboratory scale phase of process development, whereas the effects evaluation requires larger quantities of sample material and generally must be accomplished at a later stage of development.

Cautions in Using This Procedure

The following basic assumptions and limitations of this procedure should be considered in its application.

1. This procedure is for characterizing the hazards imposed by inprocess propellant and explosive materials. Use beyond this domain is possible but should be done with scrutiny.
2. Toxic, biological, radioactive and corrosive hazards are not considered.
3. Inprocess materials change continuously as they pass through a process. Therefore, only selected representative compositions are evaluated. It is possible that the material sensitivity and/or hazardous effects could intensify at a composition not evaluated. This should not be a problem if the materials to be evaluated are selected judiciously.
4. The sensitivity evaluation is structured around the process operations listed in table 1 of the following section. If a process operation to be evaluated is not listed in table 1 the user may have to select a similar (but not exactly the same) process operation which is listed in table 1 and conduct the sensitivity tests for that process operation.
5. The state of the knowledge of process plant risk is continually advancing. Therefore this procedure should be reevaluated and updated no less than once every 10 years.

Hazards Classification Procedure

The structure of the hazards classification procedure is presented in figure 1. Instructions for completing each step are provided in this chapter. Begin with Step ① at the left of figure 1.

① Sensitivity Evaluation

The five steps in the sensitivity evaluation are presented in figure 2. These steps are described further below:

What Process Operation is This?

Operations which exist in propellant and explosive manufacturing processes are listed in table 1. The evaluator must identify the process operation from which the material being evaluated comes.

Table 1

PROCESS OPERATIONS

- Chutes
- Belt conveyors
- Screw conveyors
- Bucket conveyors
- Pneumatic conveyors, jet mill, air mixer, cyclone, dust collector, etc
- Hoppers (storage, weigh, feed, etc)
- Tote bins
- Screening, sieving, sifting
- Pressing, cartridging
- Extrusion, rolling
- Mills (hammer, prebreaker, crusher, grinder, etc)
- Glazing, coating, and batch drum operations
- Dryer, dry house, oven
- Melt pour, coating
- Reactors
- Wash, mix, and hold tanks (in chemical process plant)
- Gravity spearator
- Centrifugal separator
- Product pump
- Filter
- Flaker drum, belt flaker
- Distillations, solvent recovery
- Mix-melt mix operations (filling operations)
- Packaging, filling, assembly, loading, unloading, transfer (dry material)
- Machining

SAFETY FACTOR

$$S.F. = \frac{\text{IGNITION ENERGY}}{\text{INPROCESS POTENTIAL}}$$

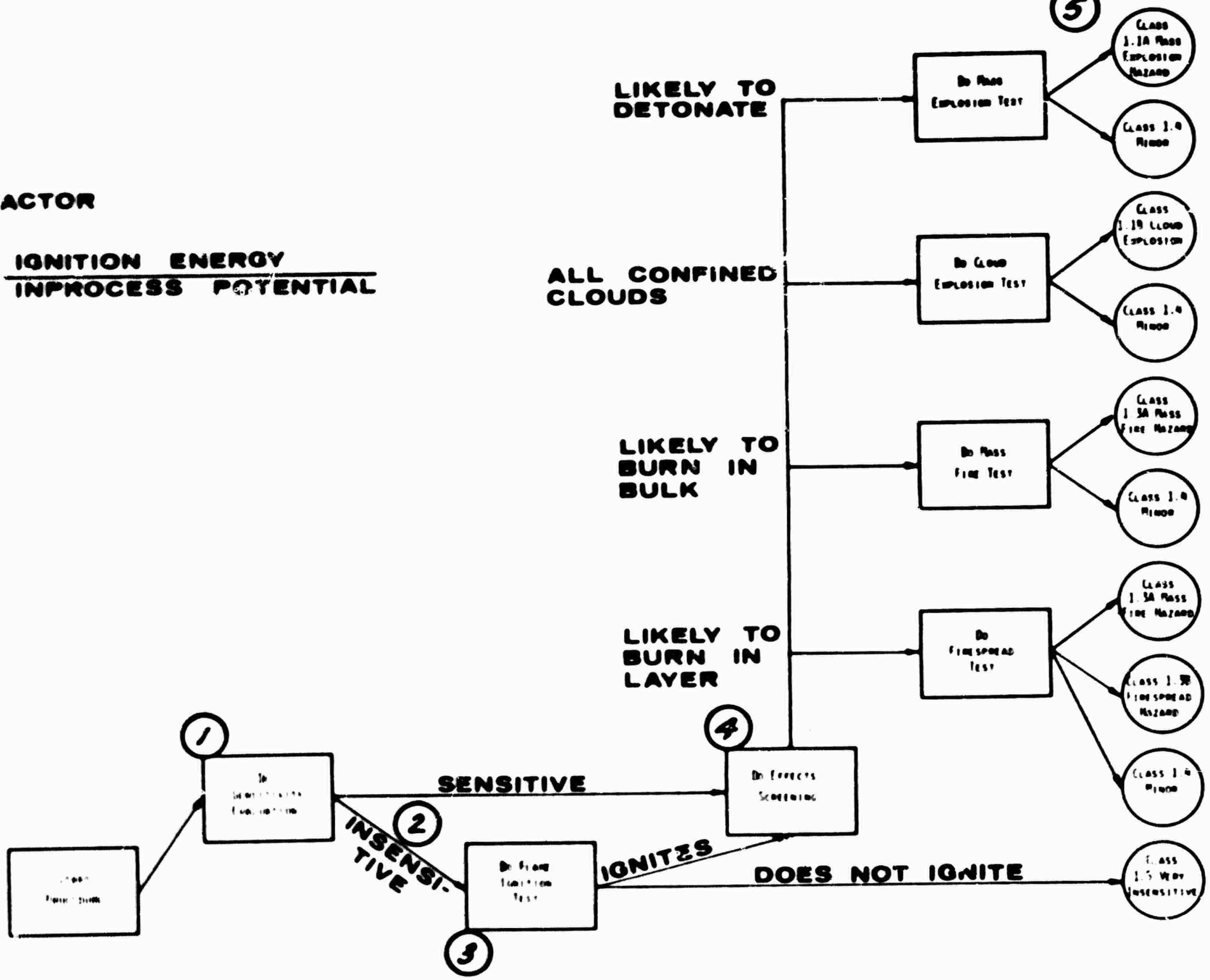


Fig 1 Overall hazards classification procedure

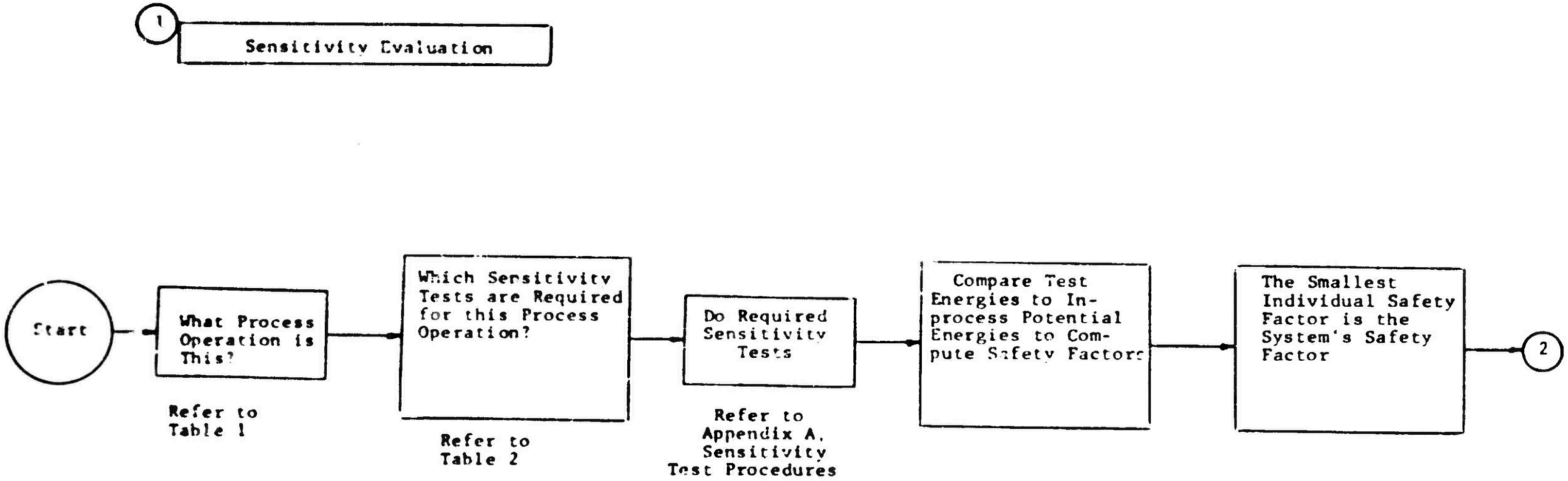


Fig 2 Sensitivity evaluation

If the operation is not listed in table 1, an operation which is very similar can be selected from the table. If no comparable operation is listed in the table, the evaluator should proceed to step ②. At step ② the material will be categorized as sensitive, and the evaluation will immediately proceed to step ④. In general, the proper process operation can be identified in table 1 and the user will merely continue with the sensitivity evaluation as described in the following paragraphs.

Which Sensitivity Tests are Required for This Process Operation?

To determine which sensitivity tests must be accomplished for the sample material, refer to table 2. In table 2, the process operations are listed down the first column on the left side of the page and the sensitivity tests required are listed along the top of the page. Identify the process operation in the table. In the column below the process operation, X-marks indicate which sensitivity tests must be completed.

Do the Required Sensitivity Tests

Each sensitivity test indicated with an X to the right of the appropriate process operation must be completed. A detailed test description and procedure is given in Attachment a for each sensitivity test. The sensitivity tests are designed to experimentally determine the energy (or energy related parameter such as temperature or power) at which there is a 50 percent probability of initiation by the stimulus being considered. This will be called the "test energy". To characterize the likelihood of ignition by a stimulus, the "test energy" is compared to a high energy level which credibly could exist in the process operation by this stimulus. This will be called the "process energy". With each sensitivity test description in Attachment a, there is a list giving the "process energies" for each type of process operation. For each required sensitivity test, the evaluator must experimentally determine the test energy and note the process energy given in the test procedure for the appropriate process operation.

Compare Test ignition Energies to Inproces Potential Energies and Compute Safety Factors

For each sensitivity test conducted, compute the stimulus safety factor. In each case, the safety factor (SF) is merely the test energy divided by the process energy.

$$SF = \frac{\text{"test energy"}}{\text{"process energy"}}$$

Table 2

Sensitivity tests required for each inprocess operation/component type

Process operation/ component type	Test required					
					Thermal	
	Localized impact	Impingement	Rubbing friction	ESD Evaluation	Regional	Local hot spot
Chutes	X	X	X	X		X
Belt conveyors	X	X	X	X	X	X
Screw conveyors	X	X	X	X	X	X
Bucket conveyors	X	X	X	X	X	X
Pneumatic conveyor, jet mill, air mixer, cyclone dust collector, etc	X	X	X	X		X
Hoppers (storage, weigh, feed, etc)	X	X	X	X		X
Tote bins	X	X	X	X		X
Screening, sieving, sifting	X	X	X	X	X	
Pressing, cartridging	X	X	X	X	X	X
Extrusion, rolling**	X		X	X	X	X
Mills (hammer, prebreaker, crusher, grinder, etc)	X	X	X		X	X
Glazing, coating and batch drum operations	X	X	X	X	X	X
Dryer, dry house, oven	X	X	X	X	X	X
Melt pour, casting	X		X	X	X	X
Reactors*	X		X	X	*	
Wash, mix and hold tanks	X		X	X	X	
Gravity separator	X		X	X	X	
Centrifugal separator	X		X	X	X	
Product pump	X	X	X		X	
Filter	X		X	X	X	X
Flaker drum, belt flaker	X	X	X	X	X	X
Distillations, solvent recovery	X		X	X	X	X
Mix-melt mix operations (filling operations)	X		X	X	X	X
Packaging, filling, loading, unloading, transfer (dry material)	X	X	X	X	X	X
Machining	X		X	X	X	X

---

[*] For reactors the system design must provide a generous safety factor and automatic sensors/controls to assure safe operation.

[**] The extrusion process involves shear loading not directly evaluated by this procedure. This operation is somewhat hazardous by its nature, and the system designer is responsible to assure that generous safety margins and automatic sensors/controllers are incorporated to assure safe operation.

What is the Smallest Individual Safety Factor?
This is the System's Safety Factor

A safety factor has now been computed for each sensitivity test (stimulus type) which has been conducted. A low safety factor indicates that the sample material is sensitive to the stimulus. The lowest safety factor computed represents the most likely ignition stimulus and is taken as the system's safety factor. The stimulus type(s) dominating the systems sensitivity should be kept in mind while handling the material in further tests and in the process plant.

② Specify Sensitivity Hazards Category Based on the Systems Safety Factor

Refer to figure 3 for specifying the Sensitivity Hazards Category. If the system's safety factor is less than or equal to 3 the sample material is assigned the SENSITIVE category. If the safety factor is greater than 3, the material is classified as INSENSITIVE. For materials classified as INSENSITIVE, one more sensitivity test is required. The flame ignition test ③ must be completed for INSENSITIVE materials. In all other cases, go to step ④ and begin the Effects Evaluation.

③ Do Flame Ignition Test

The flame ignition test is described in Attachment A and the logic diagram for the flame ignition test is given in figure 4. In the test, the material is exposed to an open flame. If ignition occurs, the material retains its INSENSITIVE rating, and the effects evaluation is required (go to step ④). If the material cannot be ignited by an open flame, it is immediately put into class 1.5, VERY INSENSITIVE, and the classification procedure is completed for that material.

④ Do Effects Evaluation

The effects evaluation is outlined in figure 5.

Cloud Configurations

If the sample exists in a suspended dust or vapor cloud inside the actual process vessel, immediately do the cloud explosion test. Based on the test results, the material will be placed in either class 1.1B cloud explosion hazard, or class 1.4, minor hazard.

Layer Configurations

If the material exists in a layer in the actual process operation, the "critical layer thickness" test should be done first. If the actual layer depth is less than the critical layer thickness, the firespread test should be done. If the actual layer depth is greater than or equal to the critical layer thickness, the "layer transition" test should be done next. When the layer transition test is done, if the critical

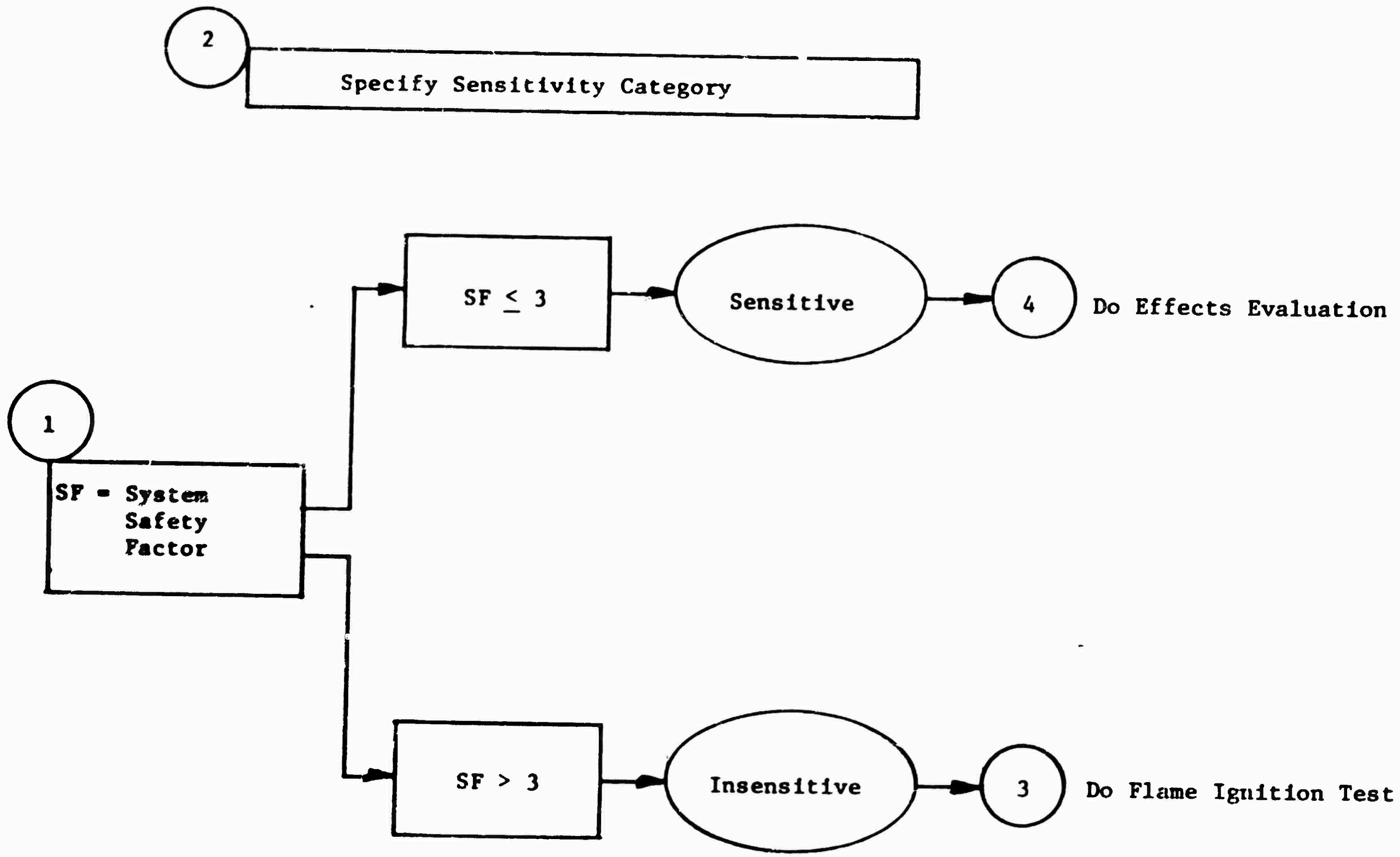


Fig 3 Specification of sensitivity category

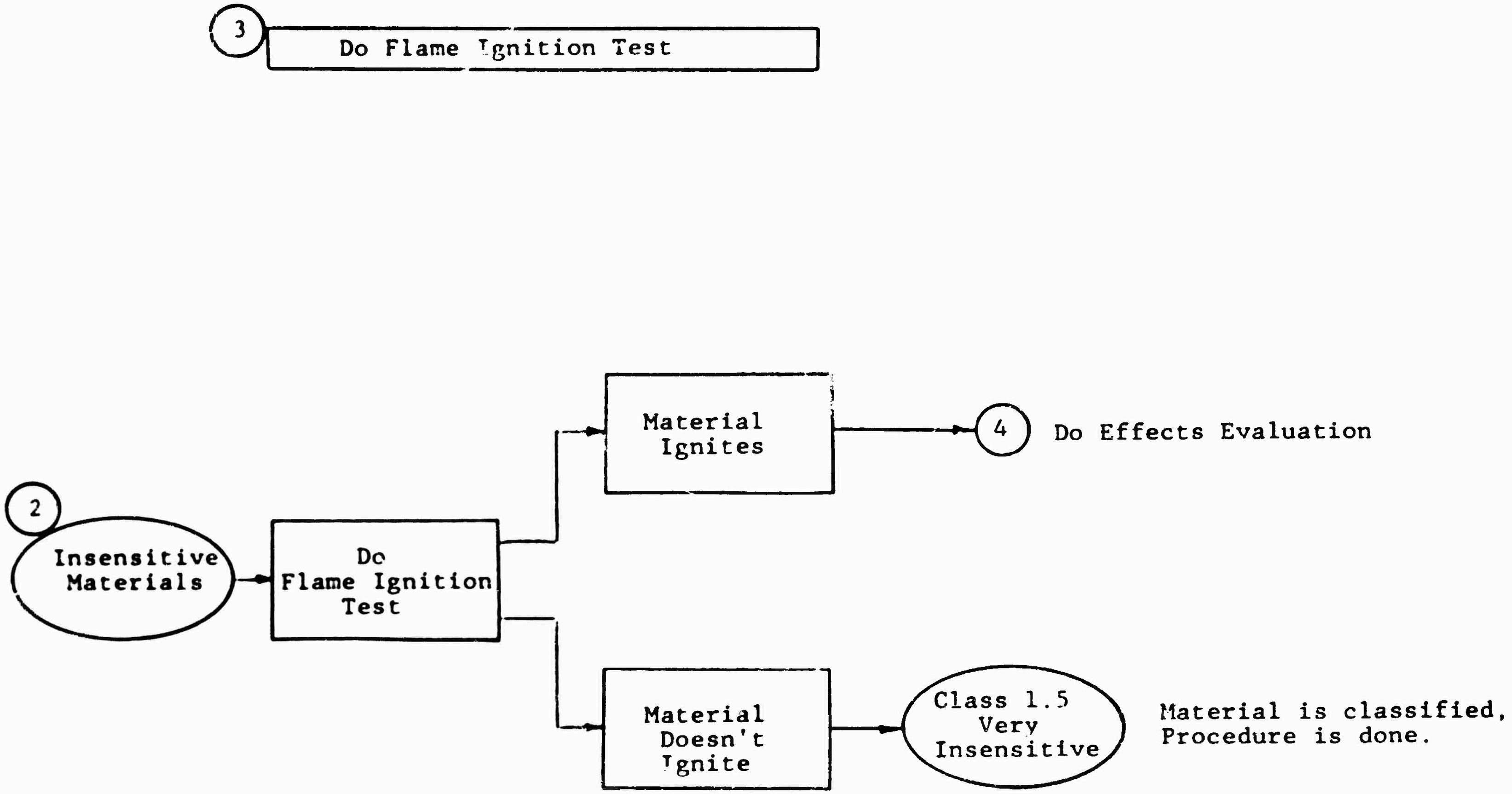


Fig 4 Flame ignition test flow diagram

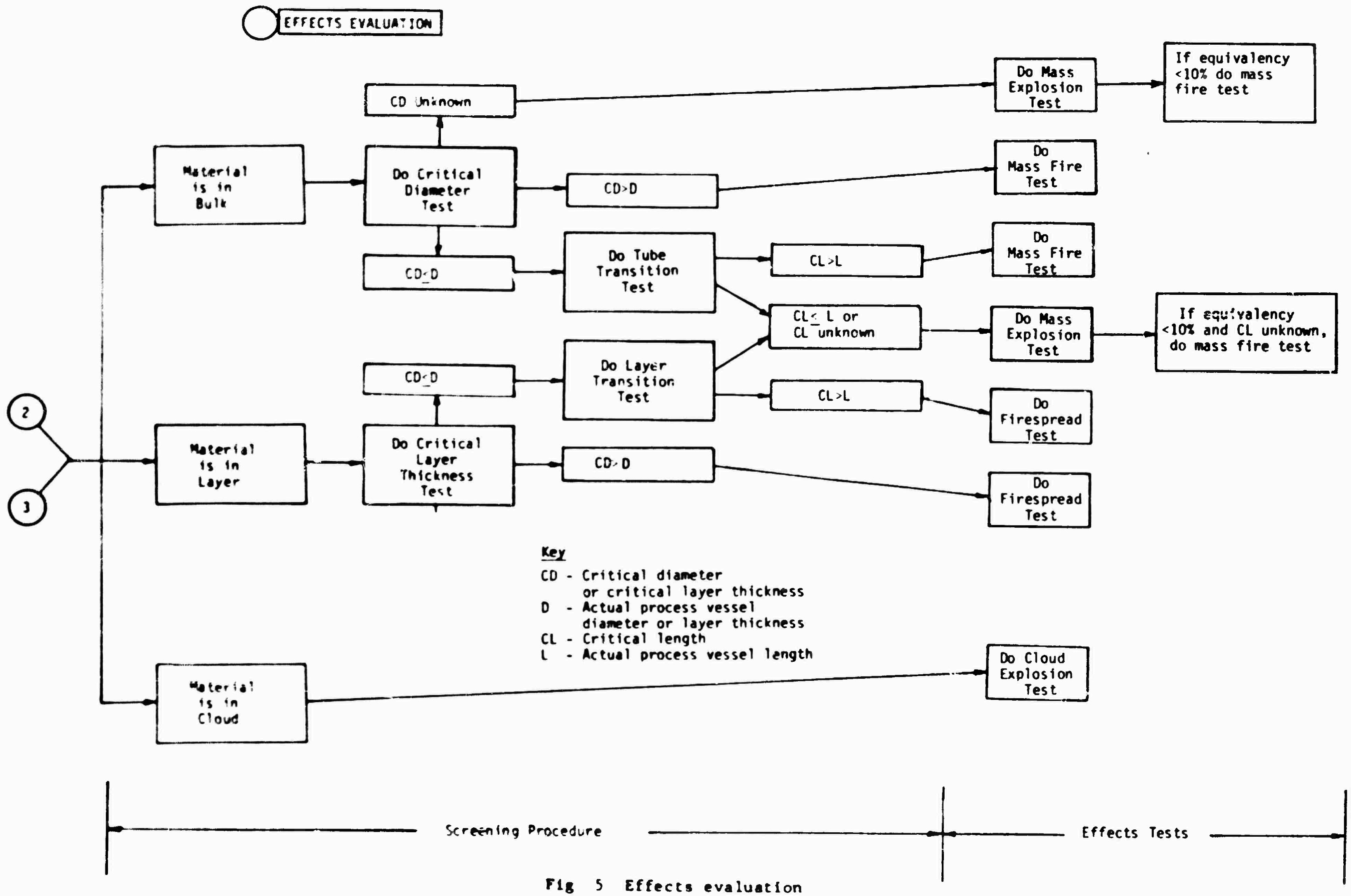


Fig 5 Effects evaluation

length is greater than the actual process layer length, the fire spread test should be done. Otherwise the mass explosion test will be required. The material is classified as described in the effects test procedures.

Bulk Configurations

If the material exists in a bulk configuration, rather than a layer or cloud, the critical diameter test should be done first. If the actual process vessel diameter is less than the critical diameter, the mass fire test should be done. If the critical diameter is too big to be determined in a practical manner at the available test facilities, the mass explosion test should be done. If the critical diameter is less than or equal to the actual process vessel diameter, the tube transition test should be done next. If the critical length (determined in the tube transition test) is greater than the process vessel length, the mass fire test should be done; otherwise the mass explosion test will be required. The material is classified based on the criteria described in the different effects test procedures.

TEST PROCEDURES

LOCAL IMPACT TEST

Purpose

The local impact test is to determine the impact energy per unit area required to initiate the process material being evaluated. This test represents stimuli such as from a dropped tool, a person hammering, a dropped cover, agitator impacts, part failure during operation, a person chipping off residue, etc.

Apparatus

Most drop weight impact machines can be modified to conduct this test as long as the actual energy at impact (based on drop weight mass and velocity) is at least 35 joules. No intermediate weight should be used and the ratio of striker pin mass to drop weight mass should be less than 0.05. The drop weight is to impact directly onto the sample holder striker pin. The two sample holders required for this test are shown in figure a-1. The type 1 sample holder is to be used for homogeneous samples such as fine powders, slurries, pastes, liquids, etc, whereas the type 2 sample holder is to be used for samples with large basic units such as pellets or strands. The sample holder must sit firmly on a massive anvil. An optical (cut light beam) velocity sensor must be set up near the bottom of the drop weight descent, just above the sample.

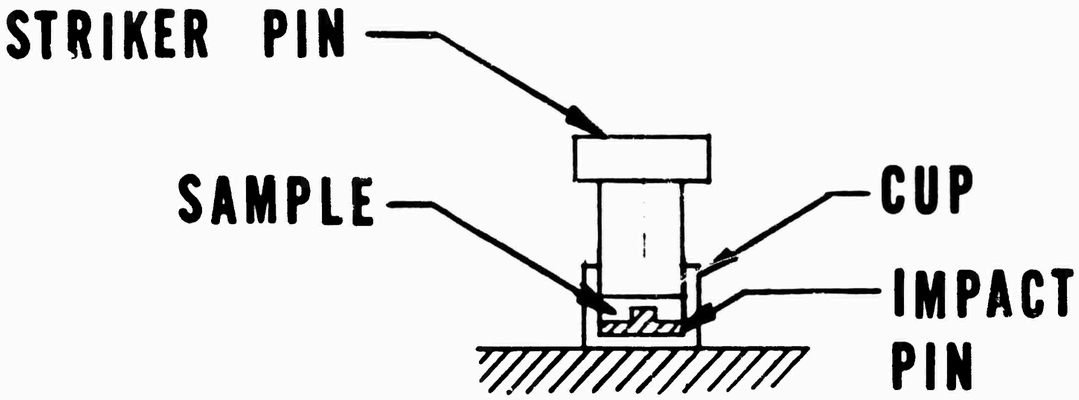
Test Procedure

1. The sample should be placed in the sample holder in the same form as it exists in the actual process. If the type 1 sample holder is to be used (homogeneous sample material forms), the sample's bulk density* must be the same as it exists in the actual process. If the bulk density in the process varies significantly, the maximum anticipated bulk density should be used. If the type 2 sample holder is to be used (nonhomogeneous material forms) the individual sample unit should be tested in all of the possible orientations. For example, pellets should be tested both on their side and standing on one end. The sample in each orientation should be considered as individual material.

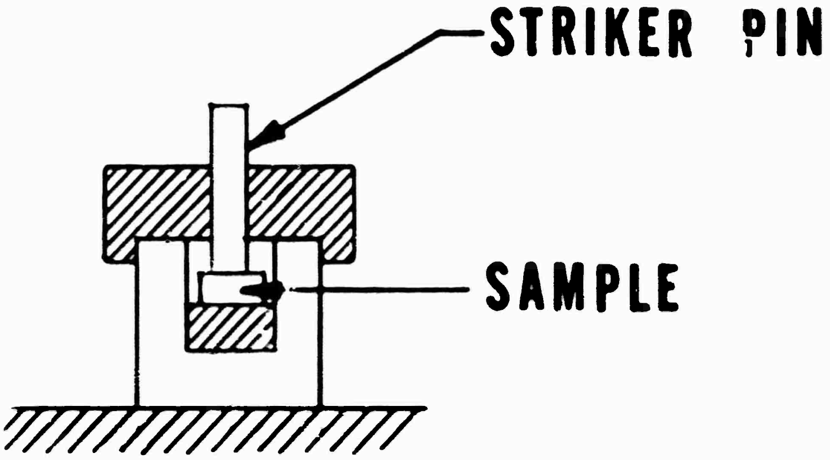
2. For each individual test (each individual weight drop) a fresh sample and clean sample holder must be used. Type 1 sample holders are expendible and should never be reused.

---

* Bulk density is the average density of the material in its process vessel not the individual unit (e.g., pellet, or grain) density.



Type 1 Sample holder



Type 2 Sample ho

Fig a-1 Sample holders

3. For each material being tested and each sample orientation (if type 2 holder is being used), a Bruceton series of tests should be conducted to determine the 50 percent probability of initiation point. The first test should be at the maximum drop height. If no reaction occurs at that height, the test should be repeated. If no reaction occurs at the maximum drop height in three consecutive trials, the testing of the sample is completed. The 50 percent ignition energy per unit area is said to be greater than the maximum energy per unit area that the machine can produce.

If a reaction occurs at the maximum drop height, the drop height should be decreased such that the impact energy (based on velocity at impact) will be equal to the previous value divided by $\sqrt{2}$. Decreasing the energy in this manner should continue until a negative result ("No Go") is obtained. At that point, the Bruceton series is entered. Each time a positive reaction is obtained, go to the next lower energy level (decrease energy by a factor of $\sqrt{2}$), and each time a negative reaction is obtained, go to the next higher energy level (increase energy by a factor of $\sqrt{2}$). A positive reaction is one in which initiation occurs and the reaction propagates to the circumference of the sample holder at at least one point. A sample data sheet is shown in figure a-2 partly filled in. Energy and drop height values are left off, because these depend on the particular apparatus being used. For tests using the type 2 sample holder, for each negative reaction inspect the samples for indentations and the striker surface for marks to estimate the approximate impact area. Record the estimated values on the data sheet. A hypothetical test sequence is shown filled in on the graph near the bottom of the data sheet. Test 5 is the first test that is considered to be part of the Bruceton series. To compute the 50 percent probability of initiation energy, count all the positive and negative reactions in the Bruceton series (tests 5 through 16 in the example). The type of result (go versus no-go) which has less points is used to compute the 50 percent point. In the example, there were seven go's and five no-go's, therefore the no-go's will be used. The no-go's are at three energy levels in our example (a, b and c). The lowest level is given an index i = 0; the next level is given i = 1, and so on. We fill in a table as the one below:

Energy Value	i	n_i	$i \cdot n_i$
c	2	2	4
b	1	2	2
a	0	1	0
Totals	--	N=5	A=6

where n_i is the number of no-go's (in our example) at the ith level and $i \cdot n_i$ is the product of i and n_i. The numbers at the bottom (N and A) are merely the column sums. To compute the 50 percent energy value the following equation is used:

$$\log_{10}(50\%\ \text{point}) = \log(a) + 0.1505\left(\frac{A}{N} \pm \frac{1}{2}\right)$$

1 Sample Holder Type: Type 2

2 Sample Material: Material XX

a Homogeneous Materials (powders/liquids)

o Material form ________

o Layer depth ________

o Sample mass ________

o Sample density ________

b Inhomogeneous Materials (pellets/strands)

o Material form pellet

o Orientation on side

Test Sequence Number	Measured Impact Velocity (m/s)	Impact Energy (joules) ($E = 1/2\ M\ V^2$)	Results (e.g., no reaction, explosion, burn, audible sound, etc)
1			Go - explosion
2			Go - explosion
3			Go - explosion
4			Go - explosion
5			Go - explosion
6			No Go - area < 0.05 in.^{2}
7			Go - explosion
8			No Go - area < 0.05 in.^{2}
9			Go - explosion
10			Go - all material burned
11			No Go - Area < 0.05 in.^{2}
12			Go - all material burned
13			Go - all material burned
14			No Go - area < 0.05 in.^{2}
15			No Go - area < 0.05 in.^{2}
16			Go - all material burned

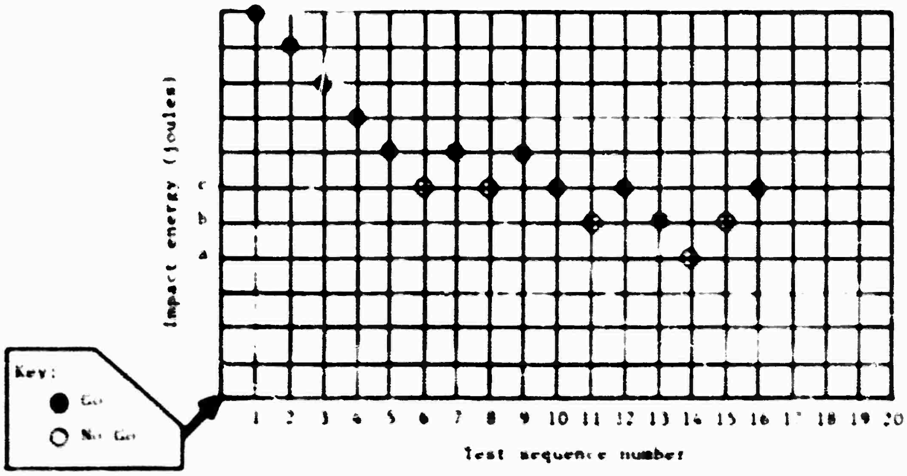


Fig a-[illegible] Local impact test data sheet where the plus sign is used if the calculation is based on no-go's and the minus sign is used if the calculation is based on go's.

Interpretation of Results

The 50 percent energy per unit area obtained from the tests is to be compared to a high credible inprocess stimulus energy to compute a safety factor using the equation:

$$\text{SF (impact)} = \frac{\text{50\% test energy/unit area}}{\text{Inprocess potential energy/unit area}}$$

The 50 percent point computed from the test is the impact energy, not the energy per unit area as of yet. Divide the 50 percent point by the impact area to determine the 50 percent test energy per unit area. The inprocess potential energy depends on the process operation being evaluated. Obtain the proper inprocess potential from the list given below.

Process Operation	Improcess Potential Load Impact Energy (J/m^2)
Belt conveyor, bucket conveyor, screw conveyor, hoppers, tote bins, screening, mills, glazing, coating, batch drum operations, dryers, chutes, gravity separators, filters, flaker drum, belt flakes, mix-melt mix operations	5.3×10^4
Pneumatic systems	2.0×10^6
Pressing, extrusion, rolling	6.76×10^4
Melt pour, casting, reactors, wash, mix, and hold tanks	4.4×10^6
Centrifugal separators	1.0×10^6
Product pumps, valves	7.4×10^6
Distillations, solvent recovery	2.6×10^5
Packaging/filling (dry)	3.6×10^5
Machining	2.1×10^4
Wet scrubber	4.8×10^4

IMPINGEMENT TEST[*]

Purpose

The impingement test will determine whether an inprocess material, moving at some velocity and striking an object, will initiate. This test simulates particle-particle and particle-wall impacts in pneumatic conveyors, jet mills, cyclone separators, particles dropping from a chute onto a surface below (e.g., inside a hopper) etc.

Operating Principle

Propelled

The sample is propelled by air (or any other fluid) at a known velocity and impinged (collided) against a massive target. This test is used to simulate the pneumatic systems outlined above.

Free Fall

The sample is dropped in free fall through a known distance onto an impingement block. This test is used to simulate situations such as particles dropped out of a chute into a hopper.

Test Description

The propelled test, figure a-3, involves injecting a test sample into a high velocity air stream or dropping it in free fall, figure a-4, through a fixed drop height and measuring the test material velocity at which ten (minimum) consecutive trials yield "no initiation" while at least one initiation is obtained at the next higher level. Initiation of the test material is detected by high emulsion speed (ASA 10,000) Polaroid film or visual means. Sample velocity is measured by cinephotography or photoelectric transducers.

Test Analysis and Limitations

Data from this test are reported in terms of impingement velocity (m/s). The velocity is measured on every trial for the propelled liquid impingement test and is calculated from appropriate correlations for the solid-propelled and the free-fall impingement tests. Instrumentation techniques[**] also allow the kinetic energy of impingement to be measured.

---

[*] This test procedure is largely extracted from TB700-2.

[**] American Institute of Physics Handbook, Second Edition, McGraw-Hill Book Company, Inc., Chap 5, pp 10-37.

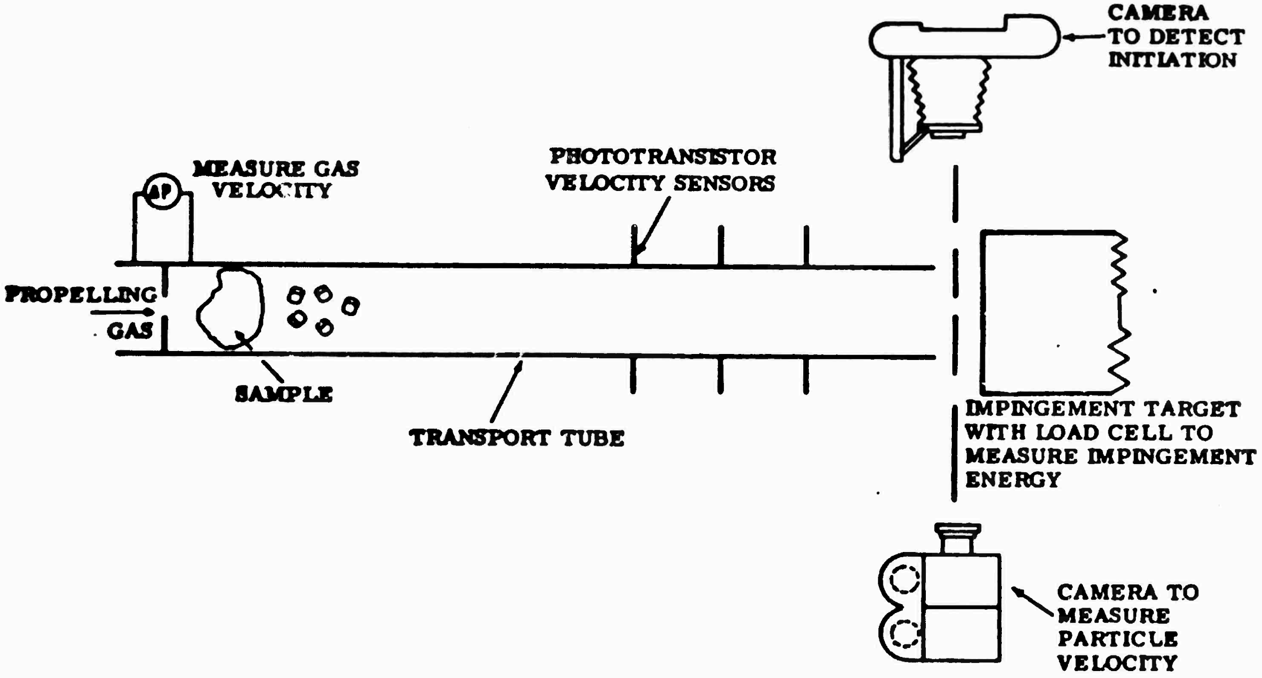


Fig a-3 Liquid and solid explosive impingement test apparatus

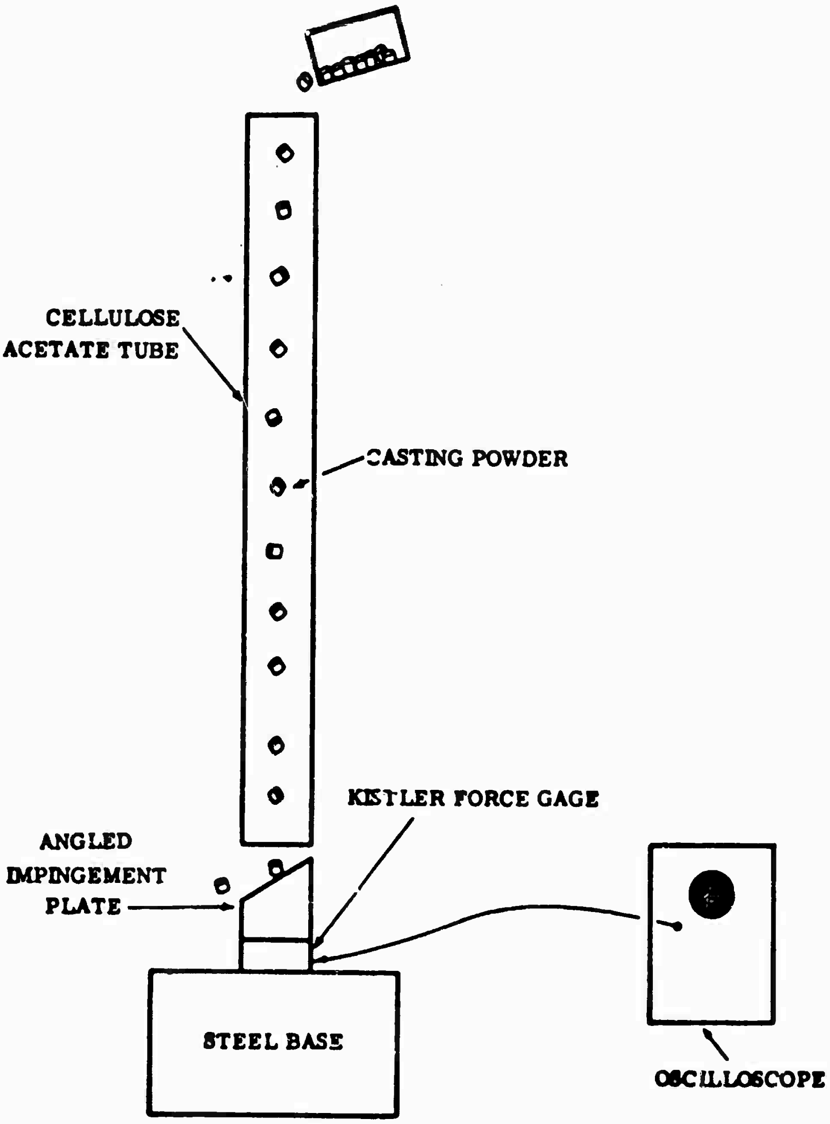


Fig a-4 Experimental arrangement for free-fall impingement test

Where appropriate, this test examines the effects of quantity, materials of construction, surface finish, and surface angle on impingement initiation.

The test is limited by the velocity at which the conveying fluid (generally air) can be carried through the transport tube and by the drag between air and the material being carried. The upper limitation is approximately the sonic velocity in air (approximately 335 m/s).

Interpretation of Results

The safety factor, SF, is computed by dividing the test impingement velocity at which ignition occurred for 50 percent of the trials by the inproces potential for the appropriate process operation obtained from the table below:

Process Operation	Inprocess Potential[*] Impingement Velocity (m/s)
Belt conveyors, bucket conveyors, screw conveyors, hoppers, tote bins, screening, mills, glazing, coating, batch drum operations, chutes, flaker drum, belt flaker, packaging/filling (dry)	10
Pneumatic systems	23
Pressing	5
Product pump and valves	26
Wet scrubber	60

---

[*] In all cases where the operating velocity is well defined and known for the specific process being evaluated, use the actual velocity plus 20 percent of the actual velocity.

RUBBING FRICTION TEST

Purpose

The rubbing friction test will determine the minimum frictional power per unit area required to initiate the process material being evaluated. This test represents (1) friction between two materials of construction in the presence of a fluid process material such as a fine powder, slurry, paste, or liquid; or (2) friction between the process material and a material of construction in cases where the process material is in the form of large chunks, pellets, strands, etc.

Apparatus

A rotary friction machine such as the one described in figure a-5 must be used to accomplish this test. The machine consists of a d.c. electric motor and controller (1/2 horsepower x 0 to 2000 rpm motor minimum capacity), a drill press type head and stand to support the axial load on the main shaft, a torque and rpm sensor, a contact point holder for steel balls for fluid samples and a contact point holder for inhomogeneous samples such as chunks, pellets, and strands, a grooved and an ungrooved contact section with a lever arm system to provide the required axial load, a hydraulic cylinder to support the weight on the lever arm until the test commences, and a light sensor to measure time from friction load application to ignition. Torque and time to ignition (light signal) will be recorded using a strip chart recorder. Rotation speed will be recorded from an appropriate dial or digital meter.

Procedure

1. A sample data sheet for this test is given in figure a-6. Four axial loads must be chosen for the test. This force is applied by adding weights to the right end of the lever arm as shown in figure a-5, however, the applied force recorded on the data sheet should be the force actually supported by the two contact points. The chosen loads will vary with friction machines, but should be on the order of 9 kg (20 lb), 18 kg (40 lb), 27 kg (60 lb), 36 kg (80 lb).

2. The machine must be calibrated so that for a preset applied force and axial speed, the rotation speed during the test can be estimated. The rotation speed will drop when the load is applied. The lowest rotation speed will be about 100 rpm, depending on the apparatus.

3. For inhomogeneous samples (chunks, pellets, strands, etc), the appropriate contact point holder and bottom plate should be used. For these tests the bottom plate is flat (not grooved). For homogeneous or fluid samples (powders, slurries, pastes, liquids) the contact holder with steel balls and grooved bottom plate must be used. For each trial a new sample is to be used and the steel balls (if used) should be

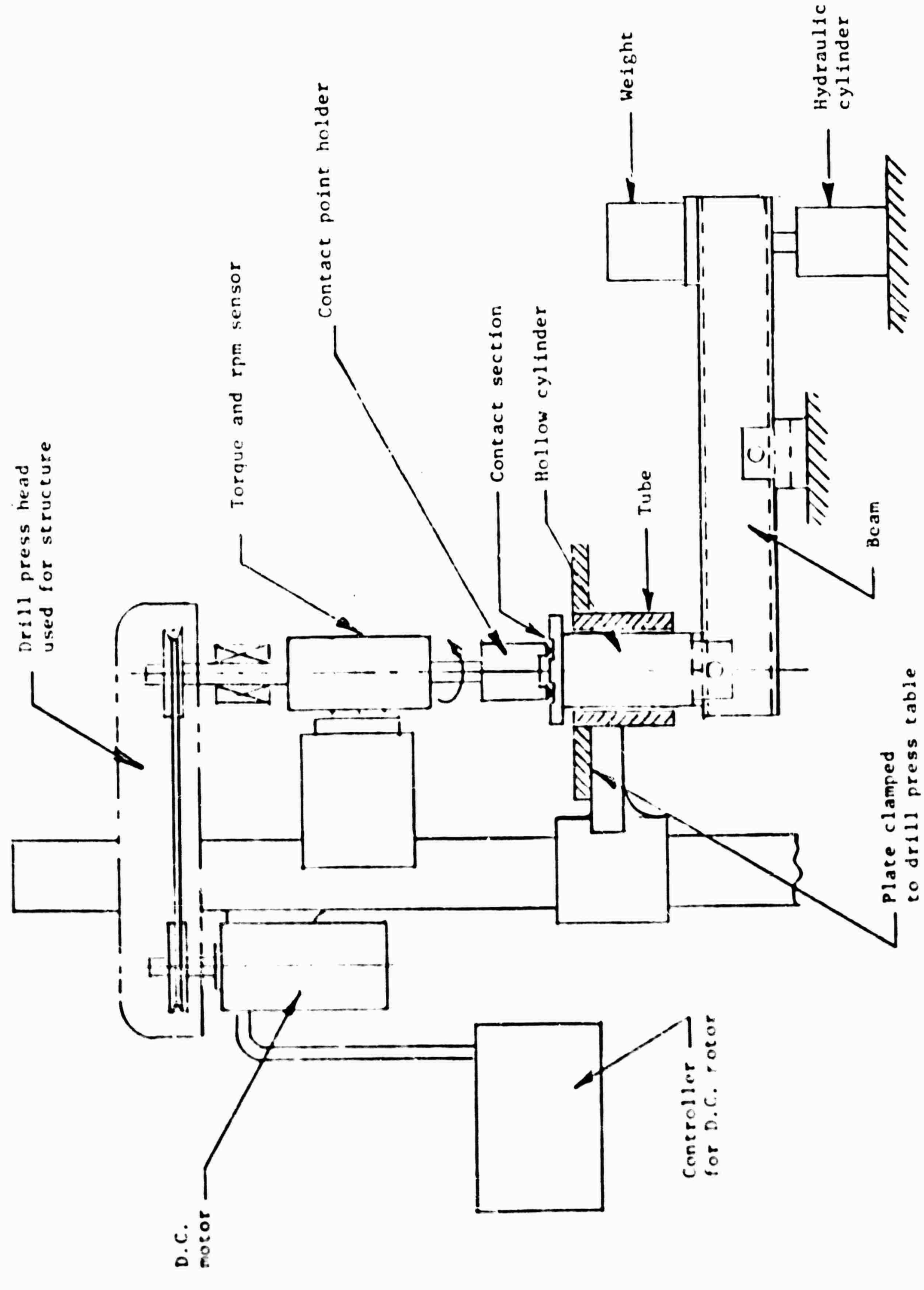


Fig a-5 IITRI rotary friction apparatus

Date ____________________

Sample Material ____________________

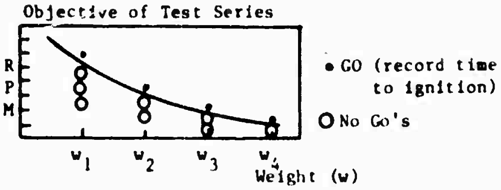
Material Form ____________________

Anvil Groove Depth ____________________

Anvil Groove Width ____________________

Conditioning of Anvil ____________________

Objective of Test Series



Test No.	Preset Conditions		Test Results (Measured Values)			
	Weight Applied	Preset RPM	RPM	Torque	Time to Ignition	Remarks
	w_4 =					
	w_3 =					
	w_2 =					
	w_1 =					

Notes:

1. Work from heavy weight series (w_4) to lightweight series (w_1).
2. Always begin next lower weight series at the highest RPM at which a "No Go" was previously observed.
3. Always do several ([illegible] or 3) higher RPM levels once a "go" is achieved in order to get an RPM versus time to ignition curve.

Fig a-6 Rubbing friction test data sheet rotated or replaced so that an unused location makes contact. The anvil surface should be cleaned reasonably well between tests using number 200 energy paper. The surface should be refinished or replaced whenever excessive wear occurs. For homogeneous samples, the sample is loaded into the groove flush with the top of the groove.

4. Each trial is conducted for 60 seconds. If initiation does not occur, move to the next trial. The first trial is at the mimimum rpm and maximum axial load to be used. Complete the entire series required using the first axial load before moving on to the next load. With the first load, increase rpm each trial in increments of 100 rpm until initiation occurs, then step back in increments of 20 rpm to more closely bracket the critical value for initiation.

Now go to the next lower axial load. Begin at the rpm level tried just prior to initiation in the previous series. Continue exactly as before to determine the critical rpm for each axial load.

Inhomogeneous samples such as pellets, strands, or chunks of material will generally not be initiated even under the maximum load and rpm conditions. These samples generally will fail structurally and deteriorate. Save the deteriorated sample material (shavings) from these tests. If no ignitions are obtained, retest the shavings using the steel balls contacting the ground anvil to determine the material sensitivity to friction.

Interpretation of Results

For each trial, the operator must record (1) the axial load, (2) the average rpm during the test, (3) the average torque during the test, and (4) time to ignition. Using (1), (2) and (3) power per unit area can be calculated from the following equation:

$$\frac{P}{A_c} = \frac{fT}{a^2} \text{ with } \begin{cases} a = \text{the sample contact radius for inhomogeneous samples (e.g., pellet cross-sectional area)} \\ a = 0.721 \sqrt[3]{Pd\left[\frac{1-\nu_1^2}{E_1} + \frac{1-\nu_2^2}{E_2}\right]} \quad \text{for the homogeneous samples.} \end{cases}$$

where f is the rotational speed (revolutions per second), T is the measured torque (newton-meters), a is the contact radius, P is the axial load (newtons), d is the ball diameter, ν_1 and ν_2 are Poisson's ratios for the balls and bottom plate respectively, E_1 and E_2 are the moduli of elasticity for the balls and bottom plate respectively.

Compute power per unit area for the critical rpm value obtained for each of the four axial loads. The lowest of the four values is taken as the rubbing friction sensitivity of the test sample. This value is the numerator in the equation giving the rubbing friction safety factor for the material:

$$\text{SF (friction)} = \frac{\text{Power per unit area from tests}}{\text{Inprocess potential}}$$

The inprocess potential is taken from the following list for the appropriate process operation.

Process Operation	Inprocess Potential (watts/m^2)
Belt conveyors, bucket conveyors, screw conveyors, hoppers, tote bins, screening, pressing, extrusion, rolling, mills, glazing, coating, batch drum operations, melt pour, casting, chutes, gravity separators, filters, flaker drum, belt flaker	4.90×10^8
Pneumatic systems	1.10×10^7
Reactors	5.34×10^9
Wash, mix and hold tanks	2.54×10^8
Centrifugal separators	1.00×10^9
Product pumps and valves	1.26×10^9
Distillations, solvent recovery	1.72×10^3
Mix-melt mix operations	6.50×10^8
Packaging/filling (dry)	2.10×10^8
Machining	4.25×10^8
Wet scrubber	6.26×10^8

LOCAL THERMAL TEST

Purpose

Inprocess explosive and propellant materials may be susceptible to initiation from localized hot spots from such sources as friction sparks, welding sparks, hot metal chips from equipment failure, hot solid or liquid particles thrown from an unplanned chemical reaction, a lit cigarette, etc. This test is to determine the sensitivity of a material to initiation by local hot spots. Hot spot temperature is the parameter which defines initiation of the sample.

Apparatus

The overall test arrangement for the local thermal test is illustrated in figure a-7. The furnace is detailed in figure a-8; the heater coil surrounding the ceramic tubes inside the furnace is shown in figure a-9; and the coverplates and heater coil leads (two needed) are detailed in figure a-10. A power supply and variac are needed for the furnace's operation. A chromel-alumel thermocouple is required to measure the temperature inside the furnace. Small metal sample dishes (approximately 2.3 cm diameter x 0.6 cm high) are required to contain the test specimens in position below the furnace. One 1-mm diameter steel ball is required for each trial to be completed. The apparatus must be encased on three sides with 2.54 cm (1 in.) thick plexiglass to protect the operator. The fourth side is left open for inserting the sample during the test.

Procedure

1. For each trial an unused steel ball and sample dish and a fresh sample are to be used. For liquid and powder specimens, the sample is to be placed in a 0.3175 cm (1/8 in.) thick layer in the sample dish. Nonhomogeneous materials such as pellets, strands, chunks, etc. should be cut and piled in the dish to approximate a 0.3175 cm (1/8 in.) thick layer with a minimum of spaces between the individual pieces.

2. A sample data sheet for these tests is shown in figure a-11. Test should be conducted at approximately 100°C intervals between 40°C and 1050°C at 10 trials per temperature level. The highest temperatures should be done first. If no positive reactions are obtained in the 10 trials at a given temperature level, there is no need to do tests at lower temperatures.

3. For each trial, begin by properly loading the sample in the dish. Heat the electric furnace to the desired temperature as indicated by the thermocouple reading. Place the steel ball into the furnace using a tweezers. The furnace must be oriented with the entrance for the steel ball facing upward at this time. Allow at least 2 minutes for the ball to come to the furnace temperature.

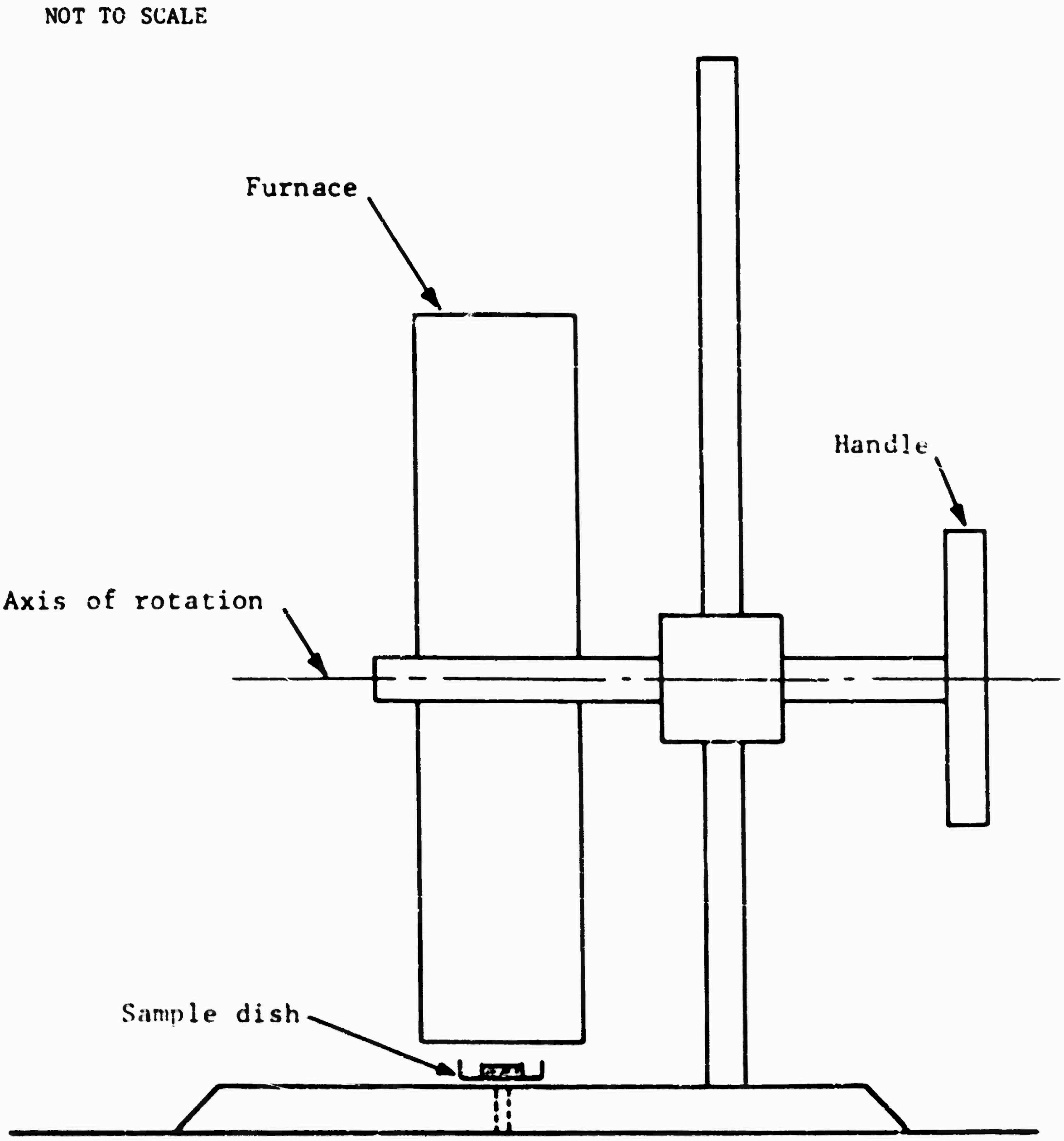


Fig a-7 Setup for hot spot apparatus

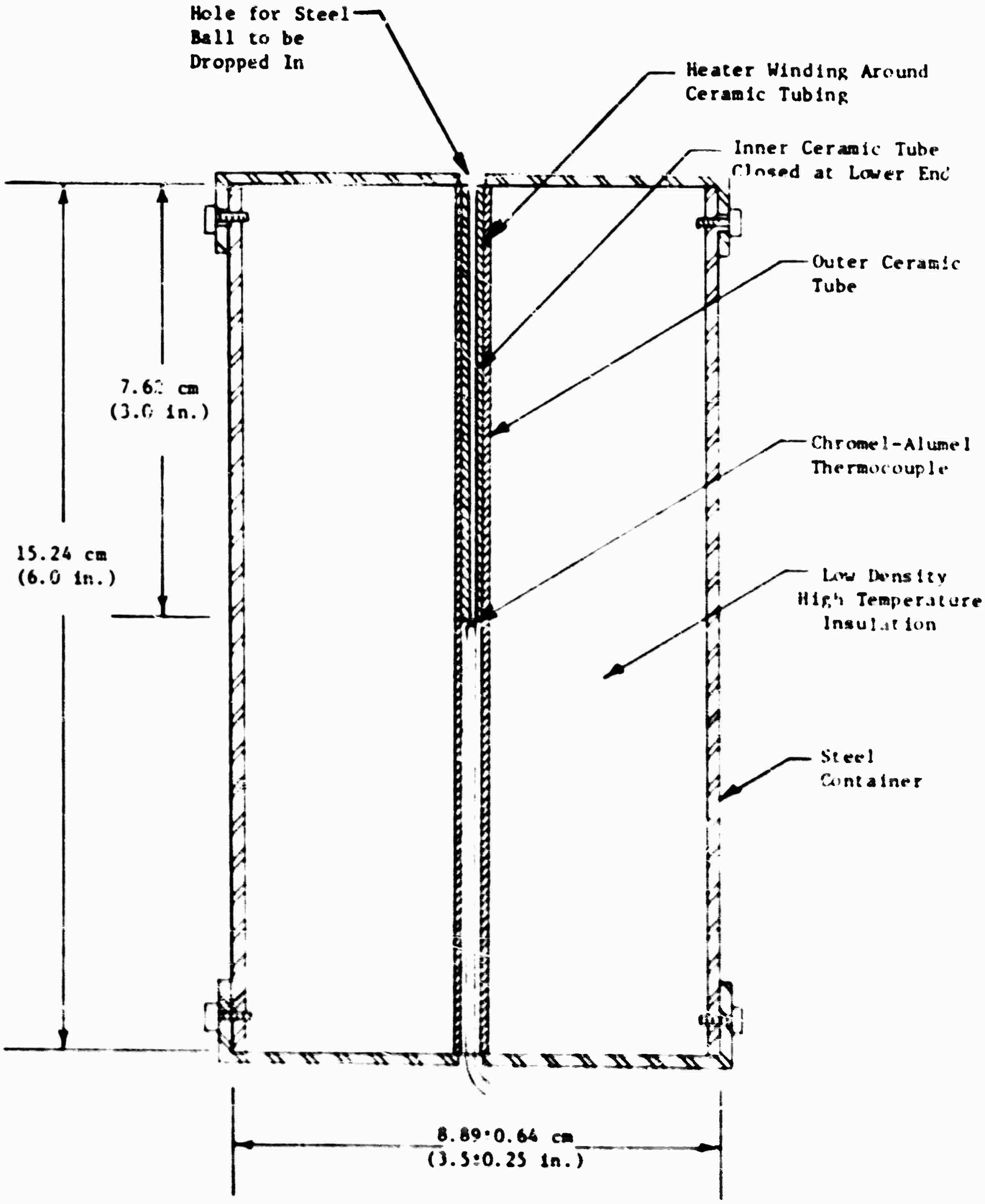


Fig a-8 Furnace assembly

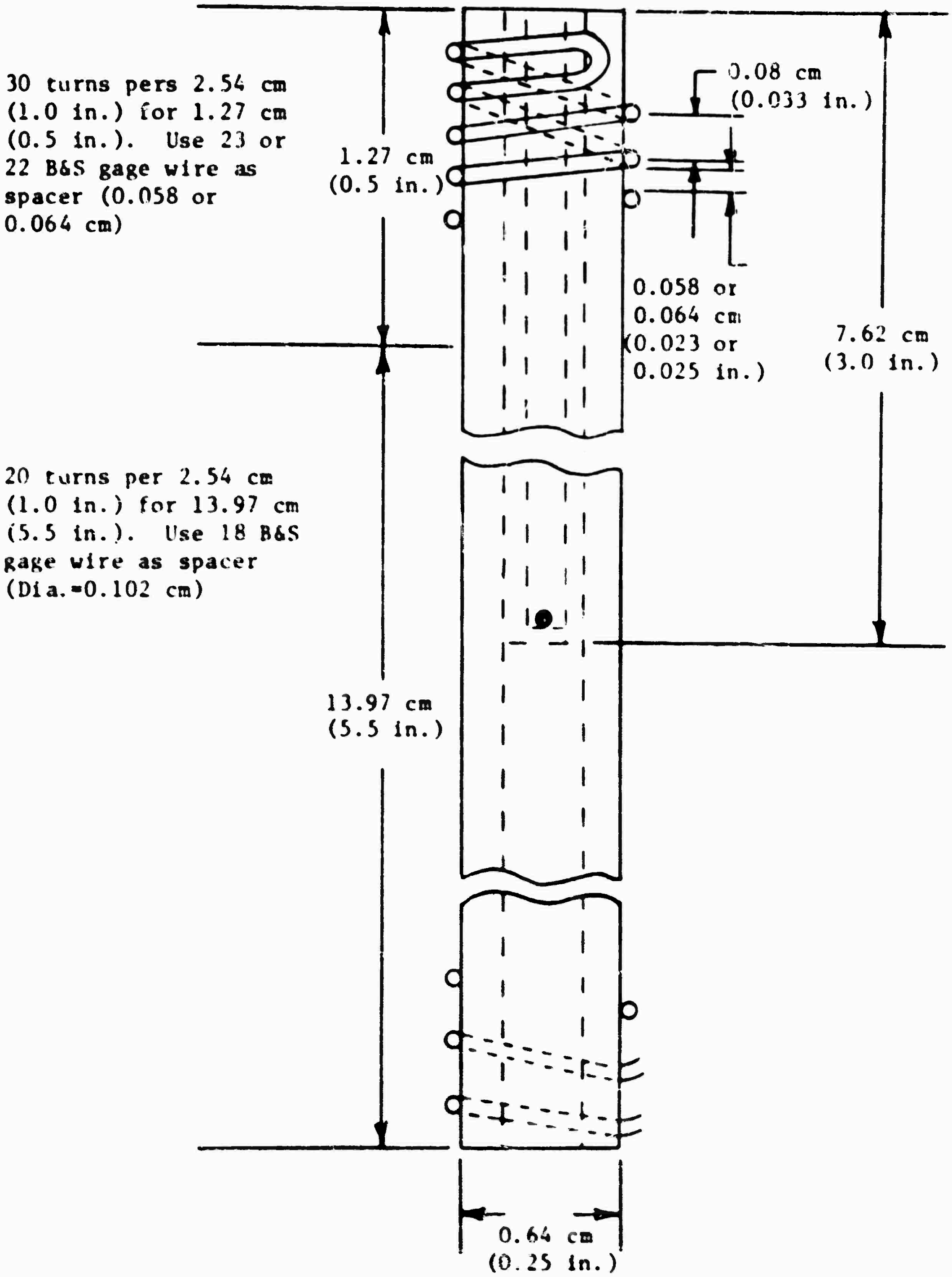
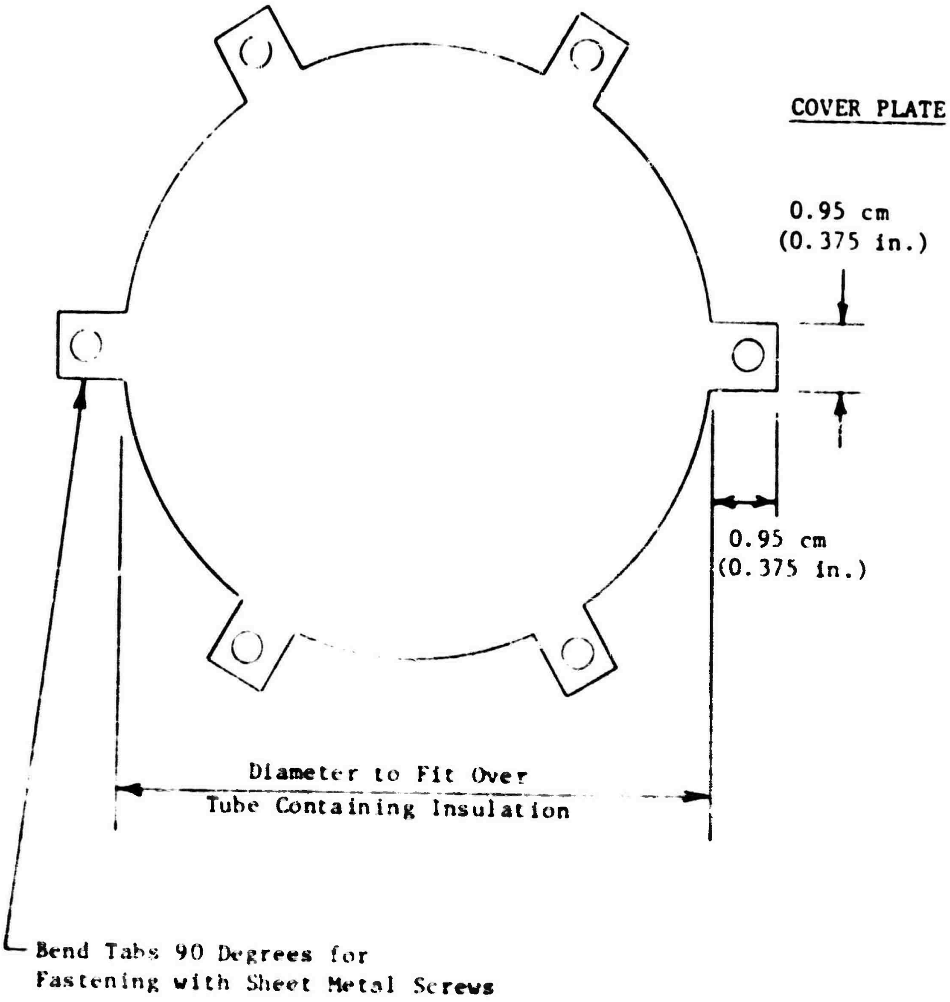


Fig a-9 Heater winding

COVER PLATE



HEATER TERMINAL

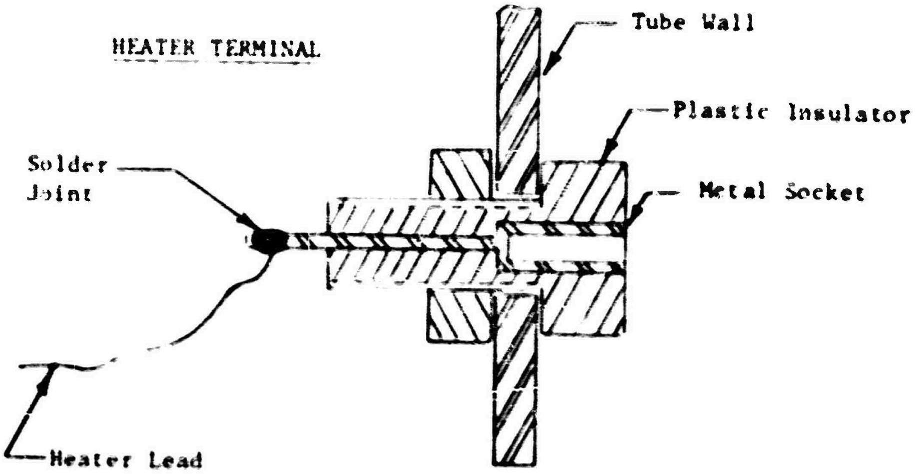


Fig a-10 Cover plates and heater terminals

Material: RDX Slurry
Density: $1.114\ g/cm^3 = 18.26\ g/in^3 = 0.0402\ lb/in^3$
Thickness of Material in Sample Holder: 0.125 in.
Can Diameter 29/32 Weight = $/4(0.906^2)(0.125)(16.387064)(1.114) = 1.471$ gm

Test Number	Explosive Weight	Ball Size	Ball Temperature	Remarks	Decision
41	1.471 gm	3/64	1450°F	Wisp of smoke, ball fuzed into RDX	No Go (smoked)
42	1.471 gm	3/64	1450°F	Same	No Go
43	1.471 gm	3/64	1450°F	Same	No Go
44	1.471 gm	3/64	1450°F	Same	No Go
45	1.471 gm	3/64	1450°F	Same	No Go
46	1.471 gm	1/32	1950°F	Wisp of smoke, ball fuzed into sample material	No Go
47	1.471 gm	1/32	1950°F	Same	No Go
48	1.471 gm	1/32	1950°F	Same	No Go
49	1.471 gm	1/32	1950°F	Same	No Go
50	1.471 gm	1/32	1950°F	Same	No Go

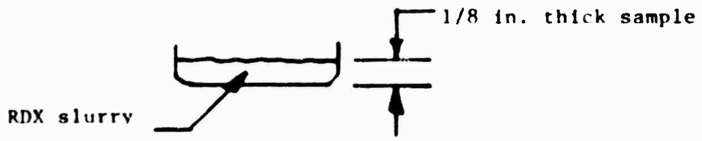


Fig a-11 Local thermal test, sample data sheet

Now place the sample cup beneath the furnace in a predetermined position such that the steel ball will hit the sample when the furnace is inverted. Finally, quickly and firmly invert the furnace allowing the steel ball to fall onto the sample. Record the observed reaction on the data sheet. A positive reaction is indicated by the reaction propagating to the boundary of the sample dish, not merely by the occurrence of a minor reaction of any type.

Interpretation of Results

The ignition temperature is the ball temperature required to initiate the sample 50 percent of the trials. This is determined by plotting the fraction of positive reactions per total trials (experimental probability of initiation) as a function of ball temperature. The temperature from the curve at which initiation will occur half the time is the critical temperature. This resultant temperature is the numerator in equation used to derive the local thermal initiation safety factor:

$$\text{SF (local thermal)} = \frac{\text{Sensitivity test initiation temperature}}{\text{Inprocess potential temperature}}$$

The denominator in the safety factor equation is given in the following list for the appropriate process operations.

Process Operations	Inprocess Potential Local Hot Spot Temperature ($^{\circ}$C)
Screw conveyors, mills	200
Screening	177
Product pumps and valves	1071
All other operations	1000

REGIONAL THERMAL TEST

Purpose

This test is to determine the lowest temperature above ambient at which a major exothermic reaction commences when the process material is uniformly heated over its volume. This temperature will be called the autoignition temperature of the material. In a process plant a material can be exposed to an elevated temperature over a substantial volume when there is a loss of cooling or agitation in a reactor, when by operator error or component failures operation is allowed to go above the design temperature, or when the material is inadvertently spilled or sprayed onto a hot surface such as an overheated pump or motor.

Apparatus and Procedure

Differential thermal analysis (DTA) and differential scanning calorimetry (DSC) are widely used techniques which provide the lowest temperature at which a significant exothermic reaction commences. Either technique is acceptable for providing this information. ASTM has standardized these test methods. The attached ASTM document summarizes the methods and references the related ASTM standards. For hazards classification, a heating rate of 10 °C per minute should be used. The onset of the first significant exotherm should be taken as the autoignition temperature. The sample size should be kept less than 20 mg, and the tests should be done with lids in place on the sample cups in an air atmosphere.

Interpretation of Results

The autoignition temperature obtained from DTA or DSC is to be divided by the inprocess potential temperature level to compute the regional thermal safety factor:

$$\text{SF (regional thermal)} = \frac{\text{Test autoignition temperature}}{\text{Inprocess potential temperature}}$$

The inprocess potential temperature is obtained from the following list for the appropriate process operation.

STANDARD METHOD OF TEST FOR HAZARD DETECTION OF CHEMICALS BY METHODS OF DIFFERENTIAL THERMAL ANALYSIS

DRAFT NO. 4 (REVISED)

INTRODUCTION

This test is one of several being developed by ASTM Committee E-27 for assessing the potential hazards of chemicals. A rough estimate of such hazards may be made by subjecting the material under consideration to the CHETAH Program (ASTM # DS51) whose scope it is to determine the maximum energy of decomposition (ΔH_D) or reaction (ΔH_R) under adiabatic conditions.

For detection of potential hazards of chemicals, Differential Thermal Analysis (DTA) and Differential Scanning Calorimetry (DSC) offer the unique advantage of using very small samples, on the order of a few milligrams. The reader is referred to ASTM # E472 and E474 for general techniques of DTA and DSC.

1. SCOPE

1.1 The purpose of this test method is to approximate the temperature at which potentially hazardous exothermic chemical reactions occur using a minimum quantity of sample.

1.2 The method utilizes techniques of differential thermal analysis and may be used on solids, liquids or slurries. The test method can be carried out under vacuum, at atmospheric or pressure conditions, in an inert or reactive atmosphere and over a temperature range of from - 100°C to well above 1000°C.

2. SUMMARY OF METHOD

2.1 A sample of the material to be examined and an inert reference are each placed in suitable holders and then heated simultaneously.

2.2 In DTA, thermocouples imbeded in both the sample and reference material are connected in series - opposition so as to measure a temperature difference(ΔT). An additional thermocouple is provided to measure the absolute temperature (T) of the sample and/or the reference.

2.3 In DSC a measurement is made of the differential energy (ΔE) required to keep the sample and reference material at the same programmed temperature throughout the analysis.

2.4 The temperature of the sample and reference is increased at a rate of about 10°C to 30°C per minute and a record is made of ΔT or ΔE as a function of T. When an exothermic reaction occurs in the sample a ΔT or ΔE is indicated by a positive departure from the base line of the temperature record.

3. SIGNIFICANCE

3.1 This method is useful in detecting potentially hazardous exothermic reactions. Committee E-27 recommends the use of this method as an early test in assessing the reactive hazards of an uncharacterized chemical substance or mixture.

3.2 The magnitude of the exotherm may not necessarily denote the relative hazard. For example, exothermic reactions are often accompanied by gas evolution which increases the hazard potential. Thus, only the presence or absence of an exotherm and its approximate onset temperature is of significance in this method.

3.3 When vaporizable substances are being studied, it is important to perform the test under a confined condition so that exotherms occuring above normal boiling or sublimation points may be detected.

4. LIMITATIONS

4.1 Although endothermic reactions may be observed with this test, it does not assess their hazard potential.

5. DEFINITIONS

5.1 Differential thermal analysis (DTA): A technique of recording the difference in temperature between a substance and a reference material against either time or temperature as the two specimens are subjected to identical temperature regimes in an environment heated or cooled at a controlled rate (ASTM E-473).

5.2 Differential scanning calorimetry (DSC): A technique of recording the energy necessary to establish zero temperature difference between a substance and a reference material against either time or temperature as the two specimens are subjected to identical temperature regimes in an environment heated or cooled at a controlled rate (see ASTM E473).

5.3 DTA curve: Record of a differential thermal analysis where the temperature difference (ΔT) is plotted on the ordinated with exothermic reactions upward and time (t) or temperature (T) on the abscissa increasing from left to right.

5.4 DSC curve: A record of a differential scanning calorimeter experiment where the energy difference (ΔE) is plotted on the ordinate with exothermic reactions upward and t or T on the abscissa increasing from left to right.

5.5 Peak: That part of a DTA (or DSC) curve which is attributable to the occurrence of a single process. It is normally characterized by a deviation from the established base line, a maximum deflection, and a reestablishment of a base line not necessarily identical to that before the peak (see ASTM E474).

5.6 Peak temperature: The temperature corresponding to the maximum deflection of the DTA or DSC curve.

5.7 Onset temperature: The temperature at which a deflection from the established baseline in the positive direction is first observed.

5.8 Extrapolated onset temperature: Empirically, the temperature found by extrapolating the base line (prior to the peak), and the leading side of the peak, to their intersection (see Figure 1 and ASTM E474).

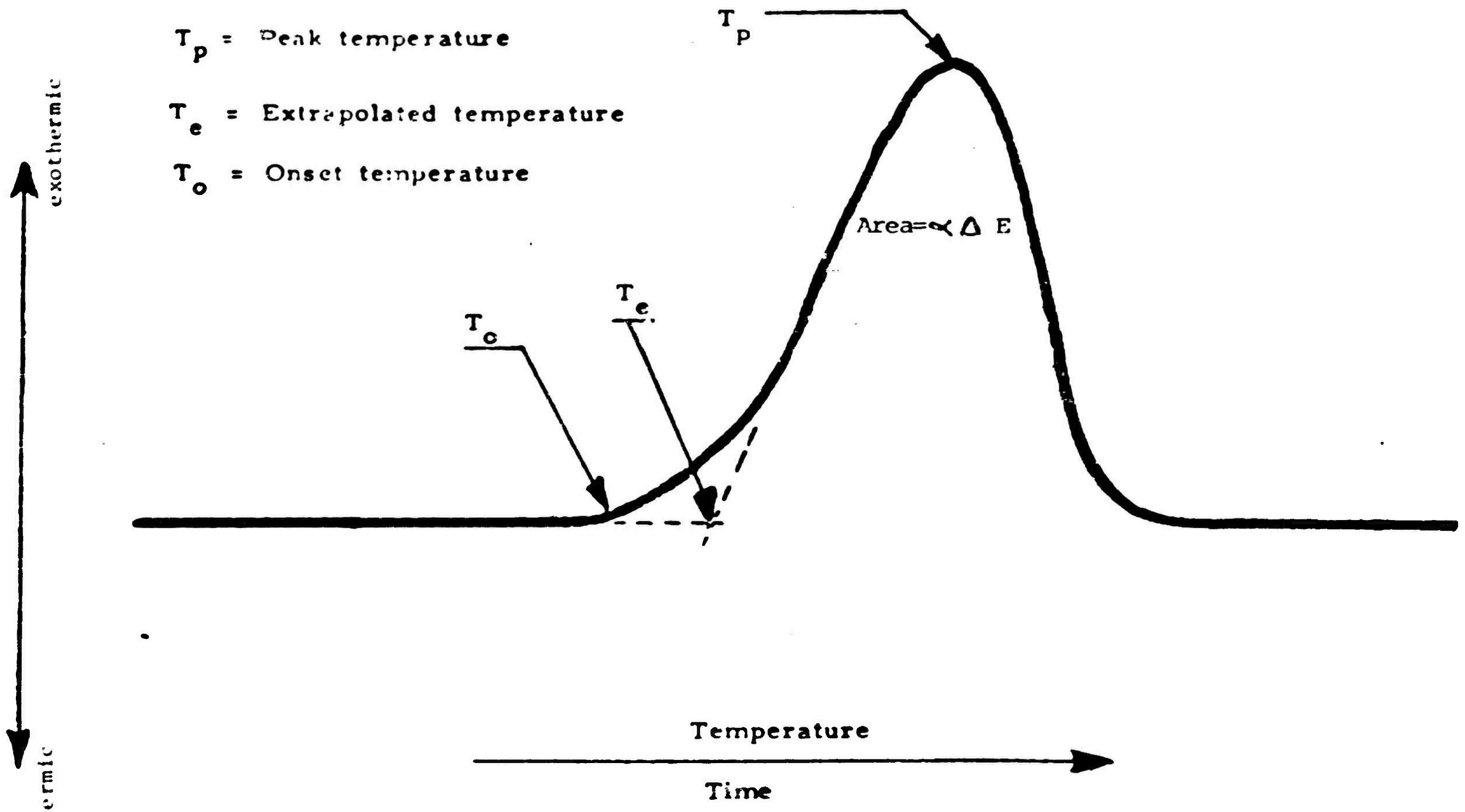


Fig. a-11.1 Typical DTA - DSC curve with exotherm

6. APPARATUS*

6.1 The equipment used in this test should be capable of displaying enthalpic changes, should be programmable and have the capability of subjecting the sample cell to different atmospheres.

6.2 Differential thermal analytical apparatus (DTA or DSC) can be purchased or custom-built to various degrees of refinement and sophistication. Basic to an apparatus satisfactory for this method are the following items:

a) Sample containers
b) Heating unit
c) Programmable temperature controller
d) Continuous measuring and recording equipment.

6.3 Analysis can be initiated at temperatures below ambient by providing a means of cooling the sample and reference, their respective holders, and the heating unit, to the same initial temperature.

7. CALIBRATION

7.1 A method for calibration of temperature scales for general use in differential thermal analysis (DTA or DSC) is given in ASTM E474-73.

7.2 For purposes of this test method, calibration of the absolute temperature scale within ±5°C is adequate.

8. SAMPLE AND REFERENCE MATERIALS

8.1 Generally the selection of an adequate sample size depends on the availability of the material, the degree of dilution required, the sensitivity of the instrument, the magnitude of the enthalpic change and the heating rate. Since this test is specifically oriented

---

*The Perkin-Elmer DSC and the DuPont 900 or 990 DTA instruments or their equivalent have been found suitable to meet the requirements of this test method.

towards assessing the potential hazards of chemicals, it should be carried out on as small a quantity of material as possible in order to minimize danger.

8.2 Samples should be representative of the material being studied and should be prepared to achieve good thermal contact between the sample and its container.

8.3 Sample size depends upon the sensitivity of the available equipment. Commercial DTA and DSC apparatus usually require from 1-50 milligrams.

8.4 In selecting the material of construction of the sample container, consideration should be given to possible interaction with the sample.

8.5 The reference material is selected so that it will undergo no thermal transformation over the temperature range under study.

8.6 Typical reference materials for DTA are calcined aluminum oxide, glass beads, silicone oils, and combinations of these; for DSC, normally an empty container is used for reference.

9. PROCEDURE

9.1 It may be necessary to use sealed containers or a pressurized system to insure the presence of a representative condensed phase. This is necessary in order to detect exothermic reactions occurring above the normal boiling point of a liquid or where the sample may be lost by volatilization prior to the temperature range of interest.

9.2 The sample and reference substances are placed in their respective containers in the heating unit of the apparatus. Sample and reference thermocouples are positioned so that adequate thermal contact is made as required by the apparatus.

9.3 The heating unit is turned on and the temperature

programmed for a heating rate of 10 C to 30 C per minute.
Note: Any increase in heating rate accentuates exothermic response, rendering it easier to observe but at the same time increases the onset and peak temperatures.

9.4 Differential and programmed temperatures are recorded. Heating is continued until the highest temperature of interest is reached or until the sample is destroyed or lost by volatilization.

10. SAFETY PRECAUTIONS

10.1 The use of this method as an initial test on materials whose hazard potential is unknown requires that precautions be taken during sample preparation and testing. For example, where particle size reduction by grinding is necessary, small samples should be used and adequate face and hand protection provided.

11. TREATMENT AND REPORTING OF DATA

11.1 Sample and reference substances should be identified by name or composition. The source, past history, purity and weight of sample should be stated.

11.2 The apparatus should be described together with the type of container used.

11.3 Sample environment should be identified as to degree of confinement, composition of atmosphere and whether the atmosphere is static, self-generated, or dynamic through or over the sample.

11.4 The test conditions should include heating rate and temperature range investigated.

11.5 The extrapolated onset and peak temperatures of all exothermic reactions should be extracted from the DTA or DSC curve and reported together with the magnitude of ΔT (DTA) or that of ΔE (DSC).

Process Operation	Inprocess Potential Temperature (°C)
Screw conveyors, pneumatic systems, hoppers, tote bins, screening, pressing,* glazing,* coating,* batch drum operations,* chutes	100
Belt conveyors, bucket conveyors, mills, flaker drum,* belt flaker,* packaging/filling (dry), machining	183
Extrusion,* rolling*	340
Dryer*	481
Melt pour,* casting*	423
Product pumps,* valves*	227
Filters*	200
Mix-melt mix operations*	124
Wash, mix and hold tanks,* gravity and centrifugal separators,* distillations,* solvent recovery,* wet scrubber	Operation specific
Reactors	Inherently hazardous unless well designed. Design must include well overdesigned cooling and agitation. A hazards analysis should be conducted to identify failure modes leading to runaway reaction and the system correspondingly designed to minimize the probability of an accident occurring.

---

*If the operating temperature is known, the inprocess potential should be taken as 1.2 x (operating temperature).

ELECTROSTATIC DISCHARGE TESTS

Background and Purpose

In process plants, electrostatic discharges (ESD) can occur in many ways. As a powder slides down a chute or out of a hopper, it exchanges electrical charge with the container wall. If the charge buildup is faster than the charge relaxation back to the wall, a sufficient voltage gradient can develop causing a discharge to occur (e.g., spark, arc, corona). If the discharge contains sufficient energy and is released in the proper manner (current density and duration) it can initiate a reaction in the material. Similar scenarios can occur in powder or aerosol clouds and dielectric liquids. ESD can also result from charge buildup on ungrounded equipment items, ungrounded persons, and dielectric surfaces. These situations are generally more hazardous in that the discharge energy can be significantly higher.

To characterize the susceptibility of a process material to develop charge, the electrical relaxation time is determined. This is estimated by measuring the material's permittivity and conductivity and taking the ratio of these values (extrapolated to a frequency of applied voltage of 1/second). The materials sensitivity to an electrical discharge is determined by passing electrical sparks of known energy through the sample with the sample in the form that it exists in the process.

Definitions

Permittivity

The permittivity of a material is a measure of its susceptibility to electrification. The permittivity, ε, is usually expressed as the relative permittivity, ε_r, with respect to the permittivity of free space, ε_o.

The relative permittivity is

$$\varepsilon_r = \frac{\varepsilon}{\varepsilon_o}$$

and is referred to as the dielectric constant, K. The permittivity of free space has a value

$$\varepsilon_o = \frac{1}{36 \times 10^9} = 8.85 \times 10^{-12} \ (\text{coul}^2/\text{n-m}^2)$$

The dielectric constant of a material can be determined by measuring the influence of the test material on the capacitance of a parallel plate condenser. A condenser is formed wherever an insulator (i.e., dielectric) separates two conductors between which a difference of potential can exist.

In the case where the condenser electrodes are plates having a constant spacing, the capacitance, C, is given by the expression

$$C = 0.08842\ K\ \frac{A}{d} \qquad \text{(pf)}$$

where

A = area of active dielectric in square centimeters

d = spacing between plates in centimeters

K = dielectric constant

The dielectric constant is a material property and is substantially independent of frequency unless polar effects are involved. For polar materials, the dielectric constant under alternating current conditions is increased. The extent to which this polar action is effective depends upon the frequency and the temperature.

Conductivity

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

$$\sigma = \frac{d}{A_s R} \qquad \text{(mho/cm)}$$

where A_s and d are the cross-sectional area and length of the sample, respectively.

Relaxation Time Constant

The ability of a material to store electrostatic charges is related to the relaxation time, τ. If the relaxation time is short, charges will be dissipated as fast as they are acquired. If it is long, more charges will be acquired than lost and the electric charges will build up on the surface of the material. The relaxation time constant for a material can be calculated

$$\tau = \varepsilon/\sigma \qquad \text{(seconds)}$$

where ε is the permittivity and σ is the conductivity. For a particular material sample

$$\tau = RC = \rho\ \frac{d}{A_s} \times \varepsilon_o K\ \frac{A_s}{d} = \rho\varepsilon_o K = \rho\varepsilon \qquad \text{(seconds)}$$

If a material is not polar, there is no problem determining the relaxation time since there is no frequency effect. If a material is polar, the measured relaxation time (ε/σ) versus frequency relation is extrapolated to a frequency of 1/second and that low frequency value is used in hazard classification.

Apparatus and Procedure

For materials which exist in dust or vapor clouds, the cloud explosion test (Hartman Apparatus) should be used to determine the flammable/explosive concentration limits and the minimum ignition energy. Refer to the cloud explosion test procedure for these cases.

Permittivity Measurements

The dielectric constant of an inprocess material is obtained by measuring the effect of the material on the capacitance of a parallel plate condenser. Figure a-12 is a block diagram of the test setup used for these determinations.

For inhomogeneous samples such as pellets and strands, the parallel plate capacitor consists of two aluminum disks 45.72 cm (18 in.) in diameter and 2.54 cm (1 in.) thick. For homogeneous samples such as powders, slurries, pastes and liquids, a smaller capacitor is acceptable (1.91 cm (0.75 in.) long by 1.91 cm (0.75 in.) diameter). Copper leads 0.0794 cm (0.0312 in.) by 0.635 cm (0.25 in.), approximately 45.72 cm (18 in.) long are used to connect the parallel plates to the measuring equipment. The measuring equipment consists 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 (or equivalent equipment).

The teflon ring is used to contain the sample in a cylindrical shape and also to maintain the parallel plate spacing. For inhomogeneous samples the teflon ring should have a diameter of 23.50 cm (9.25 in.) I.D. and 24.13 cm (9.50 in.) O.D., and a height of 1.27 cm (0.50 in.).

For homogeneous materials, a smaller teflon ring and corresponding smaller capacitor plates are also acceptable (2.54 cm (1 in.) O.D. by 1.96 cm (0.75 in.) I.D. by 0.51 cm (0.205 in.) height).

Before conducting the tests, the contribution to the measured capacitance due to stray capacitance and fringing must be determined. Small teflon spacers are used to separate the plate by the same gap as is produced by the teflon ring. The capacitance of this configuration (air gap) is measured, and an effective plate area A_a is computed from the equation

$$A_a = \frac{C'}{\varepsilon_o d}$$

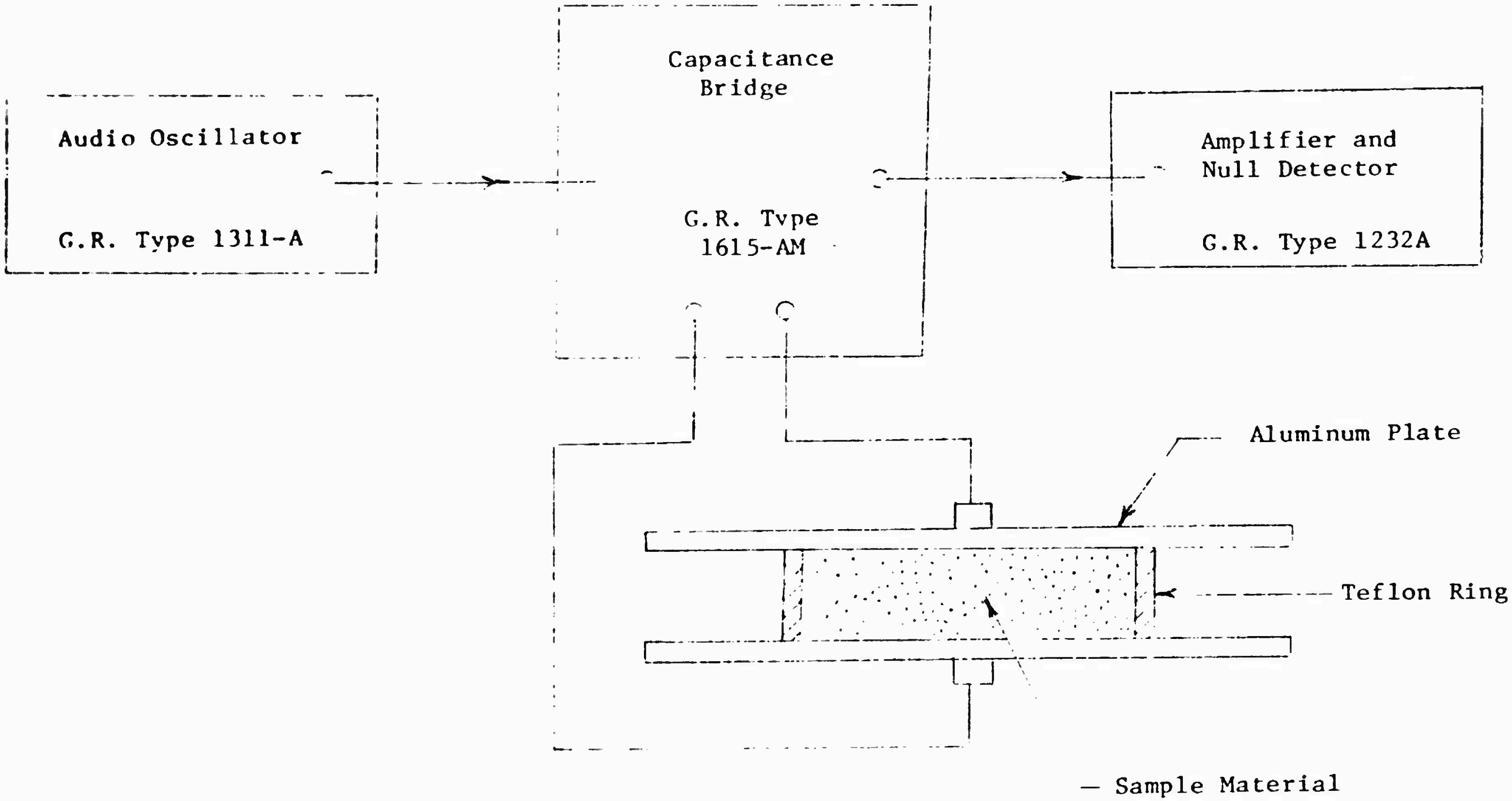


Fig a-12 Block diagram of test configuration for the permittivity test where C' is the measured capacitance, ε_o is the permittivity of the air (8.85×10^{-12} coul/n-m^2), and d is the plate separation distance. The difference between the effective plate area and the actual area is due to fringing and stray capacitance.

Next the teflon ring holder is placed at the center of the aluminum disk and filled with the test material. The second aluminum disk is placed on top of this assembly as shown in figure a-12. The capacitance of the assembly, C, is measured, and the dielectric constant for the sample is computed from

$$K = \frac{C\,d}{\varepsilon_o A_s} - \frac{(K_o A_a + K_1 A_c)}{A_x}$$

where

K = dielectric constant of sample

K_1 = dielectric constant of ring holder ($K_1 = 2.1$)

K_o = dielectric constant of air ($K_o = 1.0$)

d = plate separation (cm)

A_a = effective cross-sectional area of air area (cm^2)

A_c = ring holder cross section area (cm^2)

A_s = sample cross section area (cm^2)

Measurements are made at frequencies of 0.05, 0.10, 0.50, 1.00, 2.00, 5.00 and 10 kHz (or over an equivalent range depending on the equipment used).

Conductivity Measurements

The same electrodes and teflon ring as were used for permittivity measurement are also used to measure conductivity (or equivalently resistance) as a function of applied alternating voltage frequency. The measured sample resistance R is used to compute conductivity from the relation

$$\sigma = \frac{d}{A_s R}$$

where A_s is the sample cross-sectional area.

ESD Ignition Test

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 schematic circuit diagram of the test is shown in figure a-13 and the electrode arrangement is shown in figure a-14 for powders or liquids and pellets or strands.

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 0.1 microfarad. Two voltages must be measured to calculate the energy. These are shown in figure a-14. The time histories on the voltage across the sample, $V_{sample}(t)$, and the current through the sample, $I(t)$, are needed. By referring to figure a-14, the current can be obtained by dividing the voltage across the resistor by the resistance R_i:

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

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

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

From these two measurements the energy is calculated as:

$$E = \int_0^t \frac{V_i(t)}{R_i} \times \Big(V(t) - V_i(t) \Big)\, dt$$

Procedure

For these tests the relative humidity should not exceed 40 percent, and ambient air temperatures should be between 10 and 32°C. The needle electrodes 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.

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

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

The test sample is first placed in the sample holder and mounted in the test fixture. The electrode spacing is then adjusted as shown in figure a-14. 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.

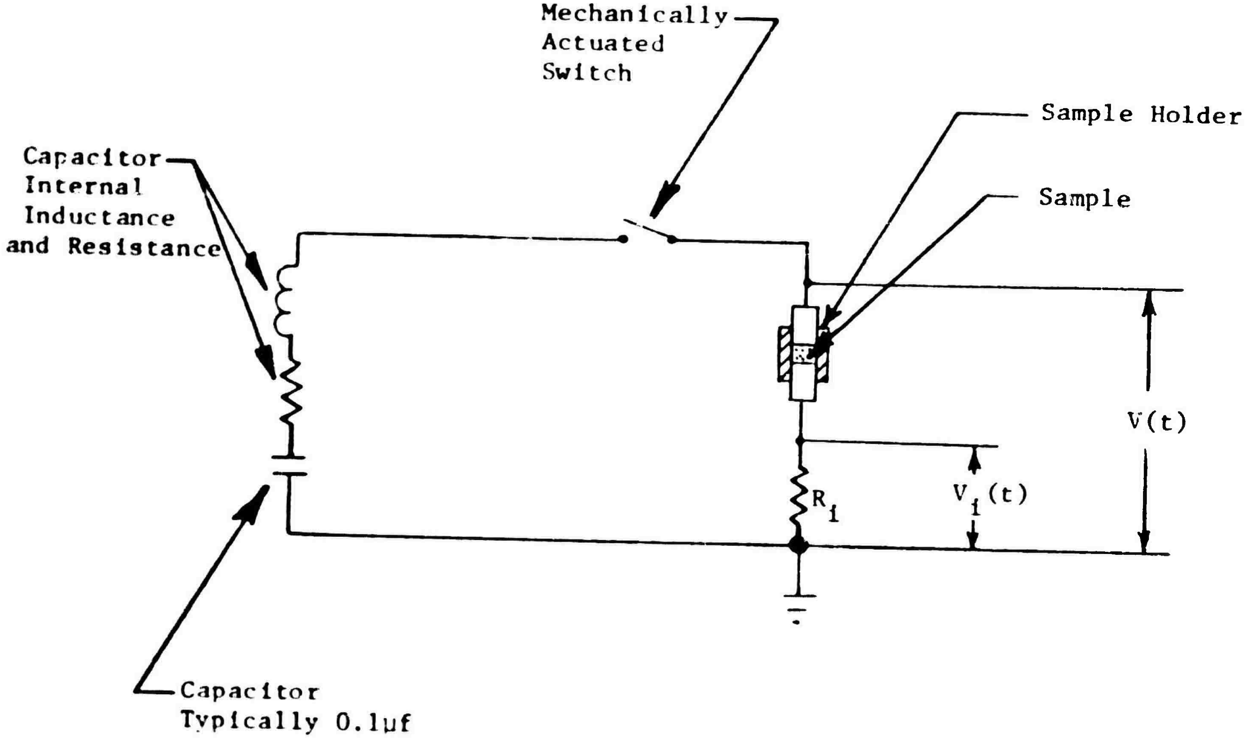
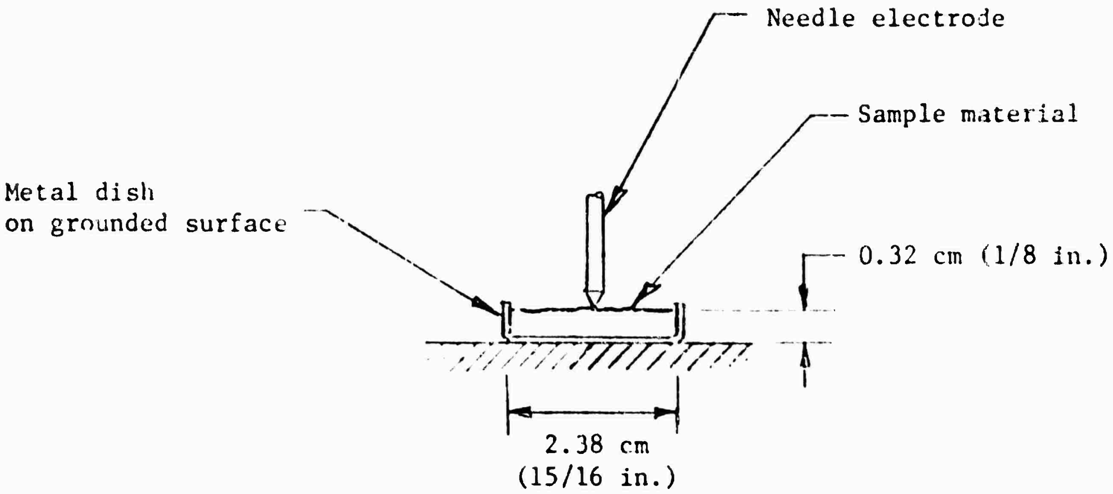


Fig a-13 Schematic circuit diagram for electrostatic discharge test



Powder samples

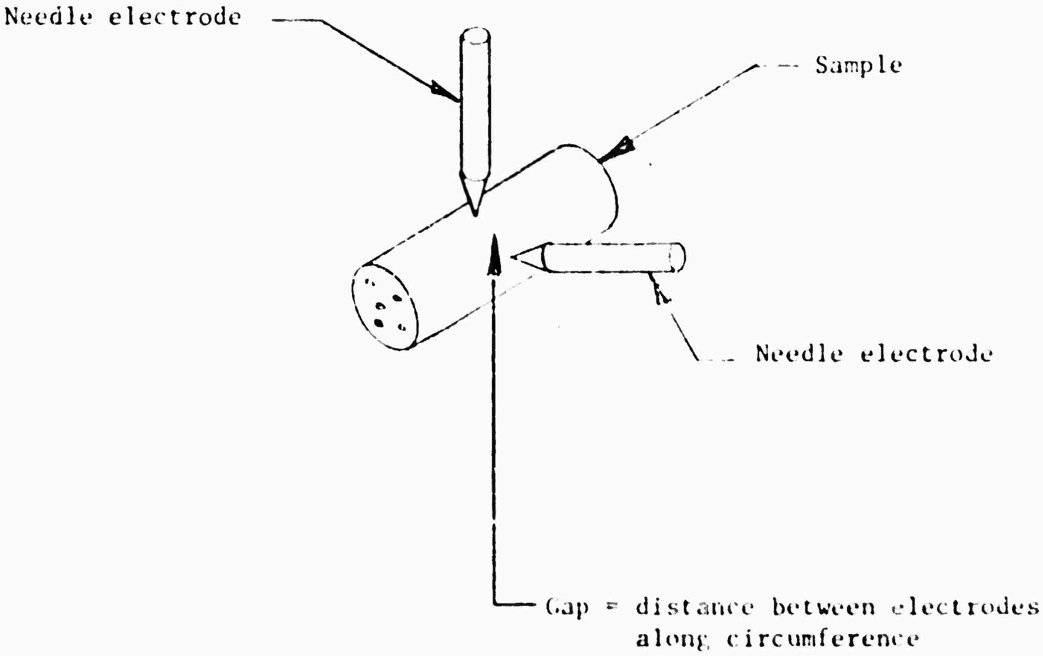


Fig a-14 Sample holders for electrostatic discharge tests

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. Based on past experience the energy actually discharged in the sample will be on the order of 10 to 30 percent (typically 18 percent) of the energy stored in the capacitor. The charging voltage should be varied ± 1 kV in Bruceton fashion over the full range of voltage. The electrode spacing should thus 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. Electrode spacings of 0.318 cm (1/8 in.), 0.159 cm (1/16 in.), and 0.079 cm (1/32 in.) should be used. Additional electrode spacings may be required to clarify the trends in the data.

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.

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:

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

The first expression is used when both positive and negative reactions have occurred at an energy level. The second expression is for cases where only positive reactions or only negative reactions were observed at the energy level. By plotting the energy on arithmetic probability paper, the 50 percent point can be estimated.

To clarify the data analysis, an example is described next. The data collected for black powder using a 0.01 μF capacitor are shown in figure a-15. 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 a-1 was prepared. This provided the information for calculating the statistical parameters summarized in table a-2. The energy is then plotted against the probability of a reaction, (p), plus or minus the deviation, on arithmetic probability paper (Fig a-16). 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.

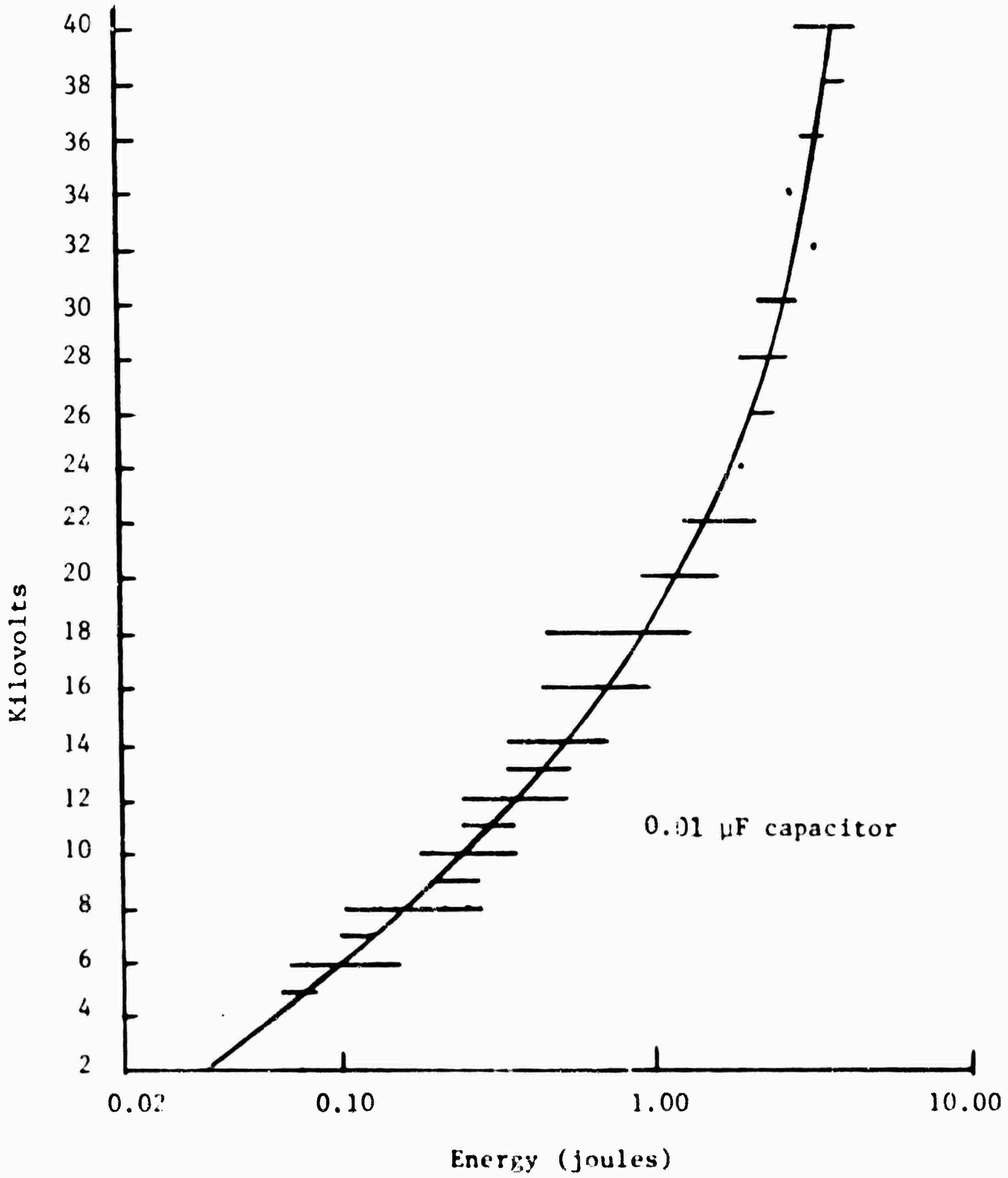


Fig a-15 Determination of 50 percent probability of ignition energy level for ESD

Table a-1

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	0.530

Capacitor = 0.01 μF

Table a-2

Statistical summary of ESD test data for black powder

E (joules)	n	p	q	$\pm 0.5\ n^{1/2}$	$\pm (pq/n)^{1/2}$
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	± 1.21	-
0.130	1	1.0	0	± 0.50	-
0.160	2	1.0	0	-	-

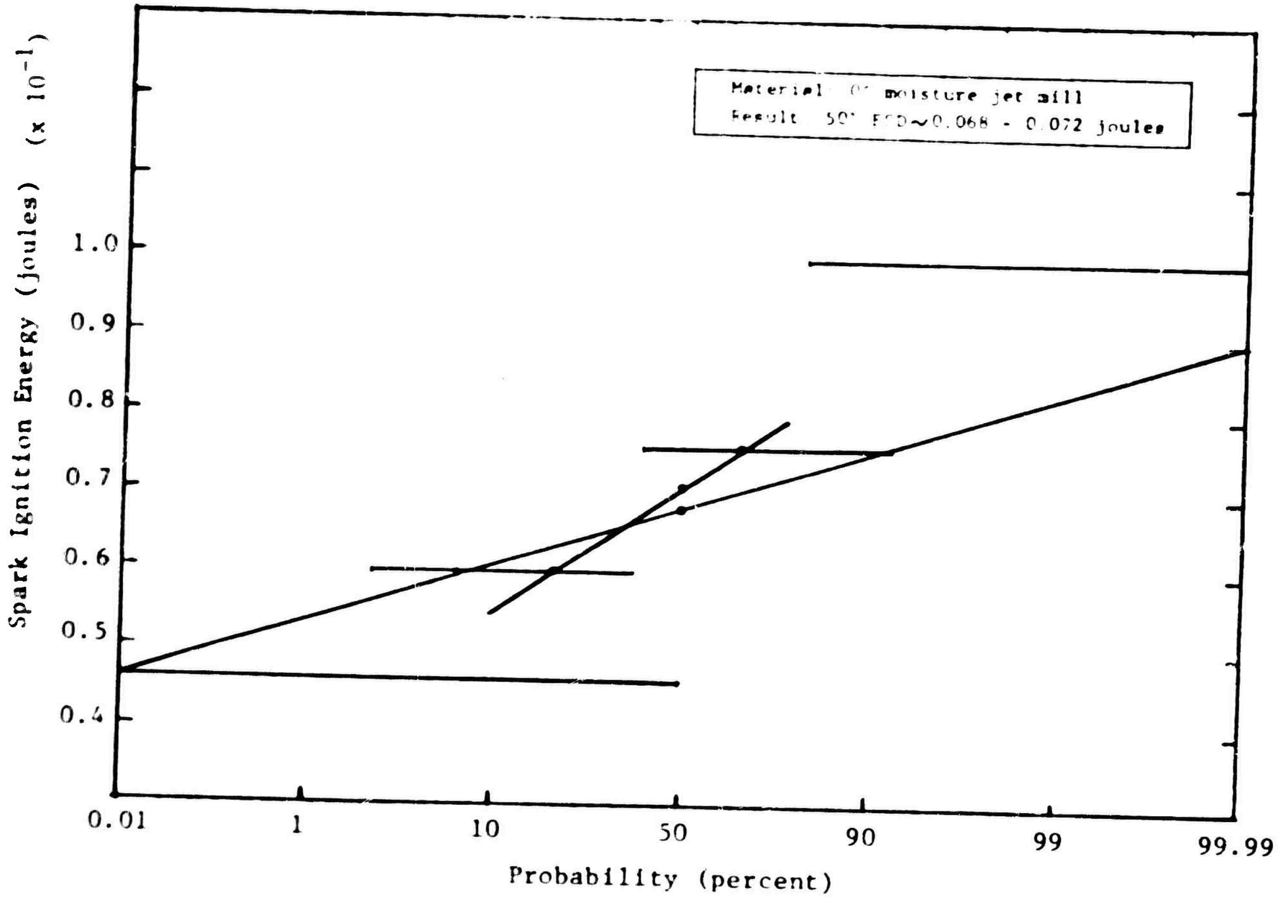


Fig a-16 Determination of 50 percent ignition energy from experimental data (example)

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.

Interpretation of Results

Two evaluations are accomplished by the ESD test series. The measured permittivity and conductivity are used to determine the electrical relaxation time (indication of charging susceptibility) and the 50 percent ignition energy is used to compute the safety factor for this stimulus.

To determine the electrical relaxation time, τ, divide the permittivity ε by the conductivity σ:

$$\tau = \varepsilon/\sigma$$

If the material's relaxation time is found to be a constant, nearly independent of frequency (i.e., the material is nonpolar) there is no problem interpreting the results. However, if the material is found to be polar, the relaxation time should be plotted versus frequency on log-log graph paper. A curve should be fit to the data points and extrapolated to a frequency of 1/second. The value of relaxation time at the 1/second frequency is taken as the material relaxation time.

If the relaxation time is found to be below 0.01 second, the material is considered to be "not susceptible to charging". If the relaxation time is between 0.01 second and 1.0 second the material is considered to be "marginally susceptible to charging". If the relaxation time is greater than 1 second, the material should be considered to be "highly susceptible to charging". The charging susceptibility of the material should be recorded with the material ignition safety factor (see below). The charging susceptibility is a qualitative indicator of how likely it is that the material will build up sufficient charge within itself to produce internal electrical discharges.

The ESD ignition safety factor is computed by dividing the minimum 50 percent ignition energy found from the sensitivity testing by the inprocess potential energy given in the following list for the appropriate process operation.

$$SF(ESD) = \frac{\text{Sensitivity test ESD energy}}{\text{Inprocess potential energy}}$$

Process Operation	Inprocess Potential Energy (joules)
Belt conveyors	0.03
Glazing, coating, batch drum	0.045
Pneumatic systems, screening, packaging/filling (dry), machining	1.0
Hoppers, tote bins	12.0
All other operations, except product pumps and valves, and mills	0.017

FLAME IGNITION TEST

Purpose

The hazards classification procedure is divided into two parts: (1) a sensitivity evaluation (how likely is an initiation to occur and which stimuli are most likely to be the cause?), and (2) an effects evaluation (what is the most likely result of an initiation and how bad will it be?). If the sensitivity evaluation shows that the material is insensitive (safety factor is greater than 3) and it passes the flame ignition test, the material can be immediately classified as "very insensitive", class 1.5. If the sample does not pass the flame ignition test the effects evaluation must be completed to classify the material.

Apparatus

A standard Bunsen burner with a 9.5 mm (0.37 inch) inside diameter barrel and a propane gas supply is required for this test. A thermocouple should be used to measure the flame temperature when adjusting the burner. The overall test arrangement is shown schematically in figure a-17. A stand and clamp are required to hold the burner at a 45 deg angle to the vertical as shown. An arrangement must be provided to remotely (no personnel in the area) move the sample in a dish into the flame.

Procedure

1. With the gas burner in its vertical position standing on a table, the burner should be lit and the flame adjusted to produce a 25 to 35 mm (1 to 1-1/4 inch) high inner cone. The top of the inner cone should be 960 ± 5°C as measured using a thermocouple.

2. Once adjusted, the burner should be placed in its holder so that the flame points downward and the barrel is at a 45 deg incline.

3. The stand for the sample dish should be positioned such that the sample is well away from the flame at the beginning of the test. The sample is placed in the sample dish and the dish placed on the stand. All personnel should leave the area and observe the test remotely (e.g., with mirrors, a safe window, or a television monitor). The sample is then remotely moved into the flame so that the tip of the inner blue cone just contacts the surface of the sample. The height to which the stand must be raised in order to accomplish this is determined before the test without the sample in place. The sample is held in position in the flame for 1 minute.

Personnel should not enter the test area until the sample has had sufficient time to cool to room temperature. If an ignition occurs at any time during the test, the material has failed the test and the effects evaluation must be accomplished. If no ignition occurs, the material is immediately classified as "very insensitive", class 1.5, and no further testing is required.

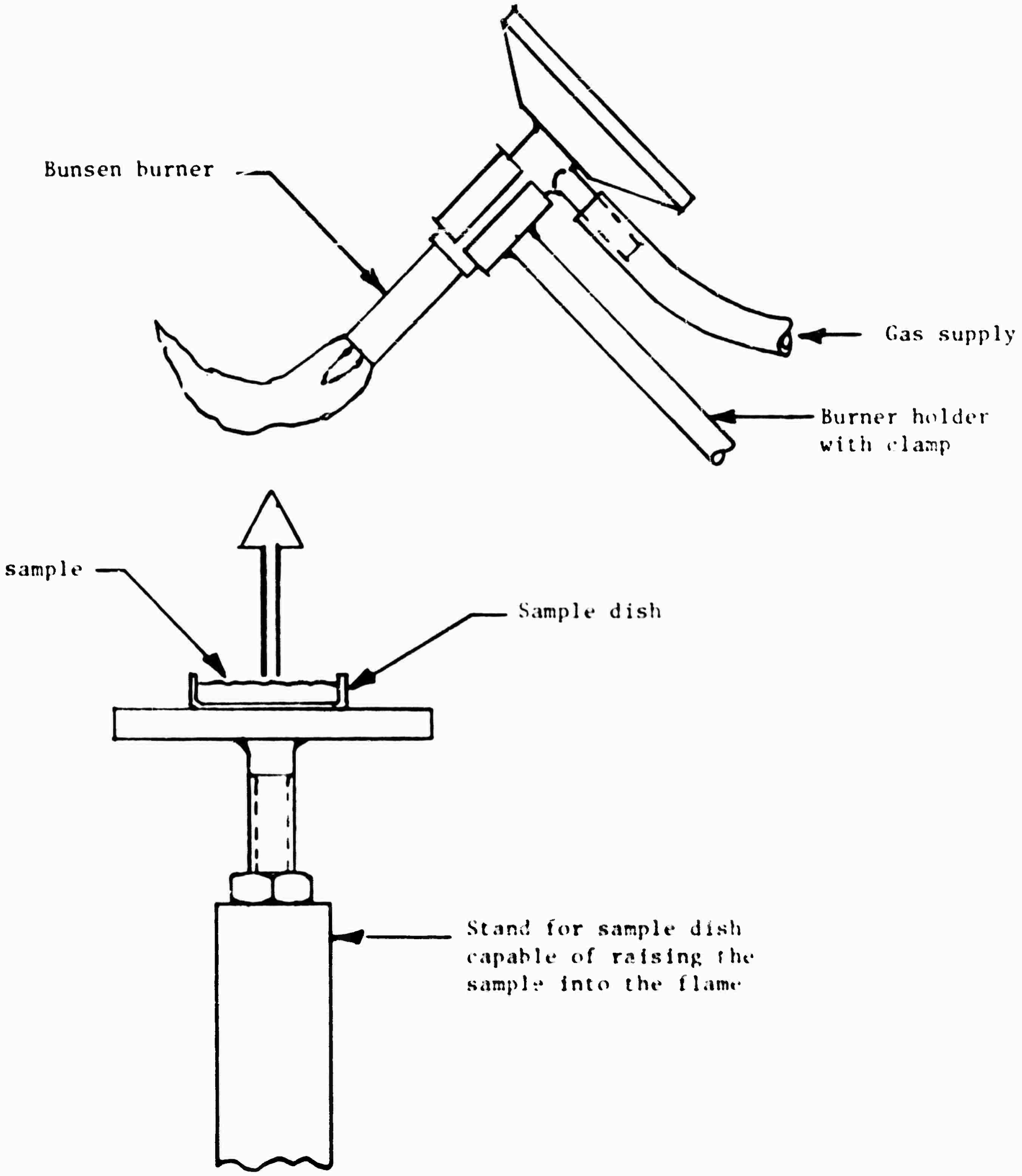


Fig a-17 Flame ignition test arrangement

CRITICAL DIAMETER TEST

Purpose

The critical diameter test determines whether or not a detonation is likely to propagate in a process vessel of known size. The "critical diameter" is the inside diameter of a circular cylindrical vessel (a tube) at which a detonation will propagate (not die out) through the process material 50 percent of the trials. Larger diameter process vessels will propagate a detonation more often and smaller diameter vessels less often. In the hazards classification procedure, the critical diameter is used as criteria to help decide which effects test (mass explosion or mass fire) should be conducted. If the equivalent actual process vessel diameter (as defined below under interpretation of results) is equal to or larger than the critical diameter, the material is assumed to be capable of propagating a detonation and the "tube transition test" should be done next. If the equivalent actual process vessel diameter is less than the critical diameter, the material is assumed to be incapable of propagating a detonation and the "mass fire test" should be done next.

Apparatus

The basic critical diameter test apparatus is shown in figure a-18. A tube of the same material as exists in the process component being evaluated[*] is welded to a 2.54 cm (1 in.) thick steel plate.[**] The tube wall thickness should be the same or slightly greater (depending on commercially available wall thicknesses) than the process component's wall thickness. The tube length should equal six times the inside diameter Ten 0.226 cm (0.0890 in.) diameter (number 43 drill) holes should be drilled through the tube wall along a line as shown in figure a-18, evenly spaced from top to bottom of the tube. These holes are for inserting fiber optic "light pipes"[#] for recording reaction front velocity as a function of tube length. Any equivalent system for measuring reaction front velocity is acceptable, however, for this test a continuous velocity probe should not be used, especially for the very small diameters (e.g., less than 10 cm (4 in.) I.D.). The continuous probe could disturb the reaction as it progresses and yield erroneous results.

---

[*] Types 1018 or 1020 carbon steel should be used if the component material is not known; an appropriate stainless steel should be used if the sample is a highly reactive chemical.

[**] For diameters less than or equal to 2.54 cm(1 in.), the witness plate thickness can be reduced to 1.27 cm (1/2 in.) or 0.95 cm (3/8 in.).

[#] One suggested supplier is E. I. DuPont deNemours and Company, Type 1110 plastic fiber optics, 16 strands, 10 mil fibers (Crofon light golds). Any equivalent fiber optic "light pipe" is acceptable.

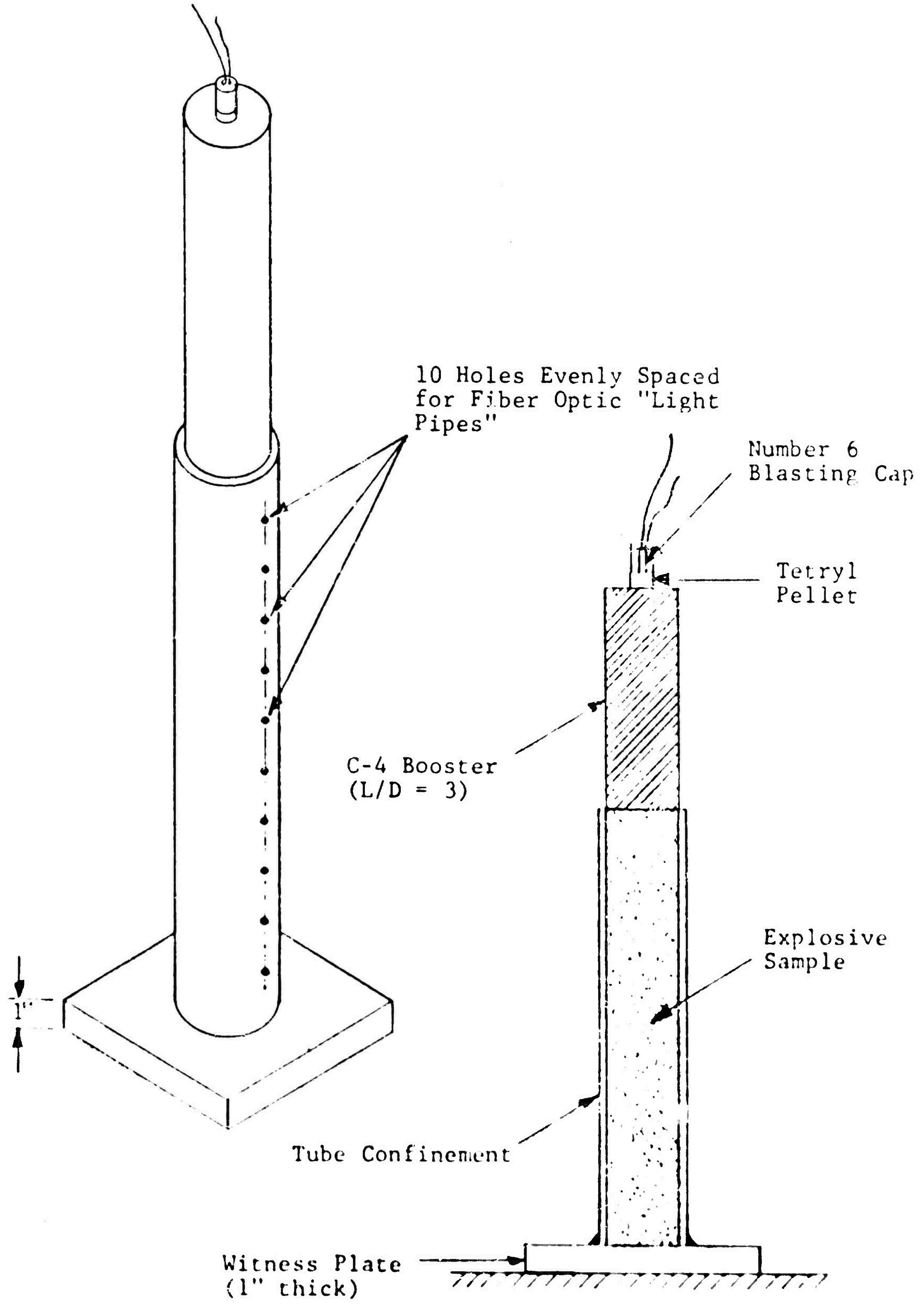


Fig a-18 Critical diameter test apparatus

When the sample is a liquid, the fiber optic probes should be sealed in the hole using a compatible glue and the tube should be welded totally to the steel witness plate to prevent leakage. If the liquid is reactive, the tips of the fiber optics may have to be protected by a glass plate or bead. The C4 booster should be wrapped tightly in a thin compatible plastic membrane to prevent contamination of the booster material by the liquid. The booster should be in good contact with the sample material through the thin plastic sheet. In all cases where the sample could react with the test fixture (e.g., a reactive liquid) the sample should be contained inside the test fixture within a thin bag which is chemically compatible (not reactive) with the sample or the test fixture should be an appropriate stainless steel.

The sample material should be in a form representative of the form in which it exists in the actual process. This does not merely mean that the sample should be chemically correct, but the process bulk density and operating temperature should be reproduced in each test as accurately as practical.

The C4 booster for typical critical diameter tests should be a cylinder with diameter equal to the inside diameter of the pipe and having a length to diameter ratio of at least 3 to assure a fairly flat initiating shock front. For larger tests, where the booster weight becomes excessive, the cylindrical booster can be replaced with a right circular cone with at [illegible] a 90 deg cone angle. In this case the diameter of the base of t[illegible]e should be equal to the inside diameter of the tube. For sma[illegible]sts the length of the booster should be at least 2.54 cm (1 in.) to assure sufficient length to develop a stable detonation in the booster. The booster diameter should be no smaller than 10 mm to assure that a strong detonation will occur in the booster.

General Procedure

A Bruceton type procedure is used to estimate the diameter at which a detonation will propagate 50 percent of the time. This is called the "critical diameter" in this procedure. In the Bruceton test series a decision must be made as to whether or not the detonation will propagate for each test. Once the critical diameter (50 percent propagation diameter) is determined, it is compared to an equivalent diameter of the actual "full scale" process vessel to determine if a detonation can propagate in the actual system.

Criteria for Distinguishing Positive Results (Propagating Reactions, Go) from Negative Reactions (No Go)

The following criteria will be used to help determine whether the result is positive (Go) or negative (No Go) for each individual test conducted.

1. If the witness plate is not damaged (still smooth and flat), the test is a No Go.

2. If unreacted material remains after the test on the witness plate, the test is a No Go.

3. If the plot of reaction front velocity versus distance is clearly decreasing throughout the tube length (no stabilizing of the curve) the test is a No Go.

4. If the tube near the witness plate "banana peals" or is left intact the test is a No Go.

5. If the reaction front velocity remains high (~2000 to 8000 m/sec) at the end of the L/D = 6 tube (at far end of tube it must show stability), the test is a Go.

6. If a clean hole is punched through the witness plate the test is a Go.

Bruceton Procedure to Determine the Critical Diameter

The first test of a series will be done at a diameter which is estimated to be about the critical diameter, if such a diameter can be estimated based on prior tests with similar materials or based on other information available. If such an estimate cannot be made, the first test will be done in a 10.16 cm (4 inch) diameter tube. The first group of tests of a series are designed to approximately find the critical diameter before entering the Bruceton procedure. If the first test is negative (no detonation) the tube diameter for the next test should be doubled. If the first test is positive the tube diameter should be halved. This procedure (increasing or decreasing by a factor of 2) should be continued as long as the results are the same as the first test. As soon as the result changes (i.e., a string of positive results with the last test negative or a string of negative results with the last test positive) the second group of tests, the Bruceton series, will be entered.

The first test of the second group will be a diameter given by

$$D_o = 10^{1/2(\log D_H + \log D_L)}$$

where D_H and D_L are the previous two test diameters tried. D_H is the previous diameter at which a detonation was observed and D_L is the previous diameter at which no detonation was observed. D_o is the average diameter on a log scale. The Bruceton series begins with the very last test of the initial critical diameter search. Beginning with that test each time a detonation is observed, the next test will be conducted at the next lower size increment and each time a detonation is not observed, the next test will be at the next larger diameter. The diameter will be increased or decreased so that the increment on the log diameter scale is always 0.1505. Equivalently, the diameter will be increased or decreased by a factor of $\sqrt{2}$. For example, if the initial diameter is 10.16 cm (4 inches), the pipe diameters used in the up-down sequence of tests will be as shown below:

	Exact		Practical Equivalent (assuming 0.318 cm (1/8 inch wall)		
	cm	(inch)	cm	(inch)	
	57.48	(22.63)	58.000	(23.000)	Special formed tube
	40.64	(16.00)	41.000	(16.000)	Special formed tube
	28.73	(11.31)	28.000	(11.000)	
	20.32	(8.00)	20.000	(8.000)	
	14.38	(5.66)	14.61	(5.750)	
Initial Diameter	10.16	(4.00)	10.000	(4.000)	
	7.19	(2.83)	7.303	(2.875)	
	5.08	(2.00)	5.000	(2.000)	
	3.58	(1.41)	3.543	(1.395)	
	2.54	(1.00)	2.540	(1.000)	
	1.80	(0.71)	1.748	(0.688)	
	1.27	(0.50)	1.300	(0.500)	
	0.90	(0.35)	0.953	(0.375)	
	0.64	(0.25)	0.660	(0.260)	

The results should be recorded as shown in figure a-19. A minimum of 10 tests beyond the initial search for the approximate critical diameter must be completed. For good accuracy at least 100 tests should be done. To estimate the diameter at which 50 percent of the tests would result in a detonation (the critical diameter), only those tests involving incrementing diameter by $\sqrt{2}$ will be used. Of those tests, only the symbols occurring less frequently are used in the calculations. In the example shown in figure a-19, the six circles (No Go's) are used in the calculations.

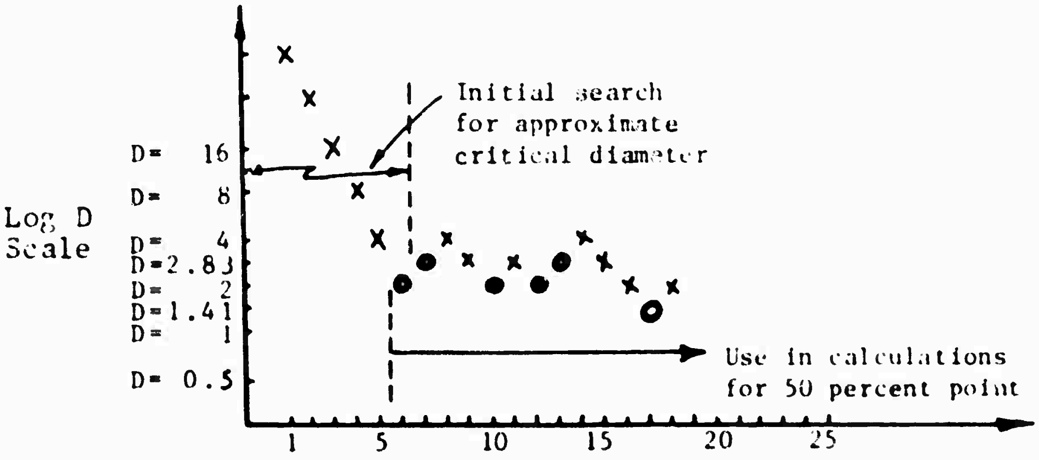


Fig a-19 CD Bruceton series

Table a-3 is set up as an example corresponding to figure a-19. In the table, i is a level assigned to each of the D values used, with 0 at the lowest level at which the symbol being used appeared. The parameter n_i represents the number of occurrences of the symbol at each level i. The parameters N and A are column totals as shown in the table.

Table a-3 Example of Bruceton calculation

	D_i	i	n_i	in_i
	2.83	2	2	4
	2.00	1	3	3
(D_o)	1.41	0	1	0
	Totals		N=6	A=7

The critical diameter is then estimated based on the formula

$$\log_{10} (\text{critical diameter}) = \log_{10} D_o + 0.1505 \left(\frac{A}{N} \pm \frac{1}{2}\right)$$

where a + sign is used if the calculation was based on No Go's (0) and a - sign is used if the calculation is based on detonations (X). In the example shown, the estimated critical diameter (diameter corresponding to 50 percent probability of detonation) is found to be 2.51 diameter units.

Interpretation of Results

The critical diameter determined from the tests described previously now must be compared to the equivalent actual (full scale) process vessel diameter to help determine which effects tests (mass explosion or mass fire) should be done. If the equivalent actual process vessel diameter is equal to or larger than the critical diameter, the material is assumed to be capable of propagating a detonation and the "tube transition test" should be done next. If the equivalent actual process vessel diameter is less than the critical diameter, the material is assumed to be incapable of propagating a detonation and the "mass fire test" should be done.

Process containers can have simple or complex configurations and in certain cases, some judgment may be required to determine the equivalent diameter. For process vessels of constant cross section along the vessel's length, the equivalent vessel diameter for some different geometries can be estimated using the equations shown in figure a-20. If the cross section size changes along the vessel length, (e.g., the vessel is a truncated cone on a sphere), the maximum equivalent diameter along the vessel length should be used.

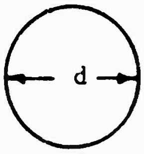
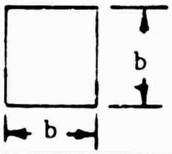
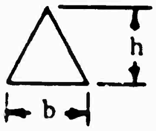
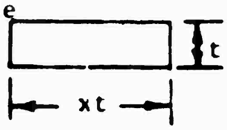
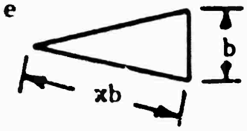
Shape and Characterizing Dimensions	Critical Value of Characterizing Dimensions
Circle 	$d_{eq} = d$
Square 	$d_{eq} = b$
Equilateral Triangle 	$d_{eq} = b/\sqrt{3}$
Rectangle  where x>1	$d_{eq} = \left(\frac{2x}{x+1}\right) t$
Isosceles Triangle  where x>1	$d_{eq} = \sqrt{\frac{2x-1}{2x-1}}\ b$

Fig a-20 Equivalent diameter for various cross section geometries

Unknown Critical Diameter

Most test sites have a limitation on the quantity of material which can be involved in an individual test. If the critical diameter can be determined from testing at sizes below the test site limitation, there is no problem. However, if the critical diameter is larger than the allowed size (based on the test site limitation) and the actual process vessel equivalent diameter is also larger than the test sites limit, we cannot say for sure that a detonation will not propagate in the full scale. In this case, the most conservative initiation consequence is assumed. The "mass explosion test" should be done next. If the TNT equivalence derived from the mass explosion test is greater than or equal to 10 percent, the material is classified as class 1.1A. If the TNT equivalency is less than 10 percent, the "mass fire test" must be done to classify the material.

Naturally, if the actual process vessel equivalent diameter is within the test site limit, even though only negative results are obtained, up to the site limitation, it is clear that the critical diameter is greater than the equivalent process vessel diameter. Therefore, in this case, the "mass fire test" should be done next.

TUBE TRANSITION TEST

Purpose

The purpose of the tube transition test is to determine the minimum length for transition from burning to detonation in a thick walled container (critical length). This test is relevant for materials in bulk in their process vessels. For example, cylindrical vessels, hoppers, and box shaped containers are relevant, whereas materials in layers are not. This is a screening test in the hazards classification procedure. If the critical length (at the proper pipe diameter) is less than the process container length, transition to detonation in the actual process vessel is credible and "mass explosion" testing should be completed. Conversely, if the process container is shorter than the critical length, the "mass fire" test should be done.* It must be noted that the tube transition test described here is quite conservative in some cases. If a container has a thin metal wall, it is reasonable to expect that a pressure vessel rupture or explosion will occur before detonation can be established. Therefore detonation may not be possible in the actual system while a short critical length is given by the test described here. This is roughly accounted for in the use of critical length in screening for selection of the required effects testing in the overall hazards classification procedure.

Test Arrangement

The test apparatus is shown in figure a-21. A black seamless schedule 160 pipe holds the sample material. The sample bulk density is chosen to be the highest bulk density likely to exist in the actual process vessel. For example, for a hopper the bulk density of material at the bottom of the hopper will be greatest since it is compressed by the weight of material on top. A 1.27 cm (1/2 in.) thick steel witness plate is tack welded to one end of the pipe. The other end (the initiating end) is threaded for a standard pipe cap.

Two small holes should be drilled in the witness plate. A hole is required for insertion of a steel cased continuous resistance velocity probe, as shown in figure a-21. The probe tip should be near to the initiating end of the pipe but must not touch the pipe cap. The probes position must be carefully recorded so that the location at which detonation begins can be estimated from the probe voltage versus time trace.

The continuous velocity probe is illustrated in figure a-22. The resistance wire, partially insulated by a space wrapped nylon thread, is inserted into a small-bore 304s stainless tube. The tube and resistance wire are crimped at the closed end to form an electrical junction.

---

*If the container is closed and a pressure vessel explosion is expected, a "mass explosion" test should be done here also.

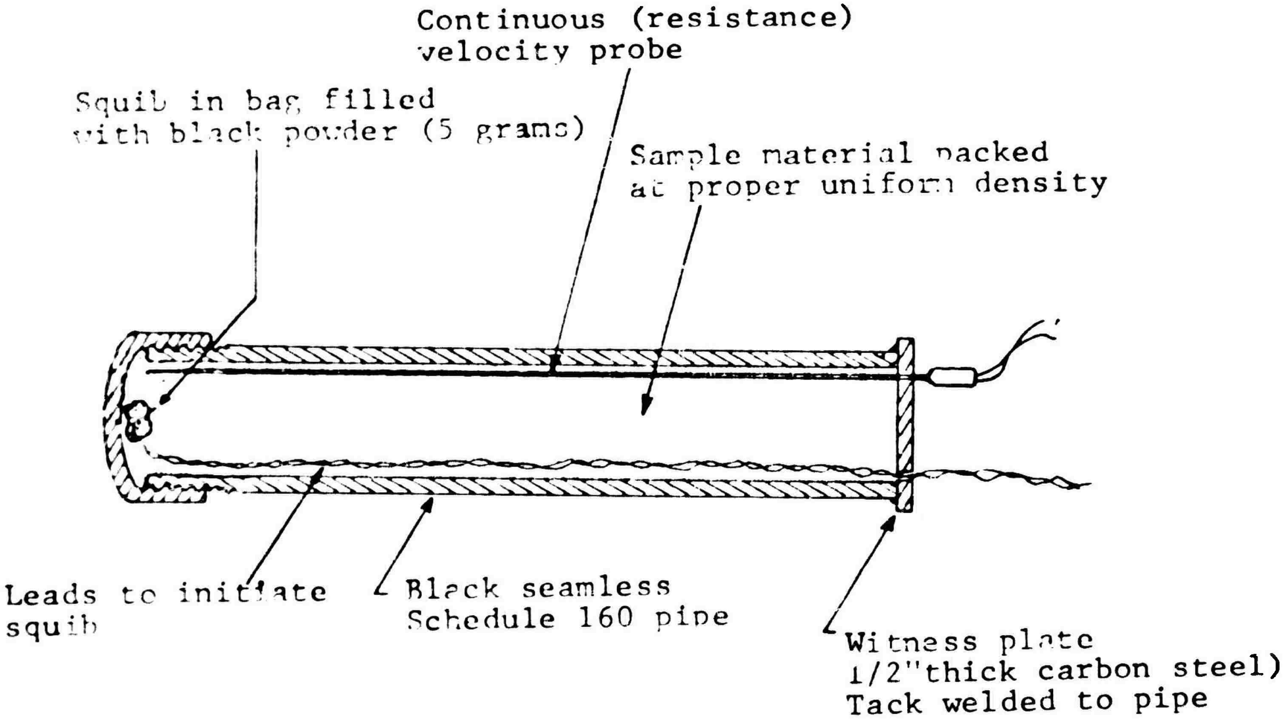


Fig a-21 Tube transition test arrangement

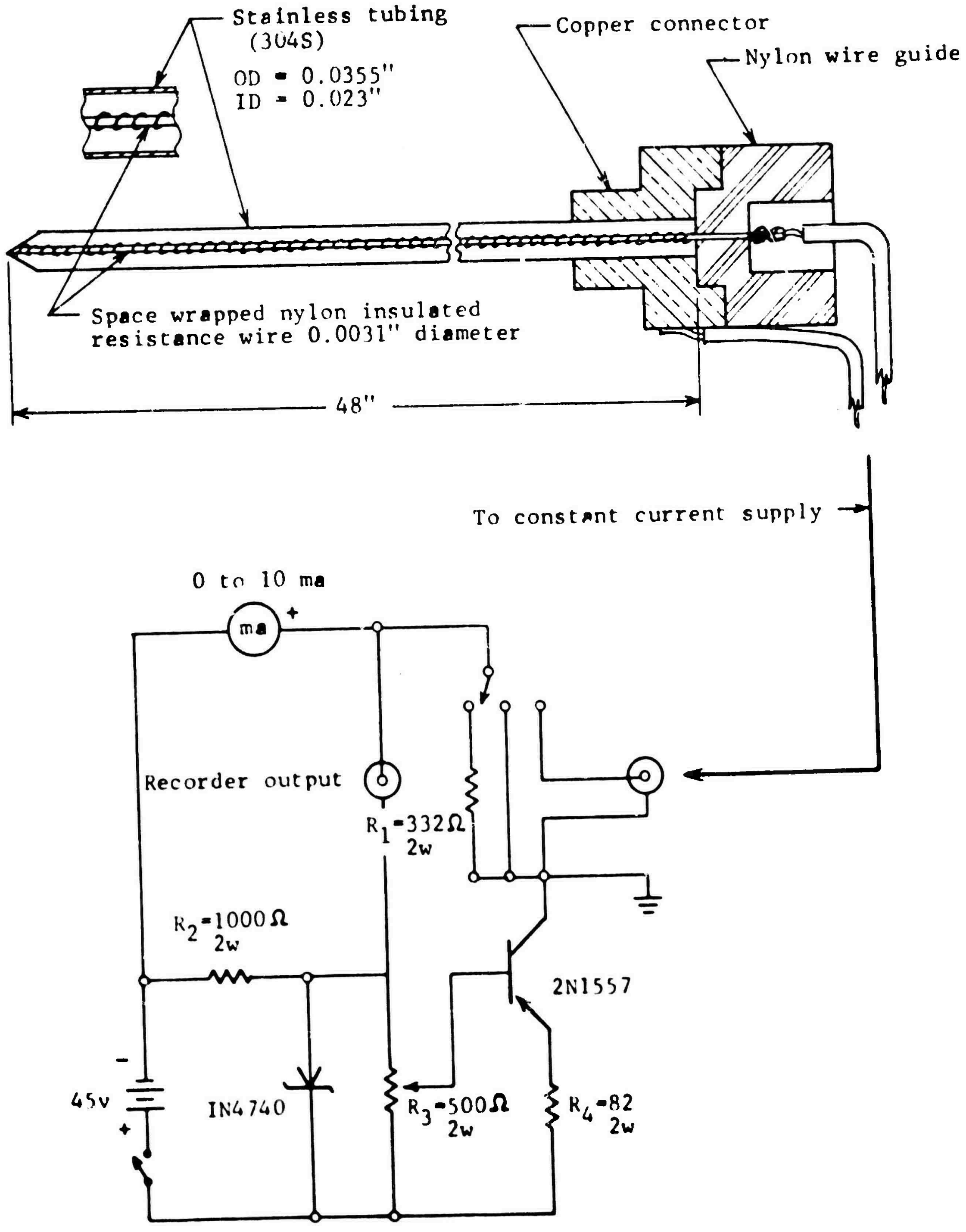


Fig a-22 Illustration of the continuous velocity probe

The resistance wire is fed through a copper crimp-on connector and, in turn, through a nylon insulated guide at the connector end. PVC insulated wires are attached to the copper connection and to the resistance wire as shown in figure a-22. Epoxy coating is used to provide a firm lead wire anchor. The resistance of the center wire should be about 272 ohm/m (83 ohm/ft) and the length of the probe should be chosen based on the required pipe length. A constant current supply is used to provide power to the probe. The power supply chassis should contain a calibration resistor, R_1, used to adjust the scale factors on the recording oscilloscope.

The second hole in the witness plate is for threading the electrical leads for the squib igniter. These leads and the velocity probe should be positioned prior to loading the pipe with sample material. The sample should be loaded in constant mass increments in order to produce a fairly constant bulk density of the desired value. Once the sample is loaded, a cloth bag containing 5 grams of black powder and one S-65 squib should be centered on the sample and the squib connected.

After the squib is connected and centered at the end of the filled pipe, the standard pipe cap is screwed down three to five turns by hand. Four turns should be set as a goal, but if the cap is still faily loose at the end of four turns, then tighten the cap until hand tight. Similarly, if the cap is extremely difficult to turn beyond three turns, it should not be forced beyond that point (no pipe wrench is to be used).

In the field, the tube transition apparatus should be positioned as shown in figure a-23 with the pipe axis horizontal. Since the pipe fragments are quite helpful in interpreting the results, the pipe should be between two mounds or in an arena order to catch the fragments.

Test Procedure

This test should be done after the critical diameter test is completed. Tube transition tests should be done in three pipe diameters greater than the critical diameter. The smallest diameter should be approximately 20 percent greater than the critical diameter. The second diameter should be double this value and the largest diameter should be four times the smallest. There is no need to conduct tests at sizes larger than the actual process vessel diameter. The pipe length must be longer than the critical length. Since the critical length is not known prior to testing, an estimate should be made based on past experience with similar materials. If an estimate cannot be made an initial length of 122 cm (4 feet) should be used. If it is found after evaluating the test results that the pipe was too short, the test must be repeated using a longer pipe section. At each pipe diameter two tests must be completed. The critical length at each diameter will be taken as the average of the two values found from the tests. It is not necessary to conduct tests in pipes longer than 120 percent of the longest actual process vessel dimension.

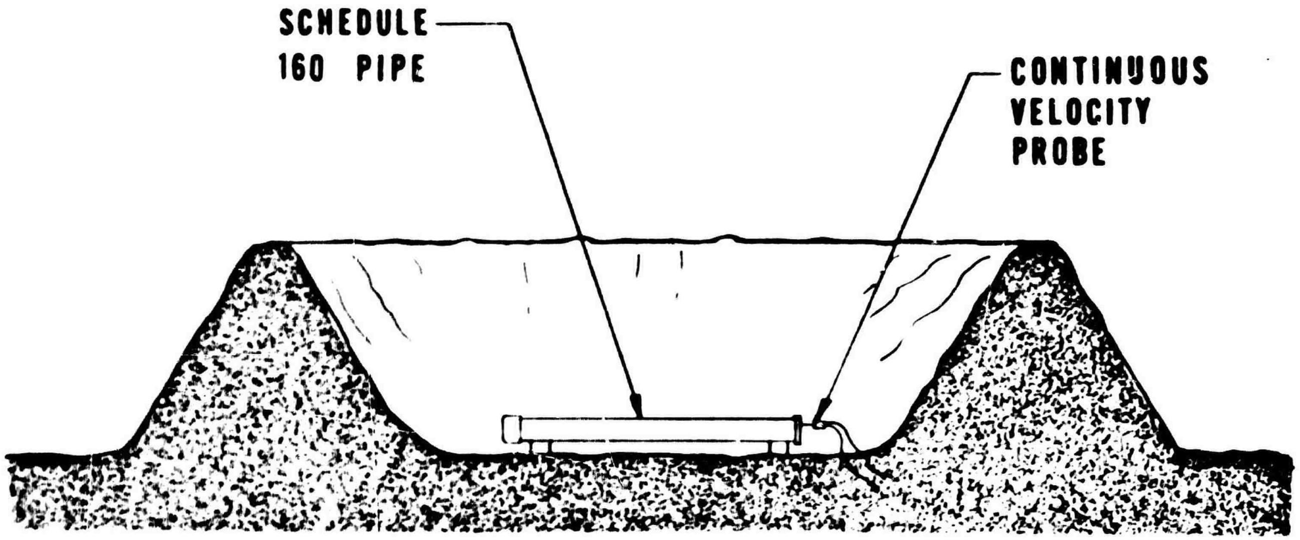


Fig a-23 Tube transition test field setup

Interpretation of Results

A test is considered successful when the following criteria are satisfied:

1. At the initiating end, the pipe cap remains intact, still screwed to a length of pipe. Some bulging of the pipe near the cap will be considered acceptable.
2. Sufficient length of pipe was available to determine critical length from the pipe remains and/or the continuous velocity probe data, i.e., it is found that the pipe was selected longer than the critical length.
3. Critical length can be determined from the continuous velocity probe record, and/or the pipe remains, i.e., the data is interpretable.

If a test is not successful, it must be repeated or not used in determining critical length.

Critical length can be determined by either of two methods. The first method is to measure the length from the pipe remains as shown in figure a-24. For an ideal test, the distance from the initiating end to the tip of each of the longest "banana peels" will be very nearly the same. The longest banana peel will generally end with a flattened tip. If the peel tip is pointed, this indicates that the failure was due to a pressure vessel explosion (large fragments) and the measurement of critical length from the pipe remains will not be reliable.

In figure a-25 an ideal continuous velocity probe record is shown. The oscilloscope trace should be triggered by a significant shortening of the probe length. Generally, triggering will occur before a stable detonation has been established. The probe will be crushed for an instant due to a high pressure region in the turbulent deflagration to detonation transition process. If the trigger is too early the oscilloscope sweep may be over by the time the detonation is established. To maximize the likelihood of obtaining a record, several channels with different sweep times should be used. The critical length as shown in figure a-25, corresponds to the voltage difference between the "probe plus leads" value and the point at which a stable detonation begins. The location of the probe's tip relative to the black powder bag must also be taken into account. Since a constant current source is used to give the probe voltage signal, voltage (V) corresponds directly to probe resistance (R) by the relation

$$R = \frac{V}{I}$$

where I is the constant current used. Length is directly proportional to resistance since the probe's resistance is a constant value per unit length.

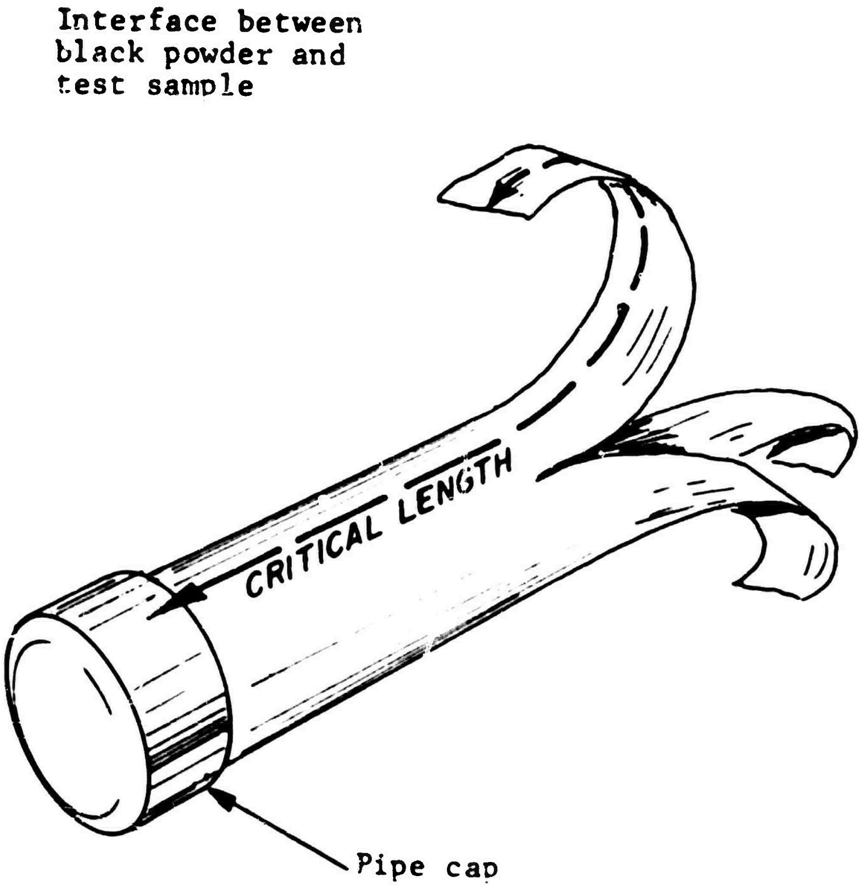


Fig a-24 Typical fragments from successful critical length test

When interpreting the results of a test, if critical length can be determined from both the pipe remains and the velocity probe record, the shorter of the two critical length results will be taken. If only one of the results is available, the available result will be acceptable.

To estimate the critical length for transition to detonation in the actual process vessel, the critical length determined for each pipe diameter should be plotted versus pipe diameter. The appropriate critical length is estimated by extrapolating the curve to the actual process vessel equivalent diameter.

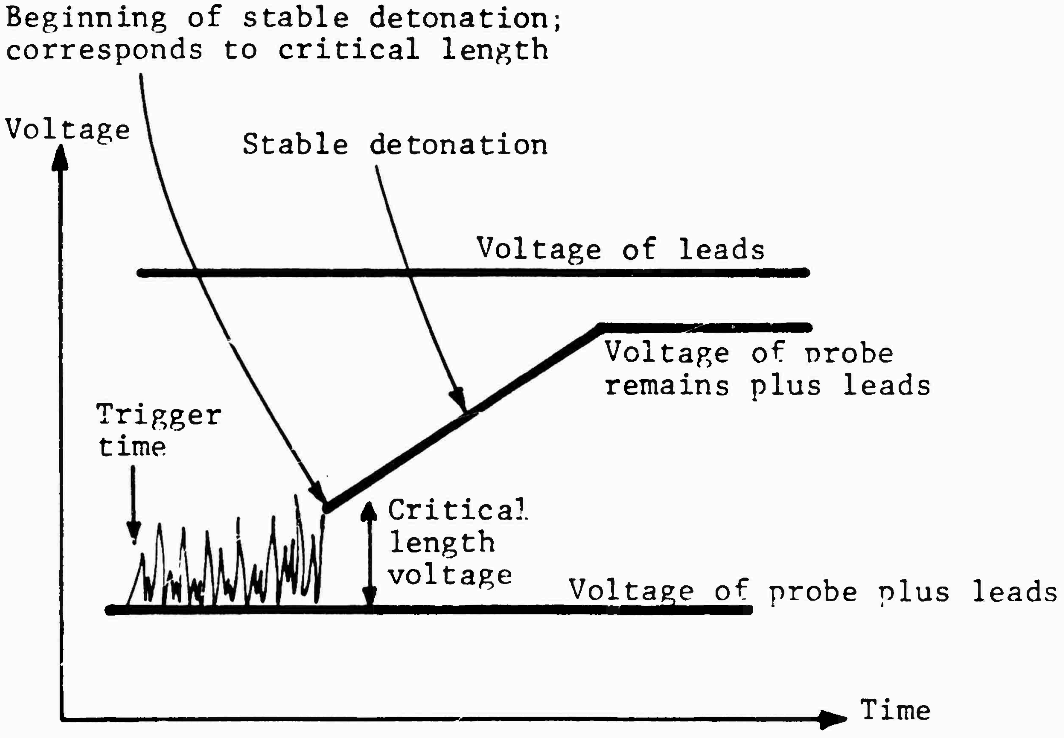


Fig a-25 Ideal record from continuous velocity probe

NOTE: Voltage corresponds to probe resistance since a constant current source is used. Probe resistance is directly proportional to probe length.

<u>Unknown Critical Length</u>

If, because of facility limitations, or for other reasons, tests cannot be conducted at large enough sizes to estimate the critical length, the worst consequence is assumed to be possible first. The mass explosion test should be done. If the TNT equivalency is found to be greater than or equal to 10 percent, the material is put into class 1.1A. If the equivalency is less than 10 percent, the potential fire hazard is judged first based on the fireball from the mass explosion test. If the material is not classified into class 1.3A based on the fireball, the mass fire test should be done as a final evaluation of the fire hazard.

CRITICAL LAYER THICKNESS TEST

Purpose

This test determines whether or not a detonation is likely to propagate in a layer of the process material of a known thickness. The critical layer thickness is the material depth in a layer configuration at which an established detonation will propagate (not die out) 50 percent of the trials. Thinner layers are less likely to propagate a detonation whereas thicker layers are more likely to propagate a detonation. In the hazards classification procedure, the critical depth is used as a criteria to help decide which effects test (mass explosion or fire spread) should be conducted. If the actual process material layer depth is equal to or greater than the critical layer thickness determined by this test procedure, the material is assumed to be capable of propagating a detonation and the layer transition test should be done next. Conversely, if the actual process material layer depth is less than the critical layer thickness, a detonation could not propagate and the fire spread test should be done.

Apparatus

The apparatus for this test is shown in figure a-26. A trough made of angle irons tack welded to 2.54 cm (1 in.) thick steel witness plates holds the sample. The sample is weighed before the test and loaded into the mold formed by the angle iron so that its bulk density is representative of the actual process bulk density. The sample should fill the mold flush with the top edge of the angle iron. A C4 explosive booster shaped as shown in the figure is used to initiate the sample. A square 1.27 cm (1/2 in.) thick steel plate with sides approximately equal in length to the trough width should be set on top of the sample at the initiating end. This is to give the detonation a chance to establish in the sample material in a confined condition and to help prevent premature dispersing of the sample material. Ten fiber optic "light pipes" should be equally spaced down the trough length as shown in figure a-26. These should lead to one (or more) photocell and be used to record reaction front velocity versus distance. The fiber optic probes should be oriented to view light produced at approximately the centerline of the trough such that stray light from reactions elsewhere is not detected. In this test, the independent variable is the layer thickness. Whenever possible, tests shall be conducted with the trough width the same as is in the actual full scale process. If the extent of the full scale layer is very large, tests can be done with the trough width equal to six times the layer thickness. The trough length, beyond the square cover plate at the initiating end, should be equal to at least six times the trough width.

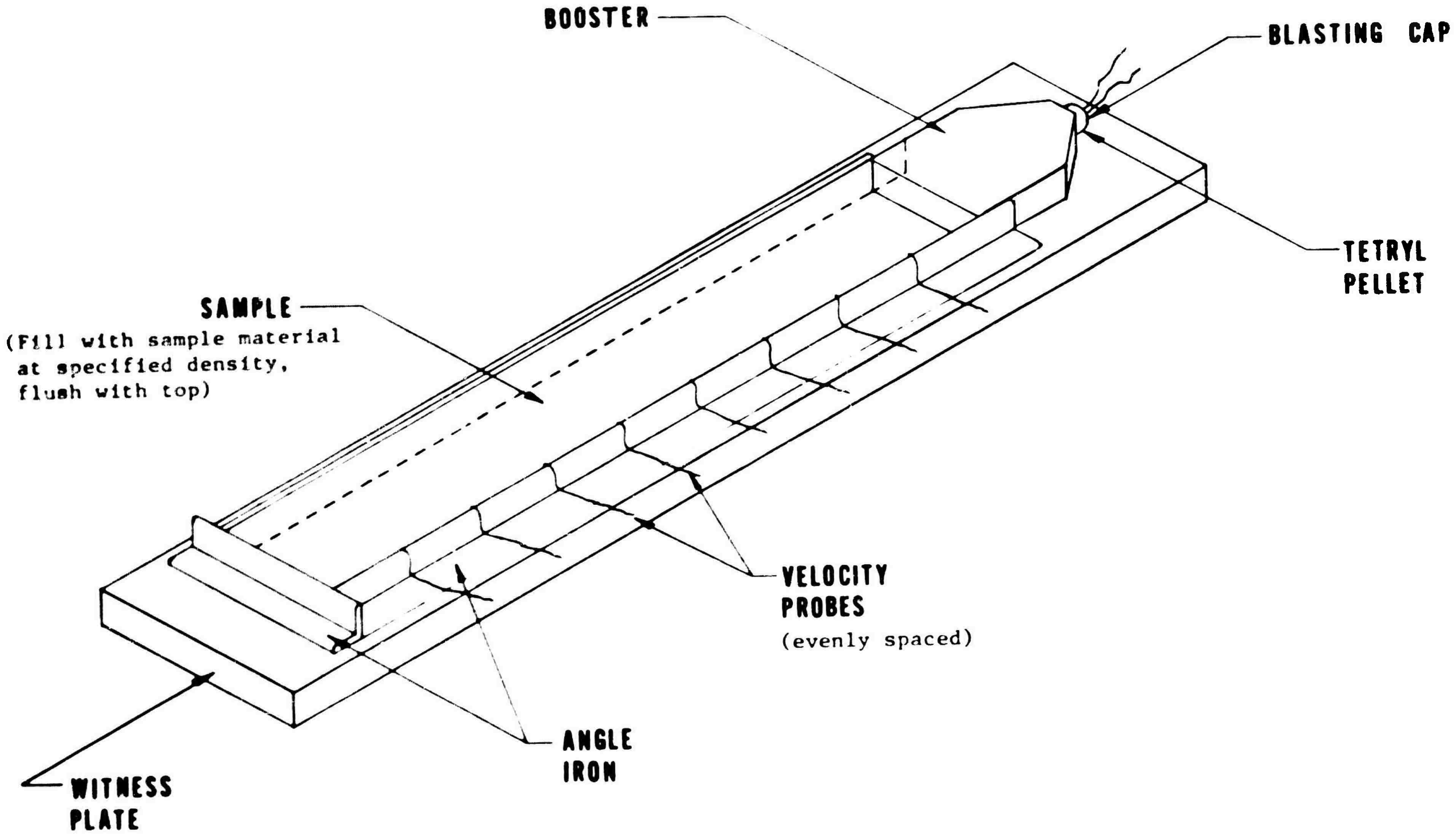


Fig a-26 Critical thickness test apparatus

Procedure and Interpretation of Results

To estimate the critical layer thickness, a Bruceton procedure as described in the critical diameter test procedure can be used, varying layer depth instead of tube diameter. This may not always be necessary in hazards classification. If it is shown that detonation will not propagate in three repeats at a layer depth 50 percent higher than the highest credible actual process layer depth, the material will be considered nondetonable (in its process configuration) and the fire spread test will be required.

Similarly, if it can be shown that the material consistently propagates a detonation (minimum of three trials required) at a layer depth of half the maximum credible layer depth, a Bruceton series is not necessary. The layer transition test will be required for such materials. For intermediate cases, a Bruceton series is required in order to estimate the layer depth at which a detonation will propagate 50 percent of the time. This layer depth will be compared to the actual process maximum credible layer depth to help specify which effects test should be done. If the critical depth is less than or equal to the actual process layer depth, the layer transition test should be done, whereas if the critical depth is greater than the actual process layer depth, the fire spread test should be accomplished.

LAYER TRANSITION TEST

Purpose

This test is required for materials which have been shown (in the critical layer thickness test) to be capable of propagating a detonation in their inprocess layer configurations. This test is to determine whether a detonation can develop from a flame ignition source in such layers.

Apparatus

The apparatus for the layer transition test is shown in figure a-27. The cross section of the test trough should be representative of the actual process container being evaluated. The layer depth and bulk density should be the maximum credible values for the process. The trough width should be the inprocess vessel width, unless the inprocess size is extremely large and impractical for testing. In that case, the trough width should be equal to six times the layer thickness. The trough structural material and wall thickness should be representative of the actual full scale vessel dimensions. This is necessary because heat transfer and confinement can be very significant influences in the test. Generally, the trough should be a minimum of 3 meters long, unless the actual process layer length is less. There is no need to conduct this test in layers which are longer than exist in the actual process. At the initiating end of the trough, a triangular extension as shown in figure a-27 should be attached. The triangular section terminates in a 1.27 cm (1/2 in.) wide slit exposing the sample to the flame of a gas burner. The gas burner should be adjusted and positioned so that the flame will wet the exposed sample at the end of the triangular trough extension. The burner should be remotely started using a solenoid valve and spark ignition system such as the one shown.

Procedure and Interpretation of Results

The trough should be instrumented with ten equally spaced fiber optic "light pipe" velocity probes (see critical diameter test description) and a continuous resistance velocity probe(see tube transition test description). The measured reaction front velocity should be plotted versus distance from the initiating end and extrapolated to the longest credible actual process layer length. If the extrapolated final velocity is representative of a detonation in any of the three trials (velocity greater than on the order of 1000 m/s), the material will be considered to be capable of transitioning into a detonation and the mass explosion test should be done. Otherwise, the fire spread test will be required. The fire spread test is essentially the same as the layer transition test except some additional instrumentation is required. The user can add the additional instrumentation to the layer transition test and obtain the required data at the same time, if he so desires.

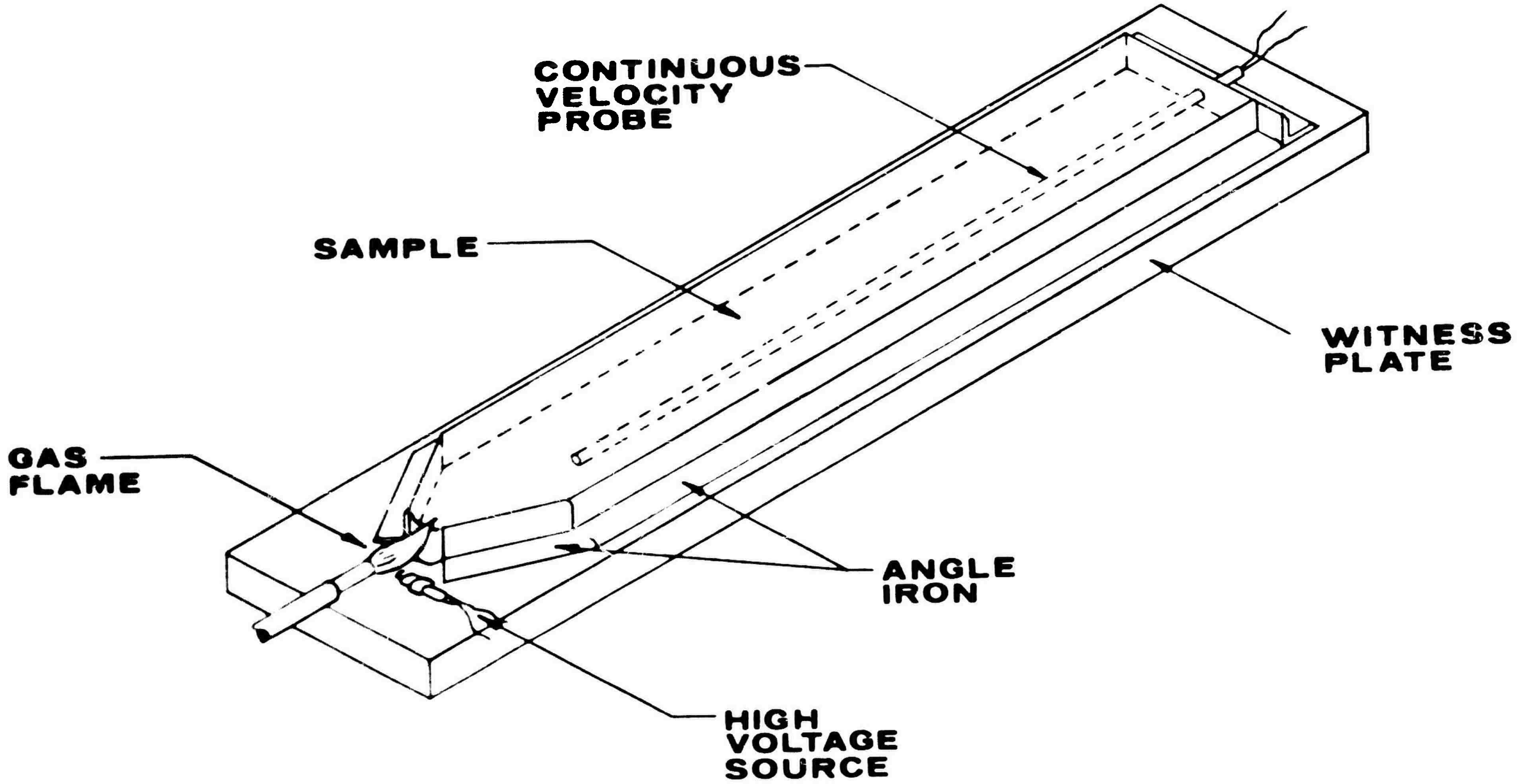


Fig a-27 Layer transition test apparatus

MASS EXPLOSION TEST

Purpose

The mass explosion test is to characterize the hazard produced by the explosion of the sample material in its process container. The effects of such an explosion will be damage by means of airblast (overpressure and impulse), fireball heating, and process vessel fragments. The mass explosion test characterizes the airblast and fireball hazard, but does not consider the fragment hazard.

Procedure, Apparatus, and Data Reduction

Figure a-28 is a typical data sheet for this type of test. The data sheet shows the test arrangement. The sample is loaded into a hemispherical shell with a 0.159 cm (1/16 in.) thick wall. The sample should be approximately its inprocess bulk density. Loading is accomplished by (1) pouring a weighed out quantity of the sample into the hemisphere, (2) pushing a void for the booster into the sample using a hemispherical mold of the proper diameter, (3) positioning the booster and thin masonite sheet onto the steel hemisphere, and (4) taping the masonite to the steel shell. It should be noted that well established geometric and confinement scaling techniques exist and can be used to model the actual process configuration. This will result in a more realistic characterization of the blast effects and is naturally also acceptable for hazards classification. The assembly is placed on a 2.54 cm (1 in.) thick steel witness plate in the field at the intersection of two perpendicular strings of pressure transducers. The pressure transducers (six transducers per string) should be positioned to measure overpressure versus time at scaled distances[*] from about 1 m/kg$^{1/3}$ to about 12 m/kg$^{1/3}$. Fastax film coverage is used to record the maximum fireball radius and the time from ignition to obscuration or significant cooling of the fireball. Slug calorimeters are used to measure the total heat pulse radiated from the fireball.

Tests should be accomplished using at least three sample masses and three booster sizes to assure that the tests are done with sample mass and booster percent both large enough to no longer influence the scaled explosive output. It is suggested that tests be done at approximately 35 kg (75 lb), 23 kg (50 lb) and 5 kg (11 lb) with booster percents of 1, 2, and 5 percent. If sufficient sample mass and/or booster percents have not been used, larger sample and/or booster sizes will be required.

---

[*] Scaled distance is equal to the distance between the pressure transducer and the center of the hemisphere divided by the sample mass to the one-third power.

Test Number ____________

Sample Material ____________

Sample Bulkности ____________

Hemisphere Diameter ____________

Hemisphere Wall ____________

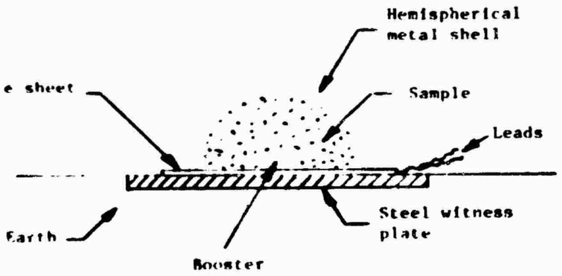
Sample Mass ____________

Booster Mass ____________

Ambient Temperature ____________

Barometric Pressure ____________

Test Arrangement



Airblast Results

Gage	R(ft)	$\frac{ft}{lb^{1/3}}$	Const $\frac{V_P}{K\cdot M}$ or $\frac{V_P}{K\cdot CA}$	$\frac{D_T(t)}{D_c}$	P(t) = Const $\frac{D_T(t)}{D_c}$	Const $\frac{V_T\cdot RC}{KMA_I}$ or $\frac{V_T\cdot RC}{K\cdot CA\cdot A_I}$	$\frac{D_T(t)}{D_c}$	I(t) = Const $\frac{D_T(t)}{D_c}$ x 10^3	Notes
1N									
2N									
3N									
4N									
5N									
6N									
1E									
2F									
3F									
4E									
5F									
6F									

Fireball Maximum Diameter = ____________ ft

Comments:

Slug Calorimeter Results

Gage Number	R(ft)	Total Heat Density $(\frac{Btu}{ft^2})$	Maximum Temperature Rise (°F)	Black Powder Response

Fig a-28 Mass explosion test data sheet

Capabilities for conducting mass explosion (or TNT equivalency) testing exist at many test facilities. For users not familiar with the basic techniques, the equipment and procedures used for airblast measurement are described at the end of this test procedure.

Interpretation of Results

If either the pressure or impulse TNT equivalency is found to be above 10 percent, the material is class 1.1A. If the TNT equivalencies are both less, the fire hazard is based on the explosive fireball. The only exception to this is for materials with unknown critical dimensions ("CD unknown" or "CL unknown"). In these cases, the mass fire test should be done to characterize the fire hazard.

To characterize the fire hazard from the mass explosion test, the first criteria is whether or not the maximum fireball radius extrapolated for the full scale vessel exceeds 3 m. From Fastax film coverage the maximum horizontal extent of the visible fireball is measured for each test. This fireball radius is plotted versus sample mass to the 1/3 power. The best fit slope of the curve is the coefficient B_1 in the scaling relation

$$R = B_1 W^{1/3}$$

Using this equation, fireball radius is extrapolated to the full scale process material mass. If this estimated fireball radius is greater than or equal to 3 m, the material is classified based on the emitted energy pulse at 3 m. Maximum fireball duration τ is scaled in an identical manner using the equation

$$\tau = B_2 W^{1/3}$$

The energy per unit area impinging on a target a distance x from the center of the fireball can be obtained from the "mass explosion" test data using the equation:

$$q = C \frac{m}{x^2}$$

where m is the mass of material in the full scale process vessel, x is the distance from the explosive fireball (center) to the target surface and C is a coefficient determined from the test data as follows:

For each data point collected, the coefficient C is computed from

$$C = \frac{q x^2}{m}$$

where q is the measured energy per unit area at the calorimeter, x is the calorimeter distance, and m is the sample mass. The coefficient C is plotted versus sample mass and by extrapolation using a curve fit, the value of C at the full scale process vessel material mass is determined.

The criteria for classifying the material's fire hazard as class 1.3A is that the energy per unit area at 3 m from the center of the fireball be greater than or equal to

$$q_c = \xi\sqrt{\tau}$$

where τ is the maximum fireball duration scaled to the full scale value, and ξ is a parameter which characterizes the target. If the most vulnerable target is not known, a value of 7.71×10^6 j/m^2-s$^{1/2}$ will be used. If the target is well known, ξ can be computed from the following equation.

$$\xi = \frac{\Delta T_c \sqrt{\pi k \rho c'}}{2}$$

where ΔT_c is the critical surface temperature rise of the target for a very short thermal pulse for causing ignition or damage, k is the target thermal conductivity, ρ is the target mass density, and c' is the target specific heat.

If the estimated full scale critical energy per unit area is not exceeded, the material is class 1.4. Otherwise, the material is class 1.3A, mass fire hazard.

BACKGROUND DETAILS
FOR MASS EXPLOSION TEST

Pressure and Impulse Measuring Systems

In this section a typical pressure and impulse measuring system for mass explosion testing is described. Any equivalent system is acceptable for this test.

Pressure time functions are monitored at locations as previously described. The pressure-time signals are integrated to produce impulse time functions. Data signals are recorded on magnetic tape and reproduced on an oscillograph recorder. The following subsections contain a description of the instrumentation equipment, calibration technique and computational procedure.

Pressure Measuring Systems

Pressure-time functions are measured as the shock wave traverses the instrumentation stations along the blast lines. The shock wave is characterized by measuring station, peak overpressure, positive phase duration, and positive phase impulse.

At measuring stations near the charge or test item, the pressure time profile is characterized by high peak overpressures and short positive durations. Piezoelectric type transducers are used at those locations to provide the required dynamic pressure range and high frequency response. Conversely, the far field pressure time profiles are characterized by low overpressure and relatively long positive durations. Capacitance type transducers are used at these locations to provide the required resolution and DC response.

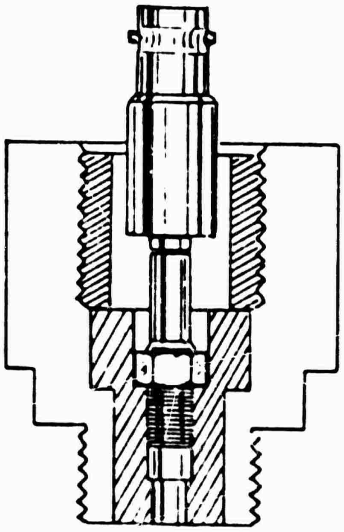
The piezoelectric transducers employ quartz crystal as their sensing element. Two types of units may be employed: the PCB Piezoelectronics Inc. (PCB) type 113A24 and the Kistler Instrument Corp (KIC) type 603A. The PCB type 113A24 has a built-in amplifier. The pressure range is 6895 kPa (1000 psi) and the 603A is 20,684 kPa (3000 psi). In the present configuration a voltage-follower amplifier, PCB 401A, is used with the unit. The addition of the voltage-follower provides operational benefits; it does however limit the pressure range of the system to about 2413 kPa (350 psi). The rise time for the system is 1 sec.

A constant current power supply (PCB series 483A) is used to supply power to the built-in amplifiers. The signal output is connected to the cal-input of a KIC model 566 charge amplifier. The charge amplifier provides a convenient means for setting the system output levels for the various test requirements.

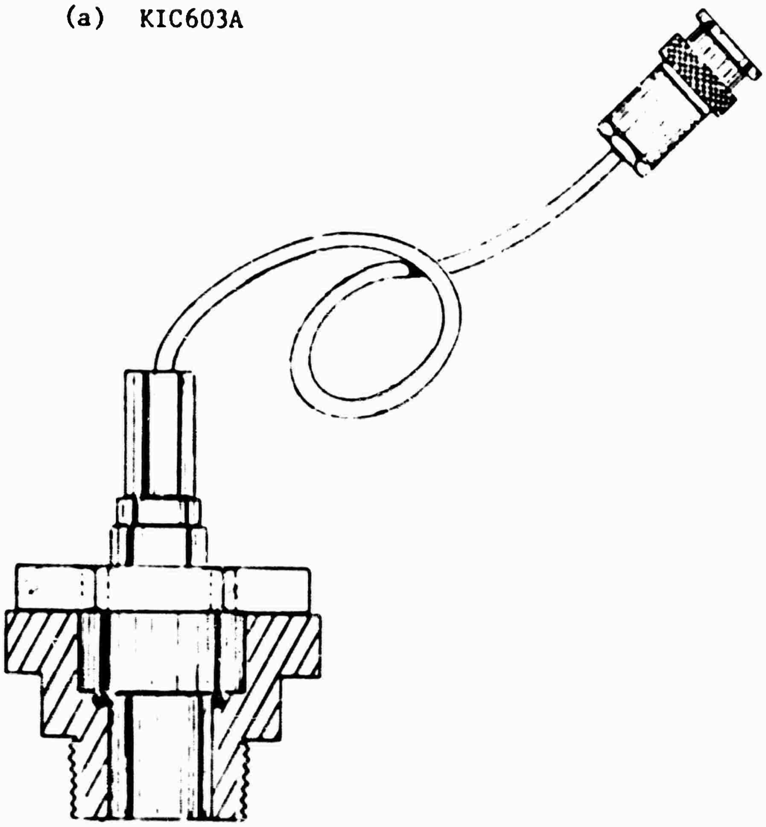
The PRP measuring system employs amplitude modulated r-f carrier techniques. The diaphragm of the transducer, in conjunction with an insulated stationary electrode, forms an electrical capacitor. The pressure to be measured is applied to the diaphragm, causing a change in capacitance proportional to the applied pressure. The transducer capacitance and a built-in conductance form a tuned radiofrequency circuit. The tuned circuit is line-coupled, by means of a low impedance cable to a Dynagage system consisting of an oscillator-detector circuit and a cathode-follower amplifier. The changes in capacitance produce changes in the diode detector impedance, and thereby produce a signal voltage proportional to the applied pressure.

Transducer Mounting Adapters

The transducer mounting adapters are illustrated in figure a-29. The piezoelectric transducers are installed in electrical insulated mounting adapters. Teflon seal rings are placed around the small diameter of the transducer to provide a pressure seal. The transducer is positioned such that its diaphragm is flush mounted with the face of the mounting adapter. Once assembled, the transducer is enclosed within the mounting adapter by using RTV-615 silicone potting compound.



(a) KIC603A



(b) PRP752A

Fig a-29 Transducer mounting adapter for the KIC603A and PRP752A

The dimensions of the mounting adapters used for these gages are identical to those designed for the PRP type 752A transducers. Accordingly, transducer assignments and subsequent changes at the various gage locations can be implemented in a timely manner.

The PRP type 752A transducers are to be removed from their water-cooled flame shields and placed in an electrical insulated mounting adapter. The adapter is designed to provide flush mounting of the diaphragm. The electrical insulation material is used to break ground loops, thus reducing interference caused by stray pickup and intercarrier beats.

Calibration Procedures

The purpose of the calibration series is to establish the sensitivity factor for each pressure measuring system. The mounted transducer, transmission cable and signal conditioner are calibrated as a system.

The transducers, in turn, are placed in the expansion chamber of the calibration fixture. The calibration fixture consists of an expansion chamber with a volume less than 33 cu cm (2 cu inches) and a compression chamber with a volume of 6227 cu cm (380 cu inches) operated solenoid valve. The pressure in the compression chamber is monitored with a pressure transfer standard (Seegers 55-2455A) for calibration pressures in the range 138 to 2068 kPa (20 to 300 psig). A mercury manometer is used to monitor pressures below this range.

When the solenoid valve is actuated, the transducer is subjected to a pressure rise. The pressure rise time in the expansion chamber is less than 2 msec. The applied pressure causes a voltage to be generated at the output of the signal conditioner.

The piezoelectric transducer sensitivity factor (K_i) is the output voltage of the charge amplifier (V), divided by the charge amplifier setting (S) and the applied pressure P.

$$K_i = \frac{V}{S\text{-}P} \quad \text{(pcb/psi)}$$

Similarly, the sensitivity factor for the Photocon Research systems is the output voltage from the Dynagage (V) divided by the applied pressure P.

$$K_i = \frac{V}{P} \quad \text{(volt/psi)}$$

The systems are calibrated at five pressure levels in the pressure range of interest. The quantity $\overline{K}$ is the arithmetic mean of the value of K_i as determined for each calibration pressure level.

The rms deviation (r) for $\bar{K}$ is determined by the relationship

$$r = \frac{1}{N}\left[\sum_{i=1}^{N}(\bar{K}-K_1)^2\right]^{1/2}$$

The percent deviation is determined by the relationship:

$$r_{100} = \frac{2r}{\bar{K}} \times 100 \ (\%)$$

Using a factor of 2 in the numerator of the equation above gives a confidence level of 95.5 percent.

The calibration data are used to access the time constants of the piezoelectric transducers. A storage oscilloscope is used to record a pressure time trace for a calibration test. The time constant (RC) is evaluated at a time (t) by the relationship

$$RC = -t/\ln \frac{V_t}{V_o}$$

where V_t and V_o are the voltage levels at time t and t_o respectively.

Signal Conditioning and Recording Instruments

The data signals from the pressure measuring systems are fed to the calibration monitor unit. This unit is used to monitor each gage line and assess the pretest condition of the pressure measuring systems. This unit is also used to provide an electrical calibration signal which is recorded on each data track immediately preceding each test run. The electrical calibration signal is a voltage simulation of a predetermined pressure or impulse level. This signal is used in data reduction as discussed in a later section.

Hewlett Packard (HP) model 8875A differential amplifiers are used to provide a voltage gain and thus condition the signal for magnetic tape recording. The pressure data signals are recorded on a Sangamo Sabre III instrumentation tape recorder. This unit is equipped with 13 FM recording tracks for data recording, and a single channel of direct record for time base signals. The tape recorder conforms to specifications for the IRIG wideband group 1 (Document 106-71).

Pressure Impulse Measurements

The pressure impulse is defined as the area under the pressure time history

$$I(t) = \int_{t_o}^{t} P(t)\,dt$$

where P is the pressure and t is the time.

The signal voltage at the output of the model 8875A amplifier is an electrical analog of the pressure-time history. This signal is used as input to a Tektronix type-O operational amplifier, where the electrical integration is performed. The integrated signal is amplified and in turn, recorded on a CP100 magnetic tape recorder. This recorder conforms to specifications for the IRIG intermediate band.

Data Reproduction

Oscillograph reproductions of the magnetic tape recordings are made by employing Consolidated Electrodynamics Corp. (CEC) type 1-172 driver amplifiers to drive a CEC type 5-124 recording oscillograph. The oscillograph is equipped with CEC type 7-363 galvanometers.

The pressure data are recorded at a tape speed of 60 ips and reproduced at a tape speed of 1-7/8 ips, resulting in a frequency division of 32. The oscillograph paper speed is 32 ips. For these conditions, the oscillograph has a horizontal resolution of 976 sec/in. and an effective frequency response from DC to 40 kHz, referred to real time.

The pressure impulse data are also recorded at a tape speed of 60 ips to produce a tape speed of 1-7/8. The effective frequency response is L to 30 kHz, referred to real time.

A simplified block diagram of the pressure and impulse instrumentation system is shown as figure a-30. Two data channels are shown: one to illustrate the piezoelectric measuring system and the second to show the capacitance type pressure measuring system.

Computational Procedure

This subsection contains the equations used to determine the relationship between signal amplitude on the oscillograph recording and applied pressure and impulse. The symbols and units are:

$p(t)$ = applied pressure (P_a)

K = sensitivity factor of pressure sensing system (volts/Pa or Pcb/Pa)

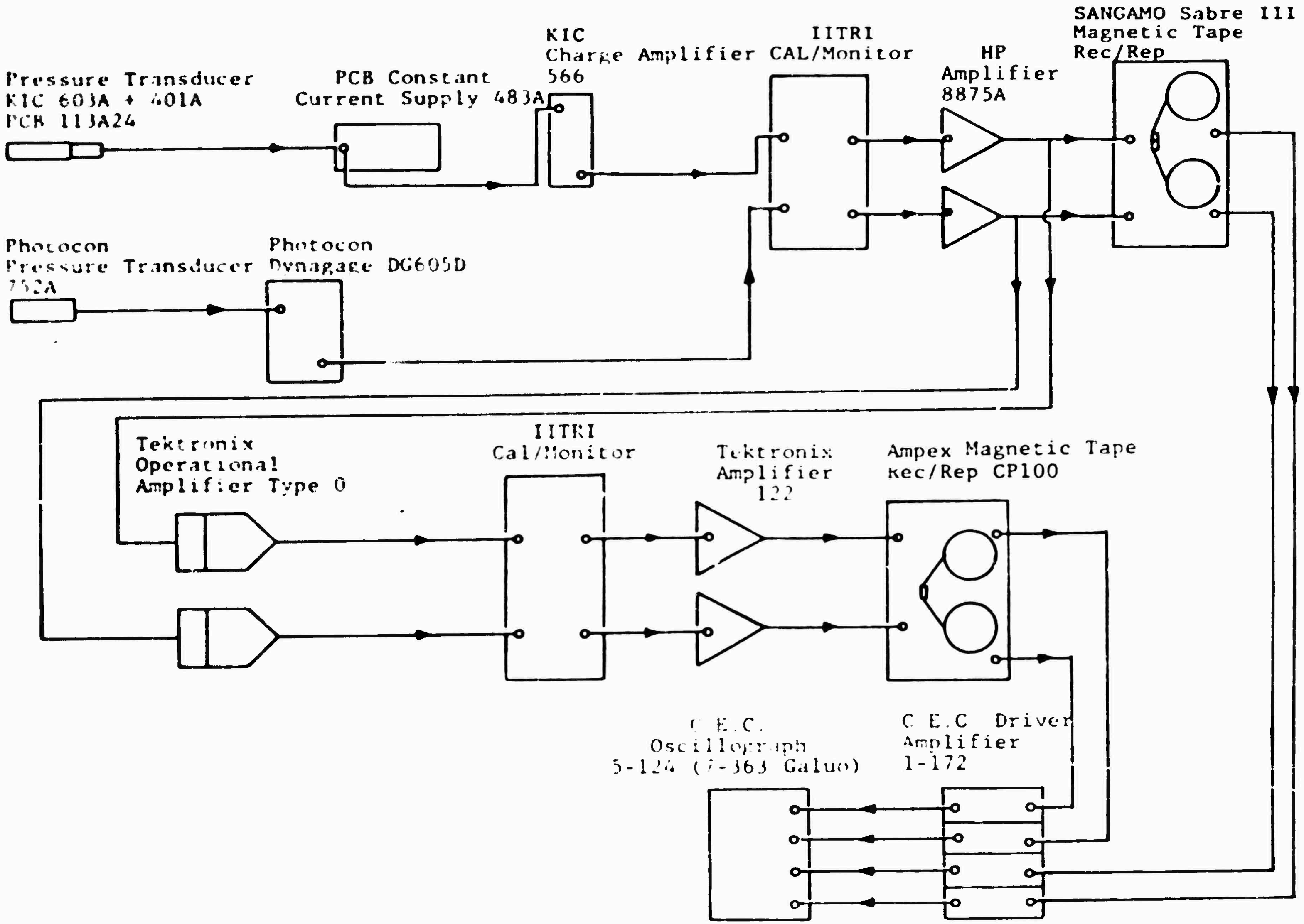


Fig a-30 Block diagram of record/reproduce instrumentation

$I(t)$ = pressure impulse (Pa-sec)

t_o = initial time (sec)

t_d = time of positive duration of the pressure pulse (sec)

E_{c1} = calibration voltage for the pressure channel (volts)

E_{c2} = calibration voltage for the impulse channel (volts)

D_{c1} = deflection of trace due to pressure calibration signal (cm)

D_{c2} = deflection of trace due to impulse calibration signal (cm)

$D_{t1}(t)$ = deflection of trace due to the applied pressure (cm)

$D_{t2}(t)$ = deflection of trace due to the applied impulse (cm)

A_1 = voltage gain of Sanborn 8875A amplifier

A_2 = voltage gain of Tektronix 122 amplifier

A_s = combined voltage gain of record/reproduce system and galvanometer sensitivity (cm/volt)

Figure a-31 shows an illustration of an oscillograph print of the experimental data. Those terms taken from the experimental data and used in the data reduction equations are identified.

The peak overpressure and maximum impulse are the important parameters determined from the recorded data. The general expression for determining P(t) is determined in the following manner.

The deflection of the oscillograph trace due to the calibration signal is

$$D_{c1} = E_{c1}A_1A_s$$

The deflection of the trace due to the applied pressure is

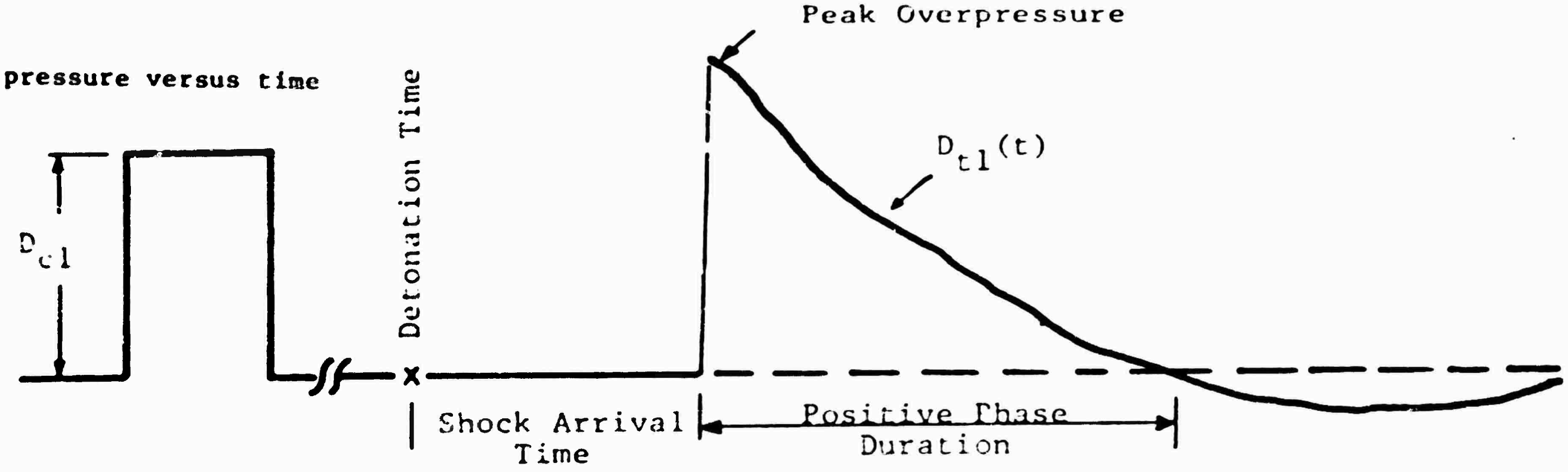
$$D_{t1}(t) = KP(t)\ A_1A_s$$

Combining these equations we obtain:

$$P(t) = \frac{E_{c1}D_{t1}(t)}{K\ D_{c1}}$$

The peak overpressure is the pressure determination at $t = t_o$ as indicated in figure a-31.

(a) pressure versus time



(b) impulse versus time

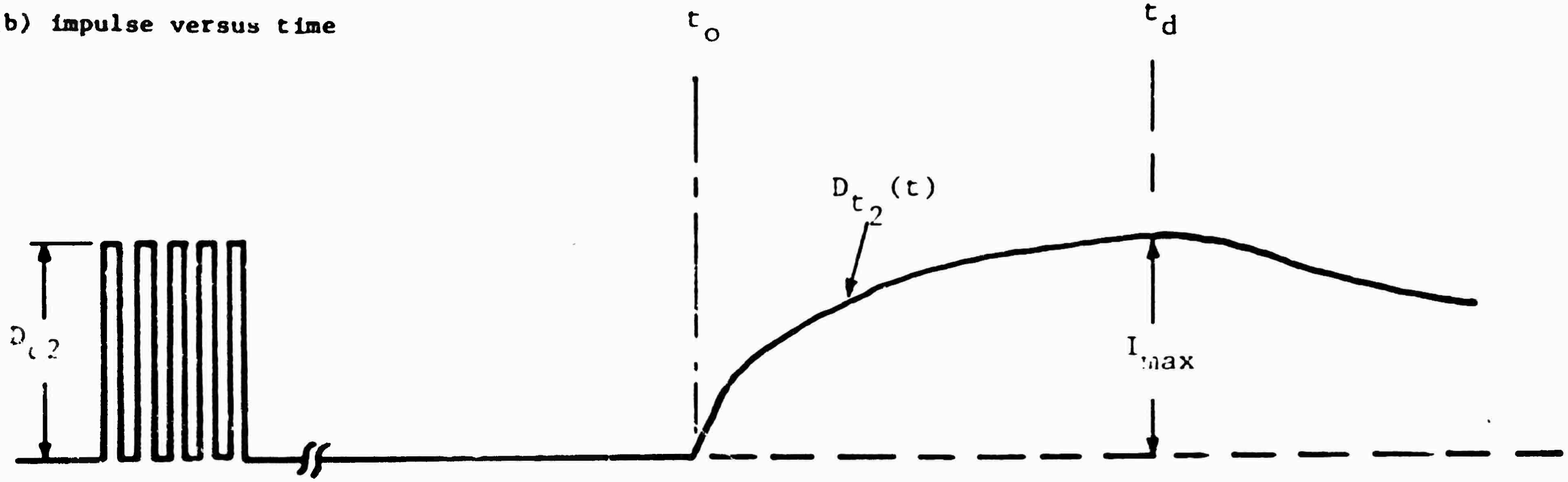


Fig a-31 Illustration of oscillograph print of pressure versus time and impulse versus time

The pressure impulse is determined by an analog integration of the pressure-time function. Referring to the block diagram (Fig a-30), and assuming a validity of the ideal gain of the generalized operational amplifier

$$\frac{E_o}{E_1} = -\frac{Z_o}{Z_i}$$

where E_i amd E_o are the input and output voltages of the operational amplifier respectively, and Z_i and Z_o are the input and feedback impedances.

For the integrator configuration

$$Z_i = R \text{ and } Z_o = \frac{1}{Cp}$$

where p is the operator, d/dt

The gain expression in terms of the circuit parameters is

$$E_o = -\frac{Z_o}{Z_i} \qquad E_i = -\frac{E_i}{RCp} = -\frac{1}{RC}\int E_i \, dt$$

Writing the gain expression in terms of the applied pressure

$$E_o = \frac{A_1 K}{RC}\int_{t_o}^{t} P(t)\, dt$$

Noting that $E_o A_2 A_s = D_{t2}(t)$

$$D_{t2}(t) = \frac{A_1 A_2 A_s K}{RC}\int_{t_o}^{t} P(t)\, dt = \frac{A_1 A_2 A_s K}{RC}\; I(t)$$

The deflection of the trace due to the calibration voltage is

$$D_{c2} = E_{c2} A_2 A_s$$

Combining the expressions for $D_{t2}(t)$ and D_{c2}

$$I(t) = \frac{RC\, E_{c2}\, D_{t2}(t)}{A_1\, K\, D_{c2}}$$

The maximum pressure impulse is evaluated at the end of the positive phase duration, t_d, of the pressure pulse.

$$I_{max} = \frac{RC\ E_{c2}\ D_{t2}(t_d)}{A_1\ K\ D_{c2}}$$

TNT Equivalency Calculation Procedure

Computational Procedure

The computational procedure used to obtain TNT equivalencies is discussed in this section. TNT equivalency for pressure is defined as the ratio of TNT weight to test explosive weight that will give the same peak overpressure at the same radial distance from each charge. Similarly, the TNT equivalency for impulse is defined as the ratio of charge weights that will give the same positive impulse at the same radial distances. The standard TNT charge used for comparison is a hemisphere placed on a rigid ground surface. Since the booster used to detonate the test explosive, propellant, or pyrotechnic may be of the order of 10 percent of the test material weight it is necessary to account for its contribution to the explosive output (i.e., peak pressure and impulse).

The symbols used in this discussion are:

W	weight, kg
R	radial distance from charge, m
$\lambda = R/W^{1/3}$	scaled distance, m/kg
P	peak overpressures, kPa
I	positive impulse, kPa-msec
E	TNT equivalency, percent

These subscripts and superscripts are self-explanatory when applied to the above symbols:

S	test sample
B	booster
TNT	TNT explosive
I	impulse
P	pressure
*	quantity is not adjusted for booster weight
TOT	total charge weight, booster plus sample

The pressure equivalency is determined by first measuring the quantities W_S, R, and P_{SB}, where P_{SB} is the peak pressure measured when the sample is detonated with a C-4 booster. It includes an energy contribution from both C-4 and sample.

One must first approximate an equivalent booster weight, in terms of the charge sample weight, so that its weight can be included in the total charge weight. The approximation is found by obtaining λ_{TNT}, from figure a-32, for $P_{SB} = P_{TNT}$.

The first approximation for TNT pressure equivalency is then

$$E_p^* - W_{TNT}/W_S = (\lambda_S/\lambda_{TNT})^3$$

where

$$\lambda_S = R/W_S^{1/3}$$

and

$$\lambda_{TNT} = R/W_{TNT}^{1/3}$$

Since the pressures are to be equal at the same radial distance, the R's cancel in the above equation. One applies this approximated equivalency E_p^*, to the weight of the booster to obtain the total charge weight.

$$W_{TOT} = W_S + (1/E_p^*)\, W_B(1.25)$$

A factor of 1.25 is applied to the C-4 booster weight to obtain its equivalent TNT weight.

A new λ is now computed from

$$\lambda_{TOT} = R/W_{TOT}^{1/3}$$

and a corrected pressure TNT equivalency is computed.

$$E_p = W_{TNT}/W_{TOT} = (\lambda_{TOT}/\lambda_{TNT})^3$$

The P subscript indicates a scaled distance for pressure and is computed from the revised sample weight. This iterative process can be repeated using the revised value of E_p to recompute the weight of the booster in terms of the sample weight, etc. However, the second iteration has a small effect on equivalency.

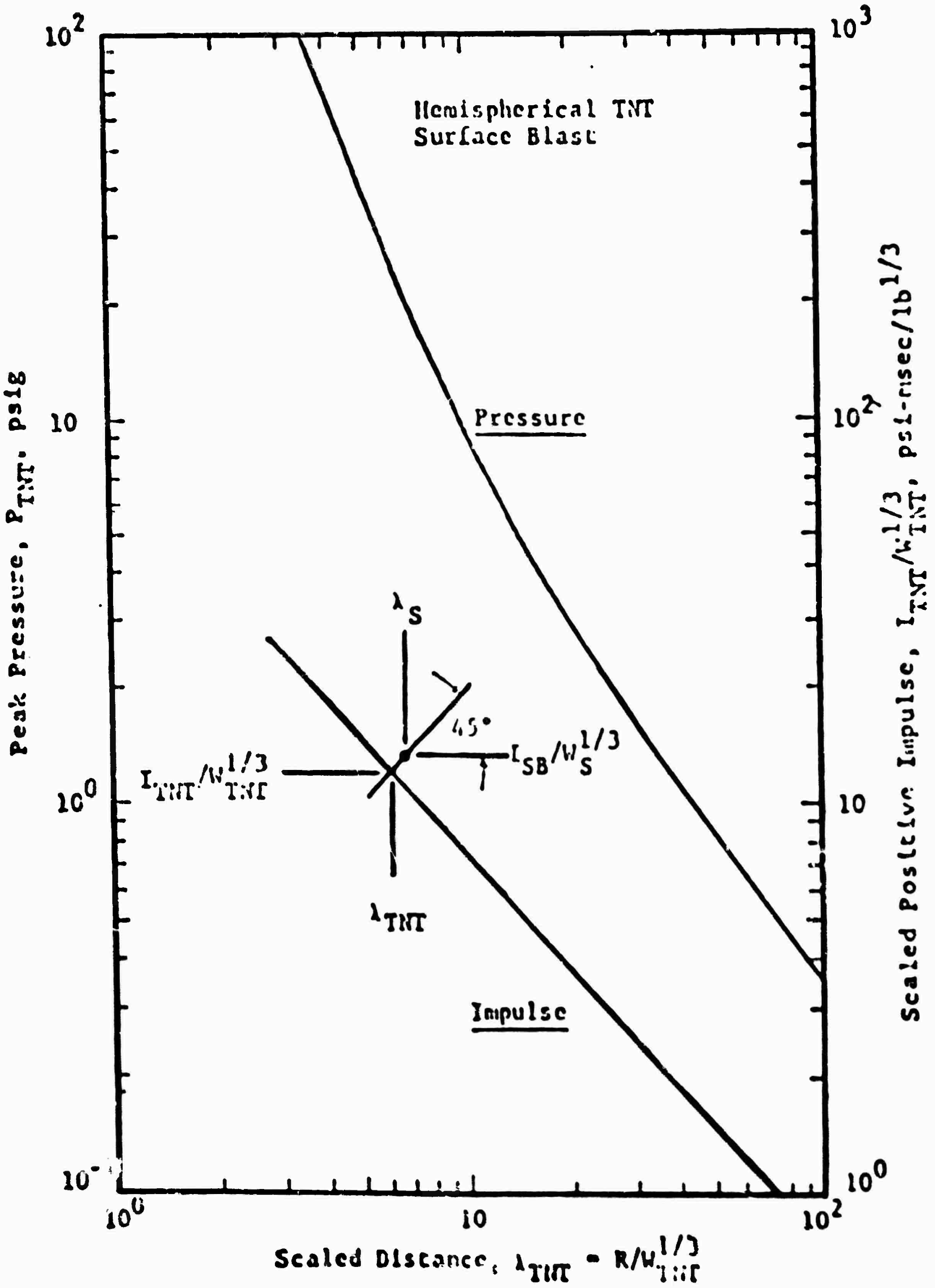


Fig a-32 TNT pressure and impulse

Impulse equivalency is determined first by measuring W_S, R, and I_{SB}, where I_{SB} is the impulse measured when the sample charge was detonated with a C-4 booster. One must first approximate an equivalent booster weight, in terms of the charge sample weight. The approximation is found by locating the data point $I_{SB}/W_B^{1/3}$: λ_S on figure a-32. A 45 degree line is drawn through this data point to intersect with the TNT impulse curve. Values of λ_{TNT} and $I_{TNT}/W_{TNT}^{1/3}$ are read at the intersection of the two straight lines. These values give the equivalent TNT weight for equal impulses and radial distances.

At the data point $I_{SB}/W_S^{1/3}$ and λ_S let

$$a_S = I_{SB}/W_S^{1/3} \quad \text{or} \quad I_{SB} = a_S W_S^{1/3}$$

and

$$\lambda_S = R/W_S^{1/3} \quad \text{or} \quad R = \lambda_S W_S^{1/3}$$

For equal impulses

$$I_{SB} = I_{TNT}$$

or

$$a_S W_S^{1/3} = a_{TNT} W_{TNT}^{1/3}$$

and for equal radial distances

$$\lambda_S W_S^{1/3} = \lambda_{TNT} W_{TNT}^{1/3}$$

Divide these two equations and get

$$\frac{a_S}{a_{TNT}} = \frac{\lambda_S}{\lambda_{TNT}}$$

Take the log of the above equation

$$\log a_S - \log a_{TNT} = \log \lambda_S - \log \lambda_{TNT}$$

This equation shows that a 45 degree construction line on a log-log plot will intersect the impulse curve and data point in such a way as to satisfy the conditions of equal positive impulses at the same radial distance.

The first approximation for TNT impulse equivalency is

$$E_I^* = W_{TNT}/W_S$$

$$E_I^* = (I_{SB}/W_S^{1/3})^3/(I_{TNT}/W_{TNT}^{1/3})^3 = \frac{1/W_S}{1/W_{TNT}}$$

Since $I_{SB} = I_{TNT}$ they cancel in the above equation.

One applies this approximated equivalency, E_I^*, to the weight of the booster to obtain the total charge weight

$$W_{TOT} = W_S + (1/E_I^*)\ W_B(1.25)$$

A new scaled distance

$$\lambda_{TOT} = R/W_{TOT}^{1/3}$$

and scaled impulse is then computed as

$$I_{SB}/W_{TOT}^{1/3}$$

This data point is now located on figure a-32 and new $I_{TNT}/W_{TNT}^{1/3}$ and λ_{TNT} values are determined from the 45 degree line intersection method described.

The correct impulse equivalency then becomes

$$E_I = W_{TNT}/W_{TOT}$$

$$E_I = (I_{SB}/W_{TOT}^{1/3})^3/(I_{TNT}/W_{TNT}^{1/3})^3$$

Computerized Calculations

The TNT equivalencies of the explosive material are generally determined by use of a computer program. The first step in the procedure is to fit a curve to the test data utilizing a manual curve fit method. That is, a curve is drawn through the data points that are most representative of the characteristic trend. To do this, the pressures with their corresponding gage distances (and, similarly, impulses with their distances) are entered into the program as input data. Scaled distances are then obtained by dividing the gage distances by the cube root of the charge weight. Where the input consists of experimental data for more than one test conducted under identical conditions, the pressure and impulse values are generally averaged before the curve fit is performed. Impulse input is converted to scaled impulse by dividing by the cube root of the charge weight. This is performed before averaging or curve fitting is done.

Having chosen the curve which best describes the test data, pressure and impulse values with their corresponding gage distances are entered into the program along with the appropriate curve coefficient. The TNT equivalence is determined twice, once using points from the fitted curve at scaled distances corresponding to the gage locations, and once using the actual data point. This is done for both pressure and impulse data.

In the program, the TNT pressure and impulse curves versus scaled distance appear as polynomial expressions. To determine the pressure equivalency, the TNT scaled distance at a pressure equal to the test pressure is determined from this equation. The TNT equivalency at each pressure data point is computed as the cube of the ratio of the scaled distance of the test data to the TNT scaled distance.

A correction is made to the equivalency calculation to include the weight of the booster in the total weight. The TNT equivalency is then recomputed on the basis of the corrected weight. This is an iterative process and continued until the change in the ratio of the scaled distance to the TNT scaled distance is negligible.

A similar procedure is followed for impulse data. Since scaled impulse is used rather than actual impulse, a correction in the total weight of the explosive to account for the booster weight involves making corrections to the scaled impulse as well as the scaled distance.

The computer output for the pressure tests includes scaled distance, corrected scaled distance, pressure, total weight, and TNT equivalency at each gage location. Output based on raw data includes scaled distance, corrected scaled distance, input pressure, TNT equivalency, and gage distance at each data point. The output for the impulse tests is similar except that scaled impulse and corrected impulse are included.

MASS FIRE TEST

Purpose

The mass fire test characterizes the threat for spreading fire that a material imposes if it exists in an open container in a bulk (not layer or cloud) configuration. Based on the screening tests, the materials which are put into the mass fire test are most likely to burn, not explode; if ignited, such materials can spread fire by the following means: (1) by the flame impinging on and spreading across the ceiling such that the radiative heat from the hot ceiling gases significantly heats other combustibles in the room from above and (2) by radiative heating directly from the flame. In addition, direct flame impingement onto a combustible material and firebrands can spread fire, but these are not evaluated by this test.

Apparatus

The apparatus for this test is illustrated in figure a-33. The test sample is packed into the test container and placed on a scale such as the one shown. The purpose of the lever arm scale is to measure container weight loss as a function of time during the test, therefore any equivalent technique for measuring weight loss is acceptable.

These tests are to be conducted in containers which are approximately geometrically similar to the actual process vessel. The actual process vessel geometry should be approximated by a simple geometry which most nearly represents the actual process vessel. Simple geometries include cylinders, truncated cones, rectangular boxes, etc. For example, if the actual vessel is most nearly a cylindrical geometry, the test containers should be cylinders of the same length to diameter ratio as the actual process vessel. If the actual process vessel geometry is not known, a cylindrical vessel with a length to diameter ratio of 1 should be assumed. The container wall thickness is not critical and should generally be chosen in the range of 20 to 28 gage based on commercially available containers (if commercially available containers are the correct shape and size). The containers should be bare steel.

Four test vessel sizes should be used and two trials are required at each size. In each case the sample material should be packed into the test vessel at its actual inprocess bulk density. The test vessel sizes should be selected based in part on commercially available container sizes but the vessels should be such that the smallest test vessel contains 1 to 5 kg (2.2 to 11.0 lb) of sample, the next vessel contains 5 to 10 kg (11.0 to 22.0 lb); the third size vessel contains 10 to 25 kg (22.0 to 55.0 lb); and the largest vessel contains 25 to 50 kg (55.0 to 110.0 lb) of sample. The test vessels should be selected to give a good spread in the sample masses tested.

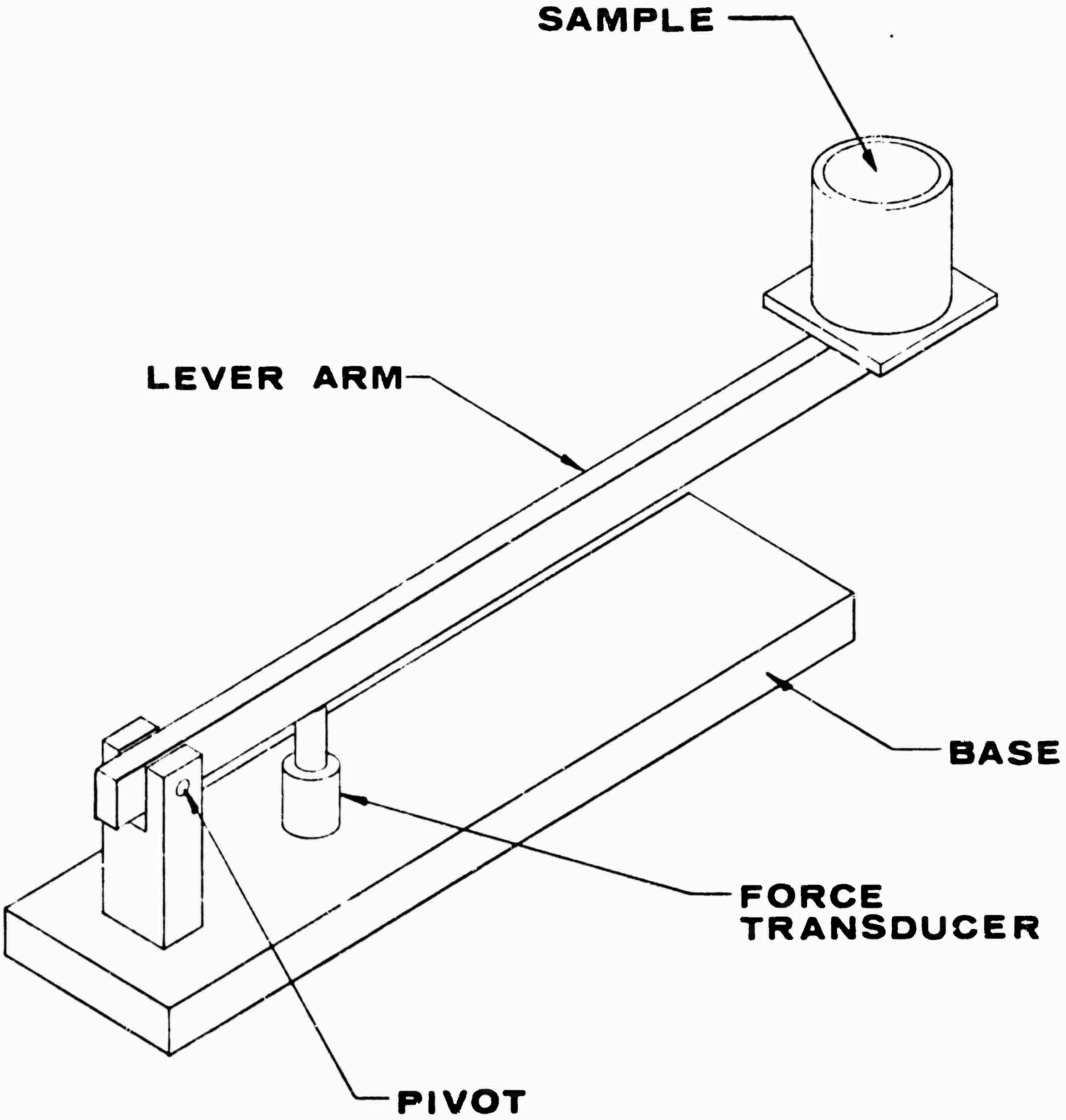


Fig a-33 Mass fire test arrangement

A cloth bag containing 5 g (0.175 oz) black powder placed at the center of the exposed sample surface will be the ignition source. The black powder is ignited remotely using an electric squib igniter.

One radiometer at 3 m (9.9 ft) from the test vessel will be used to measure remote heat flux, and real time colored movie coverage will be used to measure flame height and flame width. The movie camera should be placed far enough from the fire so that the total flame can be viewed and the flame height can be measured. Markers of known spacings should be placed in the camera's view so that the flame dimensions can be estimated.

Procedure and Data Reduction

The eight trials described above (four container sizes with two trials each) should be individually conducted. For each trial, weight loss rate (from the scale), heat flux (from the radiometer) and flame height and approximate flame width (from movie film) should be recorded versus time.

For each test, the time at which the flame height is maximum should be identified from the data. Corresponding to this time, the mass loss rate ($\dot{m}$), flame height (L), and flame diameter (D) should be recorded. This data should be fit to the following equation using the least squares method to determine the constants α and β:

$$\frac{L}{D} = \alpha\left(\frac{\dot{m}}{\rho_o D^{5/2} g^{1/2}}\right)^{\beta}$$

where g is the gravitational acceleration and ρ_o is the ambient air density. To estimate the full scale, actual process vessel, flame height, plot $\dot{m}$ versus container diameter and the ratio of flame diameter to container diameter versus container diameter. Using these curves, extrapolate to the full scale container diameter value and use the above expression for flame length to estimate the full scale flame length. If the expected full scale flame height is within 10 percent of the ceiling height (accounting for the container location above the floor in the actual process), the material is class 1.3A, mass fire hazard and the classification is completed.

Otherwise, the classification must be based on heat flux from the flame. Table a-4 should be used with figure a-34 to estimate the flame emissive power, E_f, from each radiometer for each test conducted. The heat flux is the maximum value observed during the test. The flame height and typical flame width are the values which correspond to the time or time period at which the maximum heat flux occurred (not necessarily the maximum values for the overall test). Configuration factor is determined from figure a-34 and emissive power is merely the heat flux Q divided by the configuration factor F. Note that on figure a-34 F is twice the value of F_{12} obtained directly from the graph.

Table a-4

Calculation of flame emissive power

Test No.	Container Size (m)	Maximum⊛ Heat Flux (w/cm^2)	Maximum[*] Flame Height (m)	Typical[*] Flame Diameter (m)	Configuration[**] Factor	Emissive[#] Power
1A	Smallest					
1B	Smallest					
2A	Small					
2B	Small					
3A	Large					
3B	Large					
4A	Largest					
4B	Largest					

⊛ Heat flux measured at 3 meters from the fire.

[*] Flame height and flame diameter are the observed dimensions at the time or during the time range at which the heat flux was maximum.

[**] From figure A13.1 using h = flame height, w = flame diameter, and d = gage distance.

[#] E_f is computed from $E_f = \frac{Q}{F}$

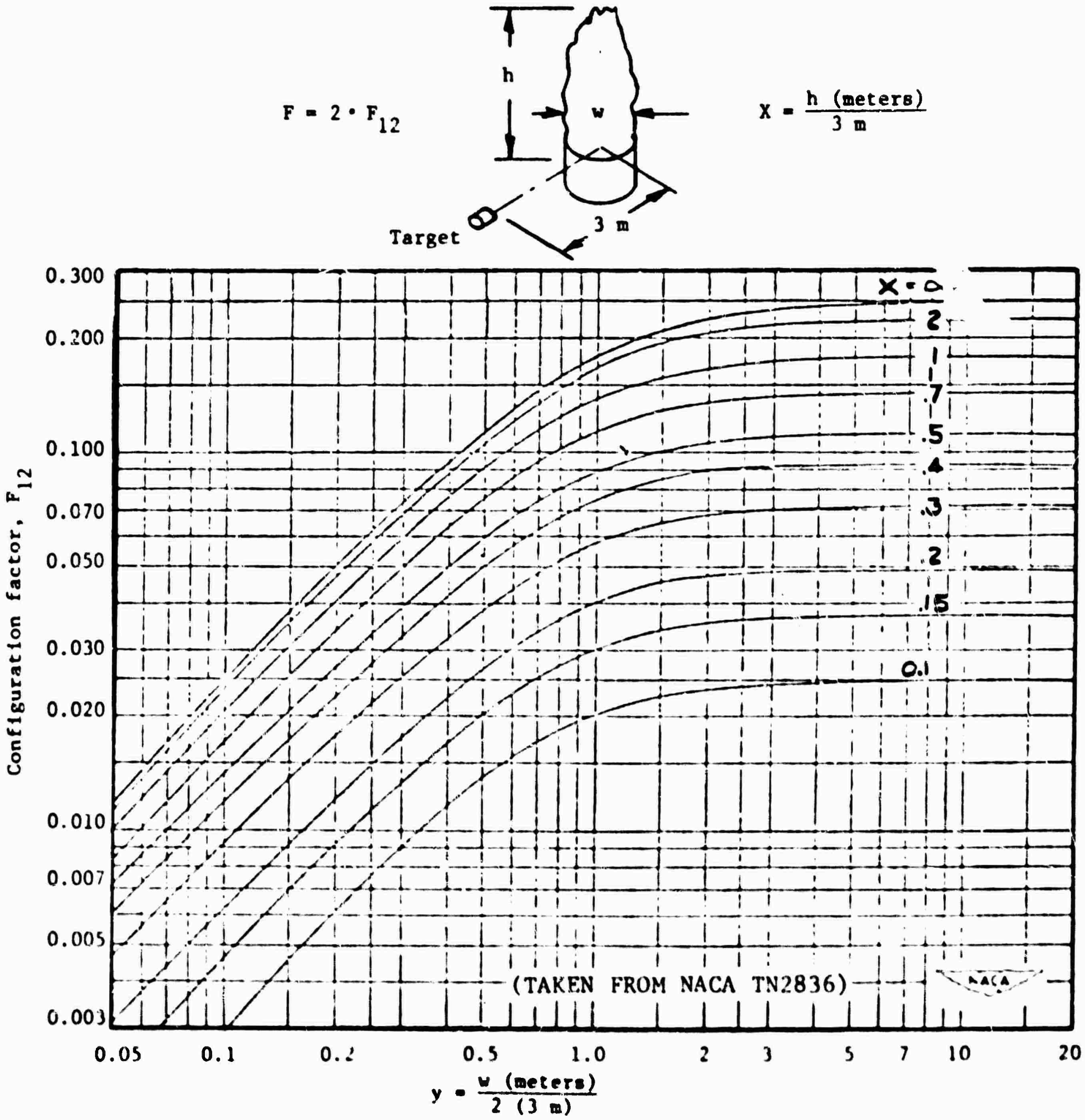


Fig a-34 Configuration-factor curve

Emissive power E_f must now be plotted versus test container size (e.g., diameter) and by extrapolation using a best fit curve, the value of E_f at the full scale container size should be estimated.

Similarly, maximum flame height (at maximum heat flux) and the corresponding ratio of flame diameter to flame height should be plotted versus container size to estimate the full scale flame height and corresponding diameter by extrapolation. The full scale flame height and diameter are then used to estimate the configuration factor F at 3 m from the fire using figure a-34 as described before. The full scale heat flux at 3 m (9.9 ft) is then

$$Q_{FS} = F_{(FS)} \; E_{(FS)}$$

where the subscript FS implies that the value is the estimated full scale value.

If the full scale heat flux at 3 m (9.9 ft), Q_{FS} is greater than or equal to 0.728 w/cm^2, the material is class 1.3A, mass fire hazard. Otherwise the material is class 1.4, minor hazard.

FIRESPREAD TEST

Purpose

This test is to characterize the potential for fire spread for materials which exist in layer configurations in their process operation. Based on the screening, test materials which are put into the firespread test are most likely to burn, not explode, if ignited. Such materials can spread fire by three primary mechanisms:

(1) by the flame impinging on and spreading across the ceiling of the process area such that the radiative heat from the hot ceiling gases significantly heats other combustibles in the area from above (applies only to uncovered layers)

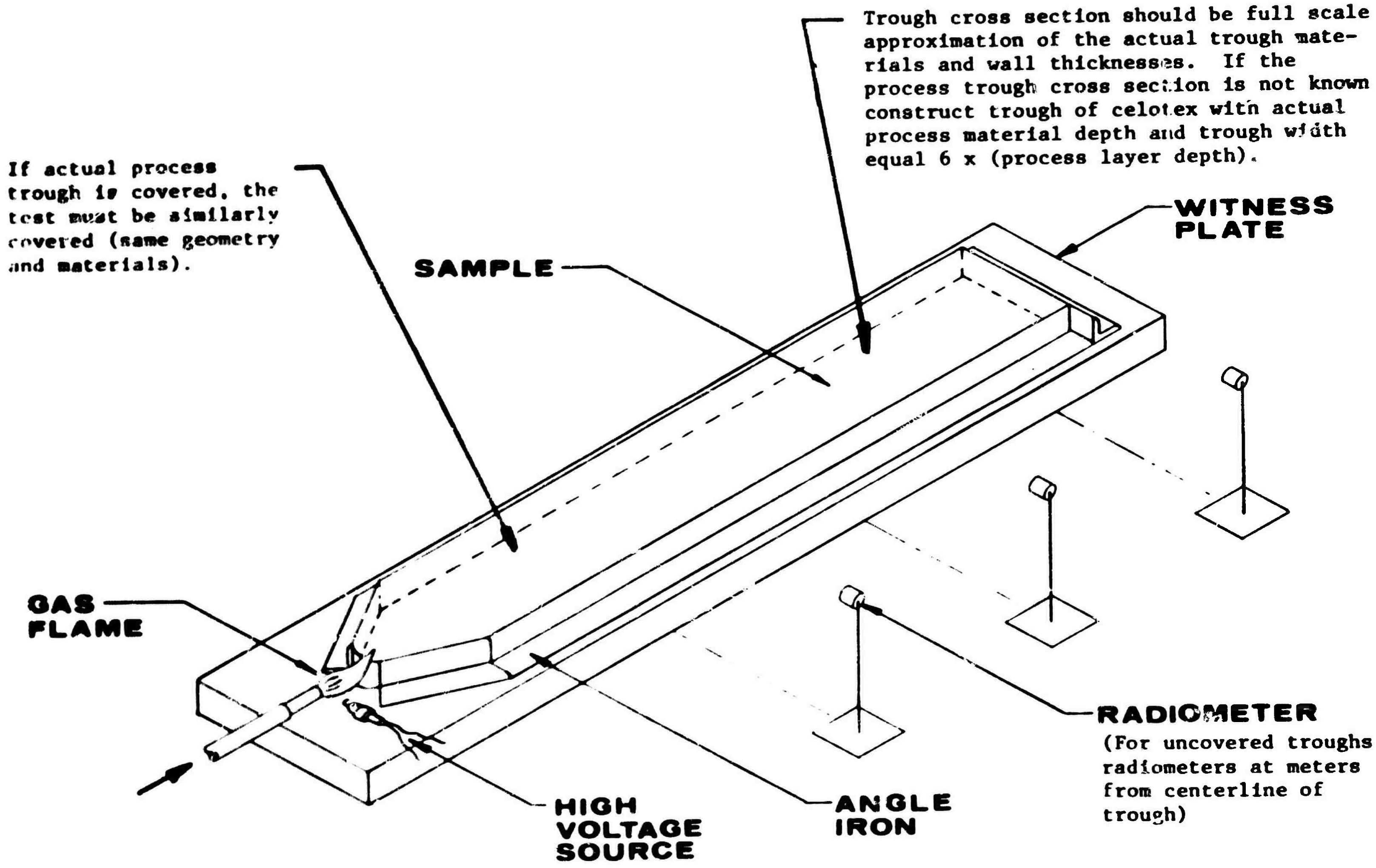
(2) by radiative heating directly from the flame (applies only to uncovered conveyors)

(3) by flame spread along the process material layer from the ignition location to the process vessels at the ends of the conveyor (covered or uncovered layers).

In addition, direct flame impingement onto a combustible material and firebrands can spread the fire (uncovered layers), but these are not evaluated by this test.

Apparatus

The test apparatus is shown in figure a-35. The sample material is loading into a 3 m (9.9 ft) long trough of the same cross section dimensions as exist in the actual process. Ignition is produced using a propane flame impinging on the sample as shown. The propane flame is ignited remotely using a spark plug. The trough materials of construction and wall thicknesses should be the same as the full scale process trough. If these are not known, the trough can be made of celotex boards. In all cases, the process material layer depth should be the maximum credible full scale layer depth, and the actual bulk density should be used. If the trough cross section dimensions are not known, the layer width should be six times the layer depth. If the actual process layer is covered, the test trough must also be covered using the same (or equivalent) materials of construction and geometry for the cover as exists in the full scale system. In this case, the classification will be based purely on flame spread velocity (class 1.3B versus 1.4) since the flame cannot impinge on the room ceiling and radiant heating from the flame is limited to within the cover. For covered assemblies, flame front velocity is measured using fiber optic "light pipes" to transmit the light signal produced as the flame passes the probe to a photocell. One fiber optic probe should be placed every 30 cm (12 in.) along the trough length. The probes should be oriented to look across the trough just above the surface of the process material. The probes should be shielded to minimize the view of the oncoming flame.



If actual process trough is covered, the test must be similarly covered (same geometry and materials).

Trough cross section should be full scale approximation of the actual trough materials and wall thicknesses. If the process trough cross section is not known construct trough of celotex with actual process material depth and trough width equal 6 x (process layer depth).

(For uncovered troughs radiometers at meters from centerline of trough)

Fig 3-35 Firespread test apparatus

If the actual process layer is not covered, flame front velocity is measured from the real time movie film record of the test. The camera should be positioned to record the side view of the flame spread test. The camera should be able to view the total height of the flame during the test. Markers of known spacing should be positioned in front of the trough in view of the camera so that flame front velocity and flame height can be measured during the test as functions of distance along the trough from the ignition point. For uncovered troughs, three radiometers are used to record radiated heat flux from the flame. The first radiometer is on a line perpendicular to the trough centerline at the middle of the first one-third of the trough length. This radiometer is 3 m (9.9 ft) from the trough centerline and is tilted slightly upward to view a point 1 m (3.3 ft) above the trough centerline. Similarly, The second radiometer views the middle one-third of the trough and the third radiometer views the final section of the trough.

Procedure and Interpretation of Results

Three trials are required for this test. Since the cross section is full scale, scaling is limited only to changes in the fire characteristics with distance from the ignition point.

If the trough is covered, the evaluation is limited to flame front velocity. Flame front velocity is measured as a function of distance from the ignition point in the three tests. If the results of the three tests are close, a curve fit should be done based on all of the data to obtain the function of velocity V(x) versus distance x. To estimate the time τ for the flame front to traverse the full scale trough length x_o, the following equation is solved

$$\tau = \int_{o}^{x_o} \frac{dx}{V(x)}$$

where V(x) is the experimentally derived velocity-distance relation. In this calculation it is assumed that the trend in velocity continues as was observed in the tests, even if the full scale trough is significantly longer than the 3 m (9.9 ft) test length. If the estimated full scale flame tranvers time τ is less than or equal to 2 sec, the material is class 1.3B, firespread hazard. If τ is greater than 2 sec, the material is class 1.4.

If the trough is not covered, flame height is considered first, flame radiative heat flux is considered second, and flame front velocity is considered (as described above) last, as the criteria for classifying the material. To estimate the maximum flame height from the three tests conducted, the visible flame height as determined from the experimental data should be plotted versus distance from the ignition point. The best curve fit describing the upper envelope should be used to determine whether flame height is still increasing at 3 m (9.9 ft) from the ignition point or if it has stabilized.

If flame height is still increasing with distance, extrapolate to the full scale trough length using the upper envelope curve. Otherwise, use the maximum flame height observed in the tests. If the maximum flame height is within 10 percent of the ceiling height, accounting for the elevation of the trough above the floor in the real process plant, the material is class 1.3A, mass fire hazard. otherwise consider heat flux from the flame at 3 m (9.9 ft).

The heat flux is measured directly at the 3 m (9.9 ft) distance of interest. Scaling must only consider changes in the heat flux with distance from the ignition point. The radiometer nearest to the ignition point is used to approximately give heat flux at 3 m (9.9 ft) for the first one-third of the trough. The second radiometer covers the middle section and the last radiometer is used for the final one-third of the trough. The flame position at any time is provided from the movie film coverage. Corrections to the radiometer data to account for the distance increasing to either side of the section center can be accounted for by multiplying the measured heat flux by

$$\frac{R^2}{9}$$

where R is the actual distance from the flame to the radiometer in meters. By plotting heat flux versus flame distance from the ignition point and extrapolating to the full scale trough length (if heat flux is increasing) the maximum heat flux at 3 m (9.9 ft) to the side of the trough can be estimated. If this maximum heat flux is greater than or equal to 0.728 w/cm^2, the material is class 1.3A; otherwise the flame spread time is used to classify the material as described above.

CLOUD EXPLOSION TEST

The objective of the cloud explosion 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.

Test Description

The Bureau of Mines "Hartmann" Dust Explosibility Test Apparatus[*] 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 figure a-36. 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 the dusts. The gas used in this test can be varied in order to stimulate 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. The maximum pressure and rate of pressure rise determined in the test are used to compute the "explosion severity" using the formula:

$$\text{Explosion Severity} = \frac{\text{(explosion pressure X maximum rate) for sample dust}}{\text{explosion pressure x maximum rate) for standard Pittsburgh coal dust}}$$

Using the "explosion severity" index, the hazard can be ranked as follows:

Relative Explosion Severity Rating	Explosion Severity
Weak	0.5
Moderate	0.5 - 1.0
Strong	1.0 - 2.0
Severe	2.0

---

[*] Dorsett, H. G., "Laboratory Equipment and Test Procedures for Evaluating Explosibility of Dusts", Bureau of Mines, RI5624, 1960.

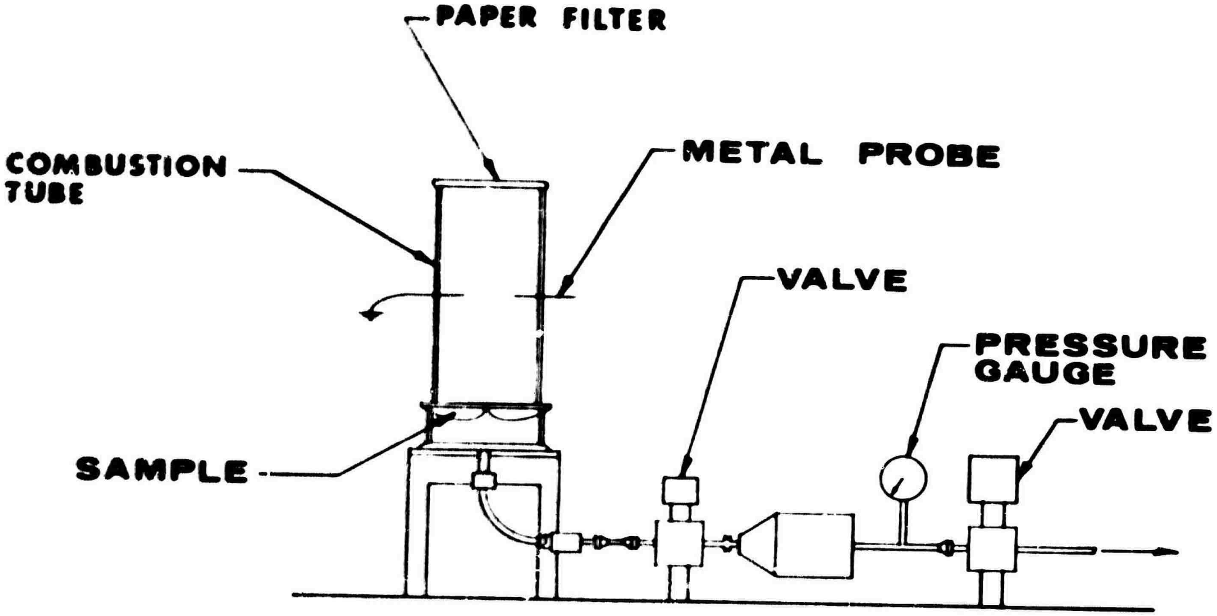


Fig a-36 Hartmann dust explosibility test apparatus

Materials which have an explosive severity of <0.5 (weak) are class 1.4 (minor hazard) whereas any higher ratings will put the material into class 1.1B (cloud explosion hazard).

DISTRIBUTION LIST

Commander
U.S. Army Armament Research and
Development Command
ATTN: DRDAR-CG
DRDAR-LC
DRDAR-LCM
DRDAR-LCM-S (12)
DRDAR-TSS (5)
DRDAR-LCU-P
Dover, NJ 07801

Commander
U.S. Army Materiel Development and
Readiness Command
ATTN: DRCDE
DRCIS-E
DRCPA-E
DRCPP-I
DRCDI
DRCSC-S
5001 Eisenhower Avenue
Alexandria, VA 22333

Commander
USDRC Installations and Services Agency
ATTN: DRCIS-RI-IU
DRCIS-RI-IC
Rock Island, IL 61299

Commander
U.S. Army Armament Materiel Readiness Command
ATTN: DRSAR-IR (2)
DRSAR-IRC
DRSAR-ISE (2)
DRSAR-IRC-E
DRSAR-PDM
DRSAR-LC (2)
DRSAR-LEP-L
DRSAR-ASF (2)
DRSAR-SF (3)
Rock Island, IL 61299

Chairman
Dept of Defense Explosives Safety Board
Hoffman Bldg 1, Room 856C
2461 Eisenhower Avenue
Alexandria, VA 22331

Commander
U.S. Army Munitions Production Base
  Modernization Agency
U.S. Army Armament Materiel
  Readiness Command
ATTN: SARPM-PBM-LA
      SARPM-PBM-T-SF
      SARPM-PBM-EP (2)
Dover, NJ 07801

Director
Ballistic Research Laboratory
ARRADCOM
ATTN: DRDAR-BLE (C. Kingery) (2)
Aberdeen Proving Ground, MD 21010

Defense Technical Information Center (12)
Cameron Station
Alexandria, VA 22314

Commander
U.S. Army Construction Engineering
  Research Laboratory
ATTN: DERL-ER
Champaign, IL 61820

Office, Chief of Engineers
ATTN; DAEN-MCZ-E
Washington, DC 20314

U.S. Army Engineer District, Huntsville
ATTN: Construction Division-HAD-ED (2)
PO Box 1600 West Station
Huntsville, AL 35807

Commander
Indiana Army Ammunition Plant
ATTN: SARIN-OR (2)
      SARIN-SF
Charlestown, IN 47111

Commander
Kansas Army Ammunition Plant
ATTN: SARKA-CE
Parsons, KS 67537

Commander
Lone Star Army Ammunition Plant
ATTN: SARLS-IE
Texarkana, TX 57701

Commander
Milan Army Ammunition Plant
ATTN: SARMI-S (2)
Milan, TN 38358

Commander
Badger Army Ammunition Plant
ATTN: SARBA (2)
Baraboo, WI 53913

Commander
Holston Army Ammunition Plant
ATTN: SARHO-E
Kingsport, TN 37662

Commander
Iowa Army Ammunition Plant
ATTN: SARIO-A
Middletown, IA 52638

Commander
Joliet Army Ammunition Plant
ATTN: SARJO-SS-E
Joliet, IL 60436

Commander
Longhorn Army Ammunition Plant
ATTN: SARLO-O
Marshall, TX 75670

Commander
Louisiana Army Ammunition Plant
ATTN: SARLA-S
Shreveport, LA 71102

Commander
Newport Army Ammunition Plant
ATTN: SARNE-S
Newport, IN 47966

Commander
Pine Bluff Arsenal
ATTN: SARPE-ETA
Pine Bluff, AR 71601

Commander's Representative
Sunflower Army Ammunition Plant
ATTN: SARSU-O
Box 640
De Soto, KS 66018

Commander
Volunteer Army Ammunition Plant
ATTN: SARVO-T
Chattanooga, TN 34701

Southwest Research Institute
6220 Culebra Road
ATTN: J. W. Gehring (20)
San Antonio, TX 78228

Weapon System Concept Team/CSL
ATTN: DRDAR-ACW
Aberdeen Proving Ground, MD 21010

Technical Library
ATTN: DRDAR-CLJ-L
Aberdeen Proving Ground, MD 21010

Director
U.S. Army Ballistic Research Laboratory
ARRADCOM
ATTN: DRDAR-TSB-S
Aberdeen Proving Ground, MD 21005

Benet Weapons Laboratory
Technical Library
ATTN: DRDAR-LCB-TL
Watervliet, NY 12189

U.S. Army Materiel Systems Analysis Activity
ATTN: DRXSY-MP
Aberdeen Proving Ground, MD 21005

Director
U.S. Army TRADOC Systems Analysis Activity
ATTN: ATAA-SL (Technical Library)
White Sands Missile Range, NM 88002