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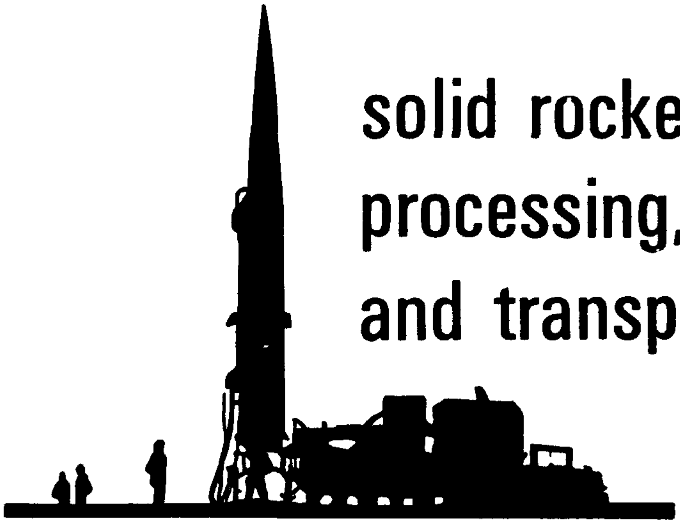
HAZARDS OF CHEMICAL ROCKETS AND PROPELLANTS HANDBOOK

VOLUME II



solid rocket propellant processing, handling, storage and transportation

MAY 1972

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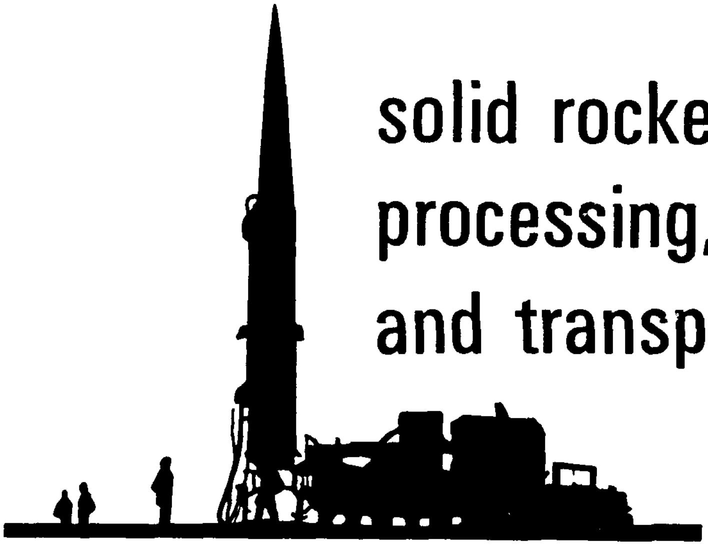

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The matter contained in this document has been prepared for information purposes to assist Federal Departments and Agencies in the safe conduct of those activities which involve chemical propellants. The reader is cautioned that the contents have been prepared for use by persons knowledgeable in the technical areas concerned and that such experts need to be duly consulted whenever the contents are to be utilized. The information and recommendations found herein have been compiled from sources believed to be reliable and to represent the best opinion on the subject at the time of preparation. Since these source publications are under continuing revision, the newest revision should be consulted in instances where specific state-of-the-art information is desired by the user.

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This manual is intended as a source of information and as a guide for the handling, processing, storage and transportation of chemical rocket propellants and ingredients; primary attention is given to the controlled, on-site, situation and circumstances. It is not intended as a regulation concerning the manufacture, storage, use and distribution operations with propellants or propellant ingredients at privately owned facilities. Regulatory bodies and responsible authorities are therefore cautioned against direct application of these guidelines to any specific location or operation without considering the judgment and experience of trained personnel in the areas of concern.

FOREWORD

This volume is intended as a source of information and as a set of basic guidelines for the processing, handling, storage, and transportation of chemical propellants and propellant ingredients. The work was accomplished under the auspices of the Joint Army, Navy, NASA, Air Force (JANNAF) Propulsion Committee, formerly ICRPG, under the technical management of the Liquid Propellant Subcommittee and by the committee members and participants of the Hazards Working Group. The complete work--Chemical Rocket/Propellant Hazards--consists of three volumes, each a task of one or more of the committees as indicated below:

Chemical Rocket/Propellant Hazards

Volume I - "General Safety Engineering and Design Criteria."
Safety Criteria Committee assisted by all committees of the Hazards Working Group

Volume II - "Solid Rocket/Propellant Processing, Handling, Storage and Transportation."
Solid Propellant Manufacturing, Handling, and Storage Committee assisted by the Committee on Environmental Health and Toxicology

Volume III - "Liquid Propellant Handling, Storage, and Transportation."
Liquid Propellant Handling and Storage Committee assisted by the Committee on Environmental Health and Toxicology

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CHAPTER 1

INTRODUCTION

1-1 BACKGROUND

Through the years the manufacturing, handling and using of solid propellant missiles and rockets have continued to take their toll in lives, disability and property damages. Technical requirements become more demanding and faster burning propellants are constantly sought. This search has evolved many propellants about whose reaction it might truthfully be said that, "they are nothing more than controlled explosions."

1-2 EXPERIENCE FACTORS

Government agencies and industry have proven that they are capable of manufacturing, handling and using these propellant systems safely if the process, once learned, is repeated in exactly the same manner every time. Standard processes make little or no allowance for the unexpected response that often comes from the human operator. Unfortunately when the response is the unexpected rather than the standard one desired, the result is an accident and the penalty of lives, disability or property damage is paid. There have been periods in which the manufacture of missile and rocket systems have gone for several years without major accidents; but presently, the accident penalties are large and exacting.

1-3 PURPOSE

Certainly one of the reasons that this is true today is that the world situation has demanded a great increase in propellant production causing relatively inexperienced personnel to be brought into the process. In addition to this, many persons, both contractor and government, who had learned their safety through experience have either retired or moved up the ladder of supervision to the point where they are no longer directly involved. Unfortunately, it seems that they did not sufficiently train their replacements to understand the common problems that have taken their toll throughout the years. Because it was believed that some of the safety know-how had been lost, a decision was made to produce this Volume in hopes of furnishing material that would once again make available to those directly involved, the knowledge that might well save their lives, their limbs or their jobs. With the guidance of the Technical Steering Committee of the JANNAF Working Group on Hazards, this attempt was begun.

1-4 SCOPE

It will be noted that all of the chapters and all the sections of the chapters are not written alike. This is easily understood when it is realized that many different men from industry and government, considered to be experts in their particular field, have contributed these different sections and chapters. There has been no attempt to include all available material on each subject since this would take many volumes; but rather the attempt has been to bring together those processes or items that continue to prove most costly, and to provide references to other applicable documents that offer a more extensive review of the subject. It does seem appropriate, however, to consider the types of hazards that exist during the life cycle of solid propellant systems. It is hoped that these brief comments cause some thinking which will result in action to increase safety throughout industry.

1-5 PROPELLANT INGREDIENTS

1-5.1 GENERAL INFORMATION. A serious hazard exists in the handling of solid propellant ingredients. A number of these materials have proven to be hazardous both from the point of fire and explosion. Some undergo detonation or low velocity reactions of catastrophic extent. Specifically, undiluted nitroglycerin and even nitroglycerin diluted somewhat with inert plasticizers may exhibit low velocity reactions which can be initiated with extremely low stimuli as compared with conventional high explosives. Other nitrate esters with lower heat of explosion exhibit the sensitivity in a lesser degree. A detonation hazard exists with fuel-contaminated ammonium perchlorate from both fire and shock initiation. Drums of ammonium perchlorate may be sympathetically detonated by adjacent detonation of material in process. High explosive additives such as RDX are hazardous from both fire and shock initiation, as is dry nitrocellulose or high specific surface material containing nitrocellulose (casting powder). Somewhat less dangerous are metallic powders mixed with air and solvent vapors at concentrations within the explosive limits in air. When the possibility of hazard has been recognized, it is usually possible for the hazard to be significantly reduced.

1-5.2 CLASSIFICATION. Ingredients may be roughly divided into two general hazards classifications. First there are those with high incidence of accidents with normal handling procedures, and secondly there are those with which accidents are of a magnitude considerably more destructive than anticipated. In the first category are the liquid explosive ingredients which respond destructively to weak stimuli. Also, novel materials containing both fuel and oxidizing elements should be considered in this category until adequate experience has been gained in their handling properties. In the second category are porous materials in which a confined flame might lead to detonation, e.g., fuel-contaminated oxidizer or dry nitrocellulose. Minimization of quantities is not only desirable but imperative in preventing an accident involving such ingredients from becoming catastrophic. Scraps accumulated through processing, machining, and trimming operations also provide this type of hazard and demand real control.

1-6 PROPELLANT PROCESSING

1-6.1 MIXING. Both experience and theory would indicate that mixing is one of the most hazardous operations which may be undertaken. It provides both the means of initiation and confinement. Everything needed for building up a reaction of explosive violence is present. Since the process of mixing requires the input of energy, thermal initiation of a mix or some part of it may be obtained by friction, impact, shear, or localized overheating. In the process of adding ingredients, reactive components may be concentrated momentarily, such that highly exothermal reactions occur in a localized area producing ignition. In many mixing operations, clearance of moving parts is kept small to ensure a high shear action of the operation. Accidental introduction of unwanted hard materials such as nuts, bolts, washers, screens, or spatulas may lead to ignition. Since the primary purpose of most mixing operations is the incorporation of solids into a liquid or rubbery continuum, there exists the possibility that at some time the solid materials have not been completely wetted and, in fact, a porous-dilatant mass exists. If ignition occurs at this phase of processing and some confinement exists, the ignition is most likely to proceed from deflagration to a detonation or at least a violent explosion. Even at later stages in a mixing operation, mixing cycles which sweep the topmost material to the bottom may distribute an ignited surface over a wide area under confinement and produce an explosive reaction with the resultant spraying of the remainder of the mix over a wide area in fairly finely divided form. Such violent reactions, while not detonations, are extremely disruptive to structures and, at a distance of a few diameters away, produce an air blast of the same order as a detonation.

1-6.2 UNCONSOLIDATED MASS. The major hazard period exists when the propellant is not consolidated, therefore, the most hazardous steps in processing are those in which ignition is possible when porous masses are present. This condition exists primarily in the early stages of mixing where complete consolidation has not as yet occurred, but work is being performed on the mix. When submerged bearings or glands are used, the probability of accidents is raised and meticulous maintenance of equipment is demanded.

1-6.3 TRIMMING. Operations requiring the use of metal-to-metal friction in the presence of propellants are also considered to be hazardous. An example is the final trimming to weight of case-bonded charges by machining or cutting operations. Ignition at this time results in a confined propellant fire usually producing serious damage to plant and personnel through its intensity. Mandrel or core withdrawal in poorly designed equipment is also considered hazardous since an ignition may well result in an explosion. Mold disassembly and clean-up operations offer many hazards and when they involve energetic double base propellants may be quite hazardous.

1-7 HANDLING OF THE FINAL MANUFACTURED PRODUCT

When reasonable care has been given to the handling of rocket and missile motors, no serious incidents have occurred. Reasonable care is equal to that which would be afforded drums of gasoline, ether, or other flammable solvents. The results of lack of care can be more catastrophic than those resulting from ignition of gasoline, but considerable experience in handling the final product has resulted in few major accidents. This excludes the intentional firing or attempted destruction of rounds which may be defective, or those which are provided with stimuli above that which may be expected during normal handling for this type of material. Routine transportation hazard testing of final rounds, such as drop tests, bonfire tests, and bullet impact tests, have not for the most part resulted in detonation. Fires and explosions do result from the ignition of confined propellants which after all were designed to liberate energy on ignition. Testing has shown the foremost hazard to be the distribution of burning fragments of propellant over relatively wide areas in large enough sizes to initiate any combustible materials.

1-8 REDUCTION OF HAZARDS

1-8.1 CONVENTIONAL METHODS. A few of the conventional systems of reducing hazards are; careful control of extraneous material which might cause ignition, adjusting the addition of ingredient in mixing to prevent the accumulation of large quantities of unconsolidated material, using remote controlled operations wherever practicable, desensitization of explosive liquids by blending inert liquids with the explosive, and using new design mixers with no submerged bearings and with greater clearances.

1-8.2 ANALYTICAL METHOD. A more unconventional system is the attempt to utilize all available test results such as thermal, friction, impact, critical diameter, and card gap to determine the stimulus needed for ignition, and then to analyze the various steps of the process to determine if the stimulus is present. There appears to be some evidence that this approach will provide enough data to be of real use in accident prevention. Many of the tests and their uses will be provided in this work.

1-9 CONCLUSIONS

Despite all attempts, safety cannot be completely categorized so the final paragraphs in this chapter will be given to some statements that history has already proven.

1-9.1 CRITICAL PHASE OR OPERATION. If manufacture or use of a product requires a series of operations, it is necessary to determine the critical sensitivity during each operation. It cannot be assumed that because the final product is difficult to initiate that there are no sensitive operations.

1-9.2 OPERATION CHECKS AND CONTROLS. It is not enough to make a product fool proof, one seldom meets fools, but every effort is required to keep the smart operator from short-cutting the required steps because he thinks they are unnecessary. Such short cuts have been known to cost lives.

CHAPTER 2

HAZARDS ANALYSIS

2-1 INTRODUCTION

Hazards analysis is a practical approach to evaluating processing hazards and is essentially the investigation of an accident before it happens. Hazards analysis emphasizes the quantitative assessment of process conditions in engineering terms and establishes the response of material to the stimuli found in the process. Thus, both process potential and material response expressed on a common basis, are quantitatively measured in assessing problems or hazards.

The introduction of any new or modified material, process or procedure in the chemical, explosive or propellant industry raises the question, does this create a hazard? The hazard question is a complicated one since most materials processed can be hazardous in a given situation. Consequently, the problem becomes one of deciding (a) the degree of hazard, (b) if the precautions to prevent initiation or explosion are sufficient, and (c) whether the protection of personnel and facilities is adequate. All of these considerations must of course be balanced against the original justification for introducing the new material or process, whether it was for reasons of economy, quality, or performance improvement. Catastrophic incidents, unnecessary and costly precautions, or the rejection of a material or process because of the inability to properly assess suspected hazards places any industry in an untenable position.

The most common approach to hazards assessment has been sensitivity testing of subscale samples to establish the tendency of a material to initiate or explode. Traditionally, these tests have represented some stimulus to which a material may be subjected during processing. The common initiation tests have been thermal, impact, friction, electrostatic, and shock. To establish properties after initiation, tests have been made to determine tendencies toward transition to explosion and propagation of an explosive reaction.

The data generated by such tests are usually abstract and are expressed in statistical terms such as the 50% probability of initiation or explosion occurring. These data are useful for comparing one compound with another at a given probability level and for determining the effect of a test technique or possibly an environmental condition. The greatest problem in using such data is deciding whether the change in sensitivity has any practical significance. If the data cannot be analyzed from a practical viewpoint, one could conclude in the case of two materials which exhibit impact sensitivity values of 10 and 20 cm respectively, that one material is more sensitive than the other, but would be unable to say if either constitutes a hazard in a given situation.

In the past, tests have been developed with considerable emphasis on obtaining reproducible data. This objective generally resulted in (a) modifying the test materials to obtain uniform samples, (b) testing under constant weight or volume conditions, or (c) using test components that represent severe or unrelated conditions or that were selected because the components were relatively unaffected by repeated tests. Although reproducible tests are certainly necessary if there is to be much faith in the results, the zeal to obtain reproducibility frequently overshadows the original reason for the tests; i.e., to establish whether a material or process constitutes an unacceptable hazard.

For years, the probable presence of hazards has been predicted on the basis of experience; that is, a new or modified material is compared to a standard compound selected either arbitrarily or because the standard compound represents some experience in a given process. This method has a number of pitfalls, the more important being as follows:

a. Any given process or material used as the standard must be operated or employed initially without knowledge of the safety of operations simply because no prior experience exists.

b. The experience usually represents a number of injuries, fires and/or explosions with the standard material or process.

c. The conclusions based on experience are valid only if no changes occur in a process which would modify the sensitivity of the material or increase the potential energies such as the introduction of new equipment or procedures.

d. This method offers no means of coping with a material which is more sensitive than the standard since no experience at this level of sensitivity is available.

e. With the advent of d, the first problem again occurs because no experience exists with this material under a specified set of processing conditions.

Therefore, the use of a method which cannot be applied to all new situations severly restricts the ability to assess the practicality, from the standpoint of economy and safety, of using new or modified materials, procedures, and equipment. Consequently, the need arises for a method of applying sensitivity data directly to process conditions. The Hazard Analysis Technique meets this requirement.

2-2 A SYSTEMS-ENGINEERING APPROACH TO HAZARDS ANALYSIS. Hazards analysis is divided into (a) Process Survey, (b) Sensitivity Testing, (c) Support Studies, and (d) Hazards Evaluation; i.e., conclusions concerning the degree of hazard, required modifications, and subsequent safety margin. See Figure 2-1.

2-2.1 PROCESS SURVEY

2-2.1.1 Observation Phase. The observation phase of Process Survey is concerned with establishing potential

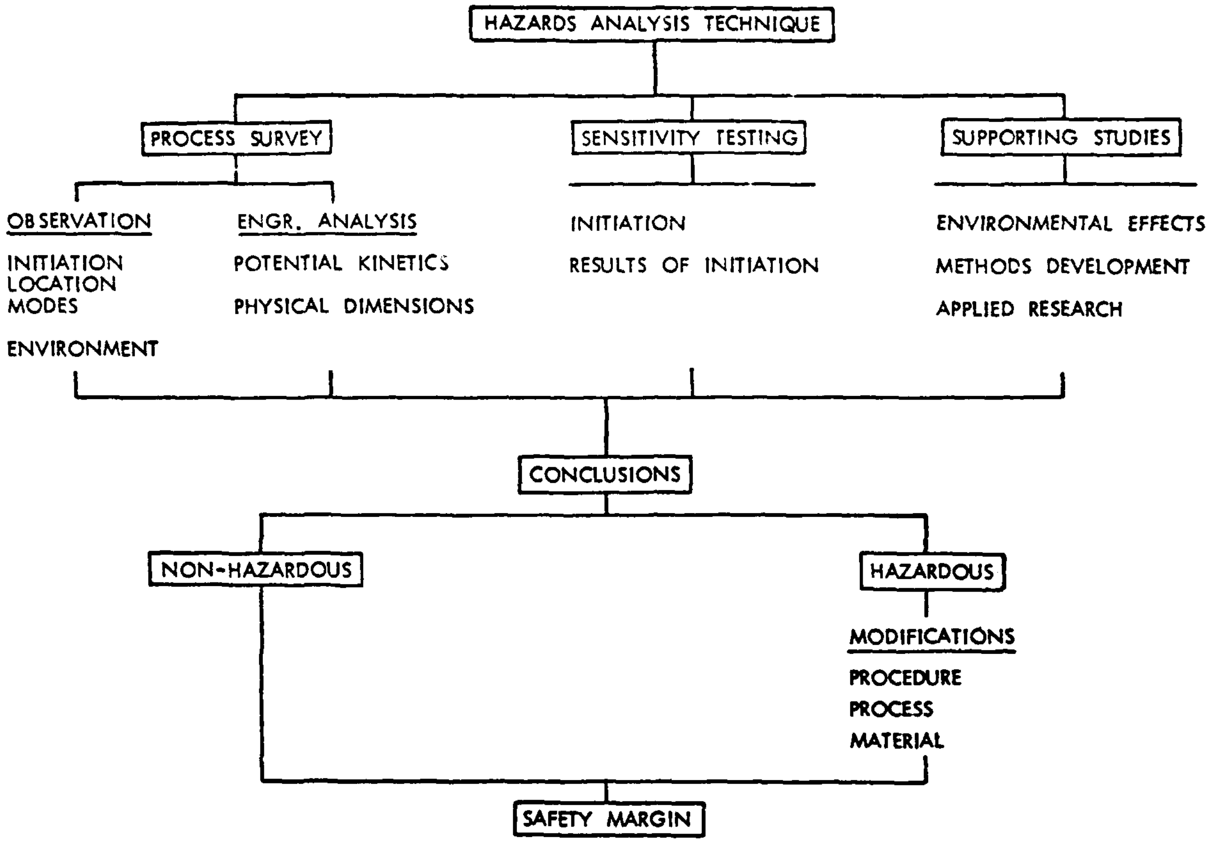

Figure 2-1. Hazards Analysis Technique

initiation points, modes of initiation (impact, friction, etc.) and processing conditions under which initiation may occur. The processing conditions to be established include chemical and physical states of the ignitable material, material of fabrication the ignitable material contacts, quantities and temperatures involved, and the likelihood that these conditions will promote transition to an explosive reaction after ignition.

2-2.1.2 Analytical Engineering Phase. The second phase under Process Survey is analytical engineering which employs experimental and theoretical procedures to establish the kinetics involved in terms of force, energy, rate or velocity; specific physical dimentions of transfer lines and containers; and, bulk densities of explosive materials. These data coupled with those of the observation phase, establish the potentials of the process, the sensitivity test conditions, and the limits.

2-2.2 SENSITIVITY TESTING. Initiation test equipment is used to determine the material initiation characteristics under the processing conditions established in the Process Survey. These characteristics are expressed in like terms, e.g., force, energy, velocity, rate, etc. This phase includes tests to establish the results of initiating a material under the conditions of mass, confinement, temperature, and the configuration existing in the process. These results of initiation are important because the prevention of initiation cannot be guaranteed under abnormal or accidental conditions. Furthermore, one must know with a high degree of certainty whether the initiation will result in a fire or some catastrophic incident. All tests and the interpretation of test results are based on establishing the threshold of initiation, transition, and propagation, since data cannot be applied unless they represent with some degree of confidence the "no reaction" level.

2-2.3 SUPPORT STUDIES. The supporting studies phase of the analysis is employed when existing tests must be modified to duplicate an environmental condition, when a new method must be developed to establish the initiation, characteristics and results of initiation, or when the mode of initiation must be determined.

2-2.4 HAZARD EVALUATION OR CONCLUSIONS. The potential kinetics and dimensions from the Process Survey are related to the threshold of initiation and explosive characteristics of the materials to establish the margin of safety. Should an unacceptable hazard or an insufficient margin of safety be found, this correlation provides a reliable engineering basis for deciding if modification is feasible and, if so, selecting the process or material to be modified. Thus, the use of the hazard analysis concept permits a decision concerning the margin of safety, feasibility of modification, and the extent or method of modification to be made on a quantitative basis rather than on the basis of opinion, experience, abstract data, or some empirical tests.

Test techniques employed in the hazards analysis concept are given in the following sections. These sections also present the test objective, operating principle, description, limitations, and application to hazard analysis.

2-3 DIFFERENTIAL THERMAL ANALYSIS (DTA)

2-3.1 OBJECTIVE. To detect exothermic and endothermic reactions in a propellant or constituent as heat is applied at a given input rate and to determine the relative magnitude of these exotherms and endotherms.

2-3.2 OPERATING PRINCIPLE. The sample and an inert reference material are heated simultaneously at the same caloric rate. The exotherm or endotherm is measured by the differing temperature recordings on a common time base.

2-3.3 TEST DESCRIPTION. In its simplest form DTA is performed by heating separately a 20 mg propellant sample and a reference material (such as alumina or glass beads) at the same rate of heat input in a common aluminum block. The two materials are instrumented with thermocouples so that individual traces of temperature versus time are recorded simultaneously. A prechosen, constant rate of reference temperature rise is maintained, which can either be extended to the highest temperature capacity of the system or be cut off at deflagration of the sample or any other arbitrary point. The extent of exothermic or endothermic reaction is determined by comparing sample and reference temperature recordings.

Variations in DTA equipment are available with a wide range in heating rate and upper temperature limit, some in pre-assembled commercial units such as the duPont Model 900 Differential Thermal Analyzer. The Naval Weapons Center, China Lake, California, has designed and fabricated an instrument which is typical of one of the more elaborate setups and which is operational to approximately 850°F (reference 1). It features the following: (a) a three-thermocouple system and recorder which plots separately and simultaneously the actual reference temperature of the system as well as the temperature differential between sample and reference material, (b) a power proportioning temperature programmer which automatically brings up the reference temperature of the moment, and (c) side-arm connections to the reaction tubes which permit DTA's to be run under inert or dry-box atmospheres, under reduced pressure conditions, or with exhausting of gaseous products of decomposition.

2-3.4 TEST ANALYSIS AND LIMITATIONS. Differential thermal analyses for elevated temperature sensitivity have gained the widest acceptance of any thermal test for propellants, components, and intermediate mixtures. DTA yields not only decomposition temperatures at various rates of temperature rise but also, by proper selection of heating rates, thermodynamic constants which are useful in basic kinetics studies of propellant grain stability in large sizes. The DTA test has its greatest value in the detection of unsuspected endothermic or exothermic reactions of new compositions and in providing qualitative estimates of their effect. Quantitative assessment of the effects requires measurements of heat capacities and heat transfer constants.

2-3.5 APPLICATION TO HAZARD ANALYSIS. Two parameters from the DTA that may be used as limiting tolerable process temperatures are the autoignition temperature and the temperature of onset of an exotherm (sometimes designated threshold temperature). However, either must be used with due regard for the kinetics of the exotherm under study. If heat transfer characteristics in the process system are good and if the time of exposure to the process temperature is short, the autoignition temperature, minus a margin for safety, may be an upper limiting tolerable process temperature. For a system with poor heat transfer (for example a large volume of propellant) or for longer exposure times, the exotherm threshold temperature would be the safer limit, provided that (a) the threshold temperature was determined at a high heating rate in the DTA, and (b) the mixture is rendered homogeneous before being exposed to the high processing temperature.

2-4 SELF-HEATING TEST

2-4.1 OBJECTIVE. To determine the temperature at which a given mass and configuration of propellant will commence self-heating to destruction from its own decomposition exotherm.

2-4.2 OPERATING PRINCIPLE.

2-4.2.1 Cook-Off Tests. Progressively larger regular-shaped pieces of propellant (for example, right cylinders), instrumented with internal thermocouples, are maintained at elevated temperature under a constant heat-transfer environment until deflagration occurs.

2-4.2.2 DTA's at Differing Heating Rates. (reference 1) The DTA is performed at a number of different heating rates from 1.0 to 10.0°C per minute, and the data are plotted as log heating rate vs 1/T, where T is the absolute temperature of the first exotherm.

2-4.2.3 Self-Heating Determination in an Isothermal Environment (reference 2). The rate of change in temperature is measured at the center, the surface, and one point on a radius of cylindrical propellant charges being heated in a constant temperature bath of established heat transfer coefficient. For all determinations, it is necessary to acquire values on the propellant for thermal conductivity, density, and heat of explosion.

2-4.3 TEST DESCRIPTION. The temperature rise data are plotted as log heating rate versus 1/T where T is the absolute temperature of the first exotherm from the DTA graph. Then from the activation energy E, and the frequency factor A, calculated from this plot, and the values of density ρ, radius a, heat of explosion Q, thermal conductivity λ, the gas constant R, and a shape factor σ; a critical temperature T_m, is calculated from the following equation:

$$T_m = \frac{E}{2.303\ R \log \frac{\rho a^2 QAE}{\lambda R T_m^2 \sigma}}$$

The critical temperature is the maximum temperature above which the temperature in the propellant charge of radius a is predicted to rise continuously. For example, a composite propellant, ANB-3066, gives a first exotherm at 10°C per minute of 505°F, and at 1.2°C per minute of 450°F. These correspond to an activation energy of 38.6 kcal/mole. When these data were used to calculate T_m under isothermal conditions, values obtained were 285°F for a 5 in. diameter solid cylinder and 168°F for a 260 in. diameter motor. A test on a 6.5 in. diameter cylinder held at 320°F showed the surface temperature of the cylinder rising above the oven temperature after 18.5 hours.

The test depends on an analysis of the DTA data by the Kissinger method (reference 3) which appears valid for most propellant cases, inasmuch as the temperature increases to a maximum without showing any decreasing rate of temperature increase.

2-4.4 TEST ANALYSIS AND LIMITATIONS. The evaluation of kinetic constants on a larger scale than the DTA test is desirable in order to decrease the amount of extrapolation needed to predict the hazard for large motors by providing values at lower temperatures. In the case of the method which utilizes an isothermal environment, knowing the rate of temperature rise at that time when no temperature gradient exists at the center, allows calculation of activation energy, as does also measurement of the thermal gradient when equilibrium is established in the bath; provided, in this latter case, that the right bath temperature has been selected.

The self-heating tests described here pertain only to finished, cured homogeneously mixed propellant. A further limitation is that one exothermic reaction must predominate and that it not be complexed with a simultaneous endotherm (for example a crystalline form change in the oxidizer). This is necessary in order to obtain sufficiently clearcut datum points to permit plotting of the curve for activation energy. The limitation of these self-heating tests to cured, homogeneous propellants without voids rules out their use for one of the most serious propellant processing hazards; namely, the destructive exothermic effect that results from high local concentration of reactive species during mixing (for example, high local concentrations of polymerization accelerators or burning rate catalysts with oxidizer). These high local concentrations can result from such factors as agglomeration, improper order of addition of components to the mixer, "dead spots" in the mixer movement, etc. The only known testing method for assessing this high local concentration hazard is to deliberately put together the anticipated mal-mixture and to test for exothermic effect at various concentrations by conventional DTA techniques.

2-4.5 APPLICATION TO HAZARD ANALYSIS. The critical temperature can be used as a value for the absolute maximum storage temperature before fire or explosion can occur. This would be useful information during exploratory work on a formulation. Exotherms from DTA studies are used in this prediction of critical temperature. However, the first DTA exotherm may not necessarily be the one that controls the deflagration. It should be noted, of course, that measureable degradation of the propellant occurs below T_m, and is evident at the onset temperature of the first exotherm.

Where the use of the rocket would expose the propellant to high temperatures of short duration, the parameters determined in the self-heat studies can be used to calculate whether the propellant decomposition reaches a stage where progression to deflagration is inevitable; however, the cumulative decomposition effect from repeated temperature cycling below deflagration would not be readily calculable. The methods described above can be used to predict when a grain of a certain geometry and composition will begin to self-heat from the chemical reactions taking place within, even at normal ambient temperature. The result of the study can be checked through isothermal analysis of samples of selected sizes. This type of study would aid in the determination of the thermal environment limitations for a very large grain, for example first-stage solid space boosters.

2-5 COPPER BLOCK TEST

2-5.1 OBJECTIVE. To express the autoignition temperature of the substance by an empirical test method.

2-5.2 OPERATING PRINCIPLE. A small sample is heated at a constant rate in a fixed environment until ignition occurs. The temperature of ignition is observed.

2-5.3 TEST DESCRIPTION. A 0.1 gram sample is placed in a well in a copper block, which is placed in a furnace of sufficient capacity to raise the temperature of the block from ambient at a controlled rate of 20 ± 5°F per minute to a level sufficiently high to assure ignition of the sample. A similar well in the copper block contains a thermocouple connected to a pyrometer of suitable temperature range. The average of five ignition temperatures is recorded as the autoignition temperature in °F.

2-5.4 TEST ANALYSIS AND LIMITATIONS. This test is a simple, economical method for expressing the comparative stability of substances to heat. However, the autoignition temperatures obtained would differ in most cases if a different rate of heating were used.

Also, it makes no provision for detecting more than one decomposition reaction, successive endotherms or exotherms, etc. Accordingly, the test is not nearly as informative as autoignition temperatures determined in the DTA.

2-5.5 APPLICATION TO HAZARD ANALYSIS. The autoignition temperature from the copper block could be used as an upper temperature limit for propellant processing or storing, but only with a large safety margin to allow for the uncertain rate-of-heating and self-heating effects.

2-6 WENOGRAD TEST

2-6.1 OBJECTIVE. To determine the temperature of explosion under conditions of minimal heat transfer effects (reference 4).

2-6.2 OPERATING PRINCIPLE. A small sample of the explosive is heated by electrical capacitor discharge. Electronic recording of the temperature at which explosion occurs is achieved within 20 microseconds by means of an oscilloscope.

2-6.3 TEST DESCRIPTION. The sample is contained in a stainless steel tube of hypodermic needle size. The coefficient of electrical resistance of the tube is known. Heating is furnished by discharging a high-level capacitor through the tube, which triggers an oscilloscope for recording the emf via a Wheatstone bridge circuit. From the amplitude of the oscilloscope trace, the voltage of the tube and consequently its resistance and temperature can be computed. The explosion of the sample ruptures the tube and breaks the oscilloscope trace. Response time of the test is reported to be of the order of 20 microseconds, to the recording of the final temperature of the tube, i.e., the temperature of explosion.

2-6.4 TEST ANALYSIS AND LIMITATIONS. Thermal test methods described previously all have an appreciable heat transfer factor, such that recorded times and temperatures cannot be regarded as a true integral of the total heat input to the entire mass of the sample. In an effort to minimize this heat transfer lag, Wenograd devised an ohmic heating mechanism for a very small sample, with instrumentation for an electronic response triggering mechanism and temperature recording.

In a critical review and confirmation of the Naval Ordnance Laboratory's test result with the Wenograd test, workers at Stanford Research Institute affirmed that the test measures a "true" induction time for the explosive, rather than the time required for a physical effect, such as heatup of the sample (reference 5). Explosion times in the Wenograd test are believed to have the same characteristic times as response in the impact test. The order of numerous explosives correlates well in the two tests.

2-6.5 APPLICATION TO HAZARD ANALYSIS. Initiation-to-explosion temperatures determined in the Wenograd test would probably be the closest approximation to a "point source" heat initiation of an explosive component in a propellant mix, for example, one corner of an oxidizer crystal under stress. Accordingly, if the frictional or impact stress at the "point source" can be calculated in the Process Survey phase of the Hazards Analysis, it would most appropriately be balanced against the Wenograd determination of explosion temperature in the Sensitivity Testing phase of the Analysis.

2-7 TALIANI TEST

2-7.1 OBJECTIVE. To gauge the temperature sensitivity of materials by measurement of the gas evolution pressure.

2-7.2 OPERATING PRINCIPLE. The sample is placed in an enclosed system and is maintained at a constant elevated temperature in a heating block. The pressure and pressure-change-rate are plotted at fixed time intervals.

2-7.3 TEST DESCRIPTION (reference 6). The apparatus (reference 7) consists of a heating block formed from a machined aluminum cylinder wound with resistance wire and controlled by a Fenwal thermoregulator. A one gram sample of the material under test is weighed into a sample tube, which is connected via a glass helix and a 3-way stopcock to vacuum, a manometer, and a nitrogen source. The tube is purged by means of the vacuum and nitrogen, is brought to a steady temperature state with the heating block (15 minutes to 110°C, normally) and is adjusted to atmospheric pressure. Heating is continued for six hours or to a pressure increment of 150 mm Hg. A plot of differential pressure versus time is made, and the values reported are the slope of 100 mm, the minutes to 100 mm, and the slope at 100 minutes.

2-7.4 TEST ANALYSIS AND LIMITATIONS. The test is most useful as a sensitivity criterion for double-base propellants and nitrocellulose. It has questionable utility for compounds containing the nitro or any other plosophoric groups except the nitrato group.

2-7.5 APPLICATION TO HAZARDS ANALYSIS. Because this test is so intimately associated with nitrocellulose-based products, its application in hazards analysis would be limited to comparative studies with other double-base propellants that have been successfully produced in the past.

2-8 STANDARD HEAT TESTS (reference 6)

2-8.1 OBJECTIVE. To evaluate thermal stability by loss in weight at an arbitrary fixed temperature.

2-8.2 OPERATING PRINCIPLE AND TEST DESCRIPTION. A number of empirical heat tests can be used, adapted principally from the high explosives and pyrotechnics industries, e.g., the 75°C International Test and the 100°C Heat Test. In the Standard Heat Test, for example, a 0.6 g sample is heated in an open test tube at 100°C for 96 hours. To qualify, an arbitrary value may be set, such as, the sample must lose less than 2% wt. in 48 hours.

2-8.3 TEST ANALYSIS, LIMITATION, AND APPLICATION TO HAZARDS ANALYSIS. As with most of these empirical tests, the results are meaningful only in comparison with substances of established sensitivity that have been tested in the past. The hazards analysis of manufacture of a new product could thus be comparatively expressed in this manner.

2-9 KI-STARCH TEST

2-9.1 OBJECTIVE. To evaluate the thermal stability of nitrato compounds by their time to react with a standard indicator paper.

2-9.2 OPERATING PRINCIPLE AND TEST DESCRIPTION. The potassium iodide-starch indicator paper test is conducted at 82.2°C for nitroglycerin and at 65.5°C for double-base propellants and nitrocellulose. Details on the sample weights, the heating bath, and the sample and test tube preparation are published in Picatinny Arsenal standard procedures (reference 6). The test paper is moistened with a glycerin-water solution and the time to coloration is reported. Standard grades of military nitrocellulose are required to have a 65.5°C KI test value of 35 minutes minimum.

2-9.3 TEST ANALYSIS AND LIMITATIONS. The test is most useful as a sensitivity criterion for double-base propellants and nitrocellulose. It has questionable utility for compounds containing nitro or any other plosophoric groups except nitrato.

2-9.4 APPLICATION TO HAZARDS ANALYSIS. Because this test is so intimately associated with nitrocellulose-based products, its application in hazards analysis would be limited to comparative studies with other double-base propellants that have been successfully produced in the past.

2-10 METHYL VIOLET TEST

2-10.1 OBJECTIVE. To evaluate the thermal stability of nitrato compounds by the time to react with standard indicator paper.

2-10.2 OPERATING PRINCIPLE AND TEST DESCRIPTION. Methyl violet indicator paper testing is done at 120°C for propellants and at 134.5°C for double-base and nitrocellulose compositions. Samples of 2.5 g are dispensed in Pyrex test tubes with standard indicator paper. Test details on the tubes, heating block bath, and immersion of the tubes and test paper are published by Picatinny Arsenal (reference 6). Samples are heated up to five hours and times are recorded to paper coloration, evolution of red fumes, and/or explosion of the sample.

2-10.3 TEST ANALYSIS AND LIMITATIONS. The test is most useful as a sensitivity criterion for double-base propellants and nitrocellulose. It has questionable utility for compounds containing nitro or any other plosophoric groups except nitrato.

2-10.4 APPLICATION TO HAZARDS ANALYSIS. As with most of these empirical tests, the results are meaningful only in comparison with substances of established sensitivity and manufacturability that have been tested in the past. The hazards analysis of manufacture of a new product could thus be comparatively expressed in this manner.

2-11 IMPACT TEST

2-11.1 OBJECTIVE. To determine the likelihood that an explosive will initiate if it impacts against a moving mass.

2-11.2 OPERATING PRINCIPLE. A sample of the explosive is crushed between a fixed anvil and a movable hammer. Impact energy is usually supplied to the hammer by a constant mass - variable drop height falling weight.

2-11.3 TEST DESCRIPTION. This test simulates impact conditions in processing operations wherein an explosive material is subjected to collision between moving components of the processing equipment, by normal handling operations, or by the inadvertent dropping of tools, bolts, etc.

This test involves placing the sample on a fixed anvil in a restricted but unconfined state. The sample thickness is similar in dimension to that occurring in processing. A hammer of known contact area is positioned above the sample and the weight is raised to a predetermined height and dropped. Normally, sample initiation is detected by audible, visual, sensory means or by infrared analysis (reference 9) of selected decomposition products. A test is comprised of twenty "no initiation" trials at a given input and at least one initiation at the next higher interval (i.e., 25% greater energy input). The energy delivered to the sample is measured with a force gage (reference 9). Figure 2-2 illustrates the impact machine concept while Figure 2-3 illustrates the attendant instrumentation for analyzing the impact energy.

2-11.4 TEST ANALYSIS AND LIMITATIONS. Data from an impact test are normally given as the product of the falling weight and drop height of the drop weight. However, it can be shown that because of nonideal collisions between the drop weight and the impact hammer, only a fraction of the drop weight energy is transmitted to the sample.

To determine the energy loss, a force gage is employed to measure the force-time history of an impact test. The data from this technique are treated by the method given in reference 9. Results are normally reported as energy per unit area or energy per unit area per unit time (ft-lb/in.^{2} or ft-lb/in.^{2}-sec). These units can be converted to any other desired units. The present apparatus is limited to maximum energy output rate of approximately 1.0×10^6 ft-lb/in.^{2}-sec. The machine output could be increased by minor redesign of drop weight and impact hammer. This test can be and has been employed to study the effects on impact sensitivity of such environmental factors as material of fabrication, sample thickness, impacted area, surface finish, surrounding atmosphere, temperature, and pressure.

2-11.5 APPLICATION TO HAZARD ANALYSIS. A process is examined for conditions in which impact could occur, and the magnitude and rate of energy inherent in the process are measured by the force gage in the same terms as the initiation testing. With the values of

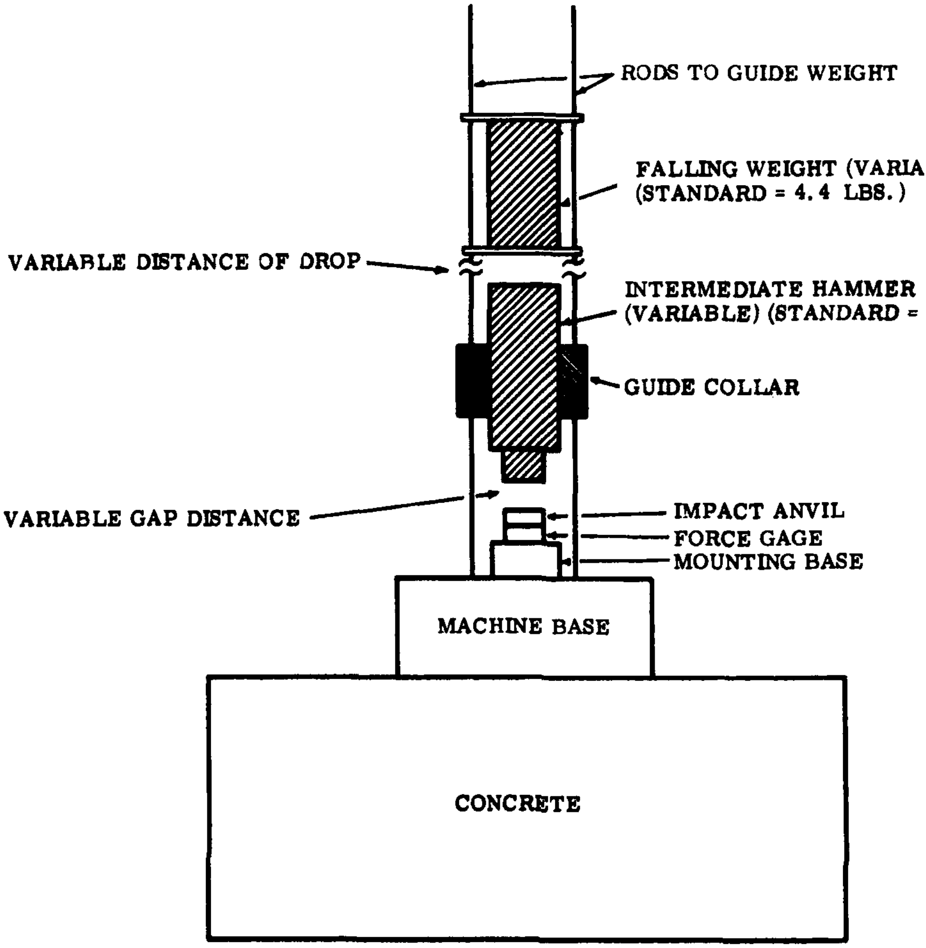

Figure 2-2 Principles of Laboratory Impact Machine

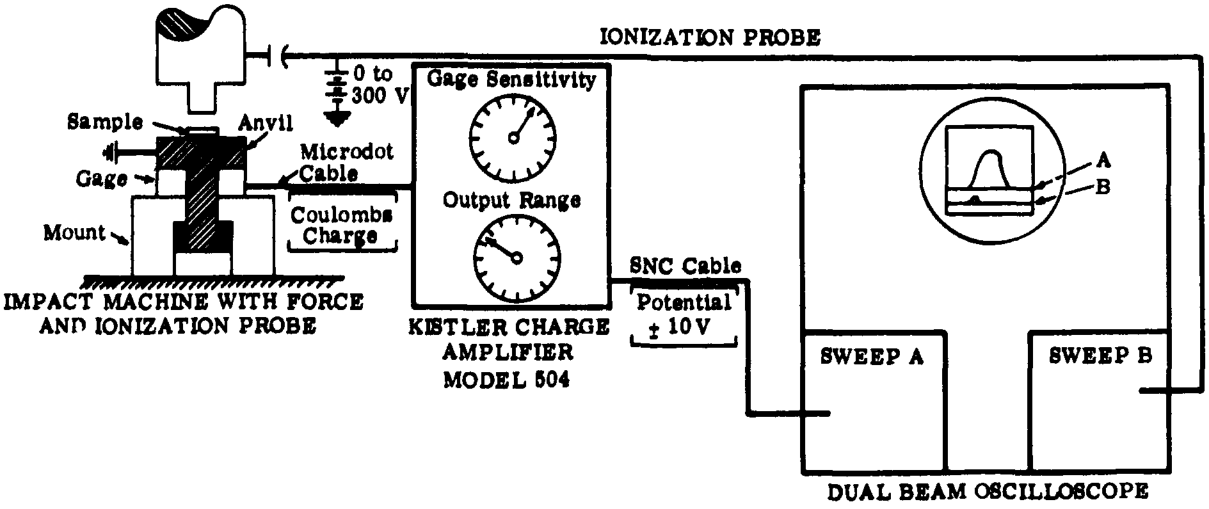

Figure 2-3 Force Gage and Ionization Probe System for Measuring Impact Energy and Initiation Time in Impact Initiation of Explosive Materials

energy required for initiation and the energy available in the process, a judgment may be made as to whether the process constitutes an impact initiation hazard. Should a hazard be found, the process can be modified to reduce the available impact energy, thus assuring an adequate margin of safety for the process.

2-12 FRICTION TEST

2-12.1 OBJECTIVE. To determine the likelihood that a sample will initiate when subjected to force at a given velocity.

2-12.2 OPERATING PRINCIPLE. Force is applied to a sample resting on an anvil. The anvil is propelled perpendicular to the force vector at a known velocity.

2-12.3 TEST DESCRIPTION. This test simulates friction conditions occurring in the process when an explosive is subjected to a frictional force between moving components, such as in the mixer, or during disassembly, machining and material handling. The test is normally carried out as shown in Figure 2-4. The sample is placed on the anvil, a known force is applied hydraulically through a stationary wheel, and a pendulum is used to propel the sliding anvil at a known velocity perpendicular to the force vector. Normally, sample initiation is detected by audible, visual, sensory means or by infrared analysis (reference 9) of selected decomposition products. A valid test is comprised of twenty consecutive "no initiations" and at least one initiation at the next higher test interval. Force levels are decreased by 25% intervals, and velocity levels are reduced 1 ft/sec whenever an initiation is obtained at 10 lb force at a given velocity. Force is measured by hydraulic gauges and velocity by a sliding potentiometer.

2-12.4 TEST ANALYSIS AND LIMITATIONS. Reported values are the calibrated velocity and the final force gage reading. The data are reported as lb-force or lb-force/in.^{2} at 3 to 15 ft/sec. Present force and velocity limitations on this test are 4 to 1800 lbf and 3 to 15 ft/sec, respectively. Compressional strength of the explosive samples and fabrication materials tested can also limit the maximum force employed.

This test also examines, where appropriate, the effects on friction sensitivity of (a) materials of fabrication, (b) surface finish, (c) slide length, (d) contact area, and (e) velocity. The coefficient of friction can also be obtained for test and fabrication materials.

2-12.5 APPLICATION TO HAZARD ANALYSIS. A process operation is analyzed for the possible friction forces and velocities occurring between contacting components during processing operations. These values are then compared to threshold friction initiation values, then a hazardous condition exists. This hazardous condition can be eliminated by modifying the material, the procedure, or the process, usually through reduction of the potential forces and velocities to obtain an adequate margin of safety.

2-13 ELECTROSTATIC DISCHARGE

2-13.1 BASIC ELECTROSTATIC DISCHARGE TEST.

2-13.1.1 Objective. To determine whether an electrostatic discharge will initiate an energetic material.

2-13.1.2 Operating Principle. Electrostatic energy stored in a charged capacitor is discharged to the sample material to be tested. Materials initiated below 0.015 joule are considered hazardous for direct handling since this value is approximately that which individuals can generate. In this case the Human Spark discharge test (see section 2-13.2) is employed.

2-13.1.3 Test Description. The electrostatic test apparatus is shown in Figure 2-5. The sample to be tested is placed on a special holder which assures that electrostatic discharge will pass through the sample. A capacitor is charged with a 5000 volt potential. The discharge needle is lowered until a spark is drawn through the sample. Twenty consecutive failures at a given discharge energy level with one initiation at the next higher input constitutes an electrostatic discharge sensitivity test. A new sample is used for each trial.

2-13.1.4 Test Analysis and Limitiation. The electrostatic energy discharge to the test specimen is calculated from the relationship $E = 1/2\ CV^2$ where E = energy (joules), C = electrical capacitance (farads), and V = charging potential (volts). Currently, electrostatic discharge data do not reflect energy losses in conductors, discharge gap, and resistivity of the test specimen. These factors lower the discharge energy and the rate at which the energy is delivered. In addition, this type of test method does not provide for testing at different discharge rates to simulate all possible process conditions.

2-13.1.5 Application to Process Hazards Analysis. Electrostatic discharge sensitivity data are important for propellant manufacturing operations which involve the processing of fine or granular materials and materials considered to be static sensitive. To establish whether an adequate safety margin is present, the electrostatic energies occurring in the process are defined and compared to the electrostatic sensitivity of the material being handled or processed. If adequate safety is not demonstrated, the electrostatic discharge apparatus can be used to verify the effectiveness of recommended processing procedures (such as the use of material coatings or of moist and/or ionizing atmospheres) to eliminate the electrostatic hazard.

2-13.2 ELECTROSTATIC DISCHARGE HUMAN SPARK TEST.

2-13.2.1 Objective. To establish the susceptibility of an energetic material to initiation when subjected to the electrostatic discharge generated by humans.

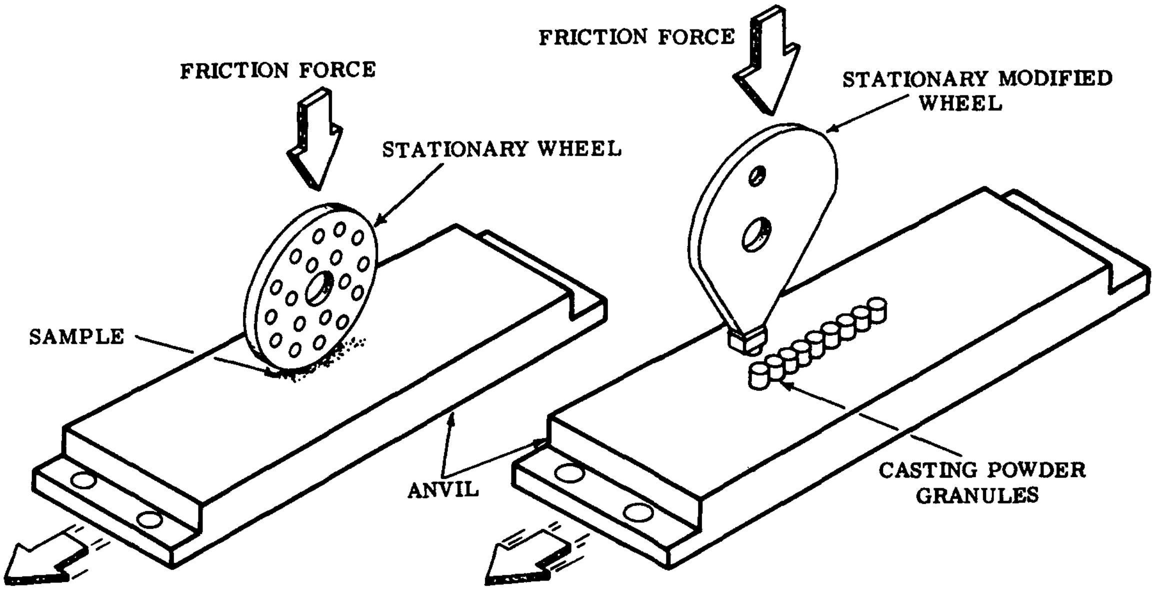

Figure 2-4 Operating Principle of Friction Test Apparatus

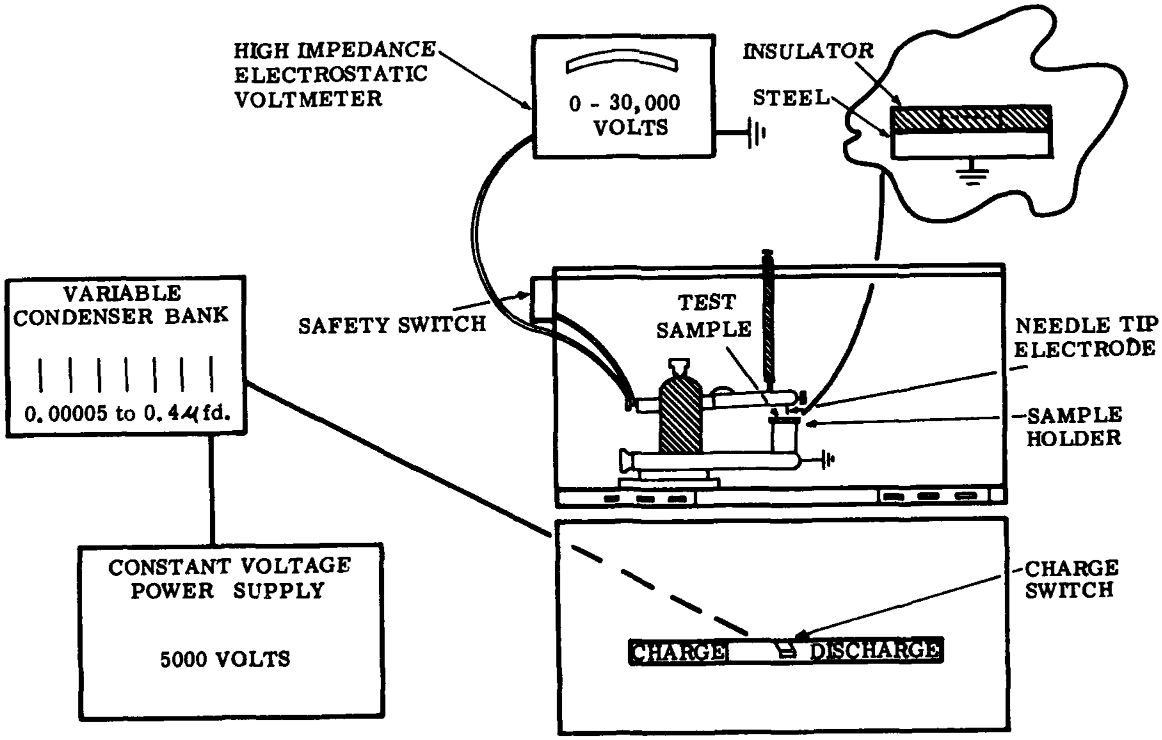

Figure 2-5 Electrostatic Discharge Spark Test Apparatus

2-13.2.2 Operating Principle. Electrostatic energy accumulated on an electrically isolated human is discharged to the sample to be tested.

2-13.2.3. Test Description. The experimental test arrangement for establishing the human discharge spark sensitivity for static sensitive material is shown in Figure 2-6. The test consists of subjecting materials to an electrostatic discharge spark by charging, then discharging a human subject at various voltage levels using a variable gap until the level is established where no initiation results in 20 consecutive trials. This failure level is defined as the threshold human spark initiation level and is reported in terms of voltage.

2-13.2.4 Test Analysis and Limitations. The electrostatic energy discharged to the sample is calculated by applying the relationship $E = 1/2\ CV^2$ where E = energy (joules), C = capacitance (farads), and V = charging potential (volts). An average capacitance for the human body is 300 picofarads. This technique does not allow for the different discharge rates that would be available from different individuals because of varying skin or contact resistances.

2-13.2.5 Application to Process Hazards Analysis. The presence of the human body in propellant manufacturing areas is considered a potential electrostatic hazard when static-sensitive materials are being processed. To determine the existence of hazards due to human spark discharge, the human spark sensitivity data are compared to the maximum energy that can be generated by humans in a processing operation. The possibility of electrostatic charges accumulating on humans is reduced by requiring periodic checks on safety shoes, floors, and equipment to ensure conductivity. When possible, materials or processing procedures are modified to eliminate the potential human spark hazard by using coatings, particle modification, ionizing and/or moist atmospheres and remote operations. Where an adequate safety margin cannot be demonstrated for handling and processing of a given static-sensitive material, the material should be eliminated from the process.

2-13.3 ELECTROSTATIC HAZARDS ANALYSIS.

2-13.3.1 Objective. To quantitatively assess electrostatic discharge initiation hazards during handling and manufacturing activities involving combustibles, explosives and solid propellant materials.

2-13.3.2 Operating Principle and Hazard Criteria. The Systems Engineering Approach to Hazards Analysis is used to make realistic estimates of the electrostatic hazard associated with handling or processing sensitive material. The response of materials to electrostatic discharge stimulus is determined initially in suitable tests which yield the "no initiation" energy (joules). This threshold value is compared to the electrostatic energy possible during manufacture determined by appropriate in-process measurement and/or theoretical calculations. Operations are considered hazardous when the electrostatic energy potential during the suspected operation exceeds the threshold initiation level for the subject material. The human body can precipitate an electrostatic discharge hazard when the material is initiated by electrical discharges less than 0.015 joule. Assessment of this situation requires employment of the Human Spark Discharge tests. (section 2-13.2).

2-13.3 Sensitivity Test Analysis. Initiation test equipment is used to determine the material initiation characteristics under the processing environmental conditions.

2-13.3.4 Engineering Analysis. Engineering analysis consists of experimental and theoretical procedures to establish the magnitude of electrostatic charges possible in processing equipment, humans or the material itself.

In-process tests employ suitable instrumentation and sensors to establish equipment capacitance and the magnitude of voltage generation and accumulation in the system for normal and the ungrounded abnormal processing situations. This test is usually made with inert materials having comparable physical properties, but with electrical characteristics (conductance, voltage generation and accumulation, etc.) exceeding those of the subject material. The measured voltage and capacitance parameters are substituted in the standard equation $E = 1/2\ CV^2$ where E = energy (joules), C = capacitance (farads), and V = voltage (volts) and the electrical energy capacity of the system or operation is determined.

Theoretical and simulated test procedures are employed when process design does not permit equipment capacitance determinations or when scheduling or economy does not permit detailed in-process measurements. Standard equations exist for capacitance determinations for different geometrical bodies (reference 10). Simulated tests are employed to confirm a "no initiation" hazard for a particular equipment piece or in-processing situations with low margins of safety. In these tests, processing equipment and/or humans are isolated from ground and a known "charge potential" discharged from the body to the subject material.

2-13.3.5 Analysis Limitations. The simulated in-process tests are a more realistic analysis since the experiments take into account "energy losses" attributable to resistance (material, air gap, lines, etc.) and inductance and duplicate the rate energy is applied to the sample for a particular equipment piece or body. The threshold electrostatic discharge level obtained using the spark test apparatus in Figure 2-5 may be conservative if inductance and resistance generally are less than the environment under consideration. To be totally applicable the electrostatic discharge test equipment circuitry should duplicate these variables in the discharge path. This more costly and more extensive test procedure is needed only when the materials are known to be borderline or when analysis shows that a hazard truly exists.

2-14 IMPINGEMENT TEST

2-14.1 OBJECTIVE. To determine whether an explosive, moving at some velocity and striking an object, will initiate.

2-14.2 OPERATING PRINCIPLE.

2-14.2.1 Propelled. The sample is propelled by air (or any other fluid) at a known velocity and impinged (collided) against a massive target.

2-14.2.2 Free Fall. The sample is dropped in free fall through a known distance onto an impingement block.

2-14.3 TEST DESCRIPTION. The propelled test, Figure 2-7, involves injecting a test sample into a high-velocity air stream or dropping it in free fall, Figure 2-8, 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.

2-14.4 TEST ANALYSIS AND LIMITATIONS. Data from this test are reported in terms of impingement velocity (fps). 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 (reference 11)also allow the kinetic energy of impingement to be measured.

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 1100 fps).

2-14.5 APPLICATION TO HAZARD ANALYSIS. During propellant manufacture, liquid and solid explosives are normally moved by fluidized transport. It is therefore mandatory to know if operating conditions during a particular transfer operation can produce energy sufficient to initiate the material handled.

The process is examined to determine the conditions and velocities present in transfer operations. These velocities are then compared to the impingement threshold velocities established during initiation testing. If a transfer velocity in a process operating exceeds the impingement threshold velocity, then a hazardous condition exists.

If a hazardous condition does exist, the transfer process must be modified to secure an adequate margin of safety. This is best accomplished by reducing transport velocities through flow obstructions or by using shorter conveying distances.

2-15 THIN FILM PROPAGATION TEST

2-15.1 OBJECTIVE. To determine whether a thin film of explosive material, once initiated, can transit to a more intense reaction and consume the material at this increased rate.

2-15.2 OPERATING PRINCIPLE. A liquid explosive of varying thicknesses simulating process conditions is initiated by impact, and the extent and rate of reaction are established by monitoring the pressure front accompanying the explosive reaction.

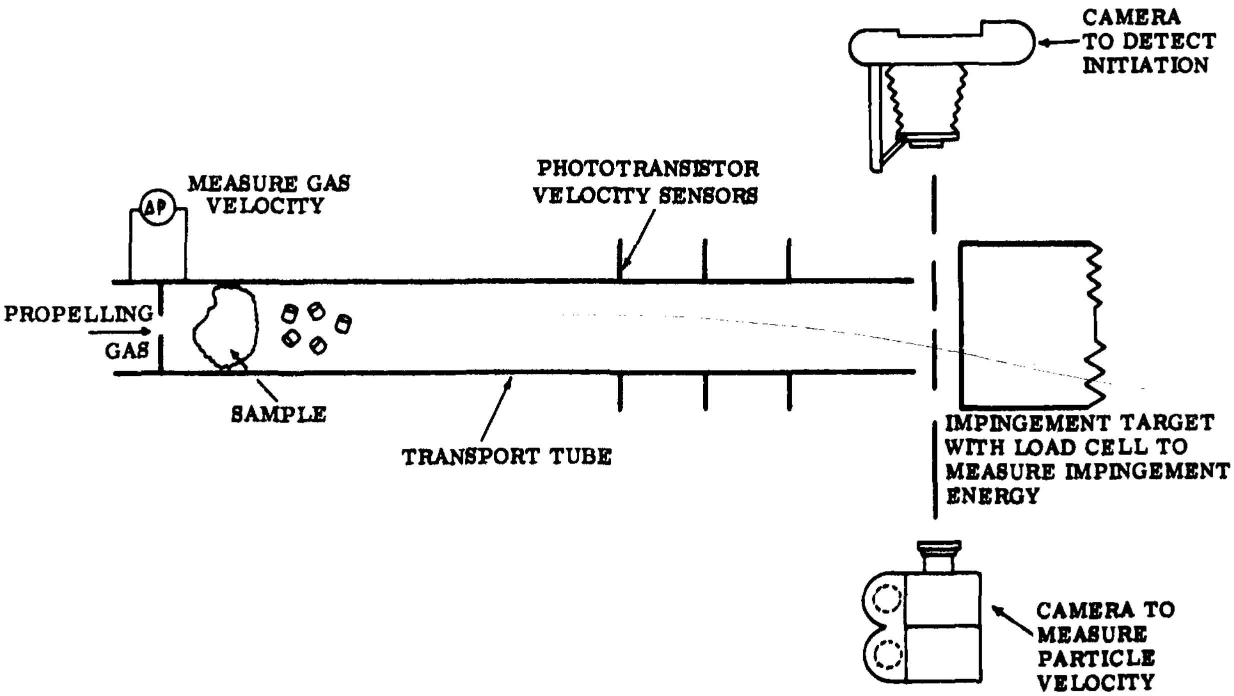

Figure 2-7 Liquid and Solid Explosive Impingement Test Apparatus

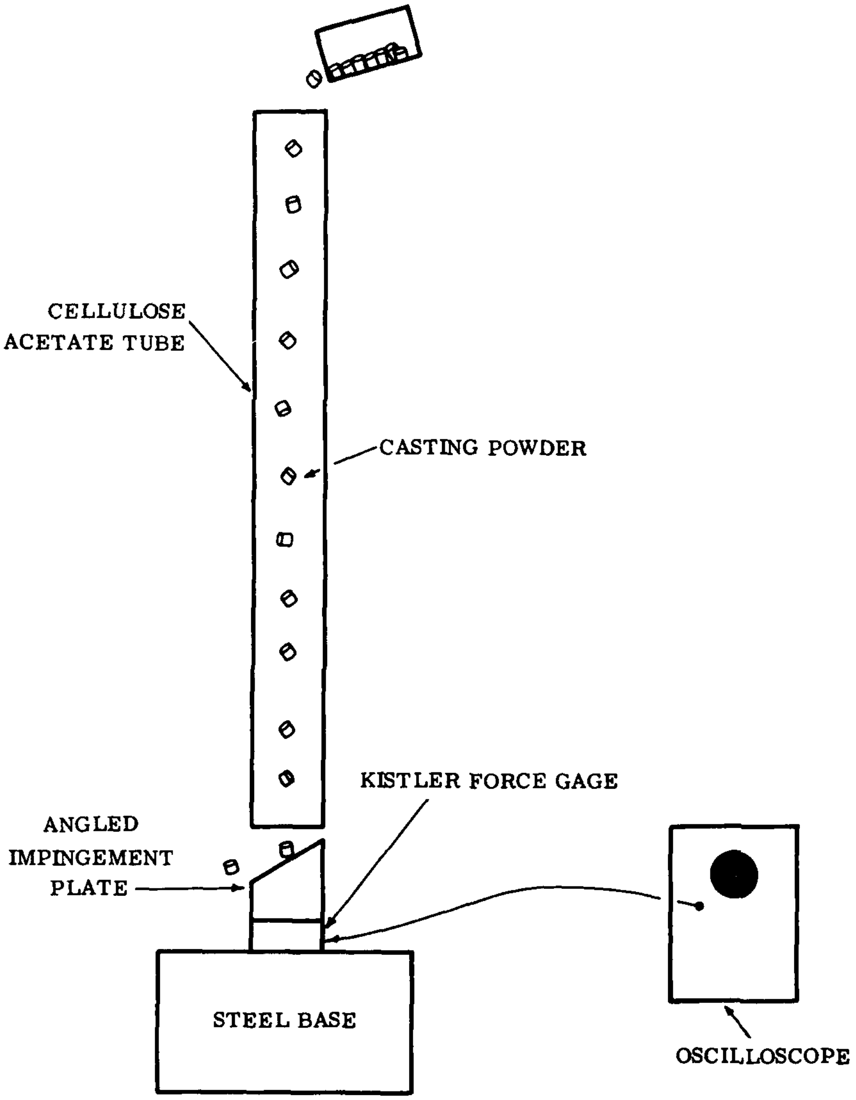

Figure 2-8 Experimental Arrangement for Free-Fall Impingement Test

2-15.3 TEST DESCRIPTION. The unconfined thickness of the material to be tested is measured under process conditions, and the propagation test is conducted in a sample holder (Figure 2-9) approximating this material thickness (both puddle and propagation slot.) A resistance wire probe (Figure 2-10) is the pressure sensor for following the extent and velocity of propagation. An impact machine is used to initiate the explosive contained in the puddle which in turn furnishes the energy for further explosive propagation. Maximum initiation of the sample in the "puddle" is required for a valid trial, and any probe reaction beyond that caused by the initial reaction is classified as a propagation. A test consists of twenty trials.

Previous work has shown the sample holder material does not influence the propagation; further, once a maximum amount of the "puddle" has been initiated, the impact energy will not affect propagation. The test has shown that a critical dimension for propagation exists (i.e., a propagation slot width or depth below which a propagation does not occur).

2-15.4 TEST ANALYSIS AND LIMITATIONS. Data from this test give the frequency and extent of propagation of reaction from an impact initiated sample. Also, the instrumentation employed gives the extent of a propagation as a function of time, thereby yielding propagation velocity. A typical set of data could be:

Propagation frequency	66%
Propagation extent	2 inches
Propagation velocity	1200-1500 meters/sec.

This test can be used to determine not only the effects of sample dimensions but also of energy input and materials of fabrication on the extent and velocities of the propagating reaction. Use of the test is limited to testing those materials reacting strongly enough to activate the pressure-sensitive probe system.

2-15.5 APPLICATION TO HAZARD ANALYSIS. In those cases where accidental initiation of reaction cannot be positively eliminated, the consequences of this initiation must be determined. If initiation results in a fire, then preparations can be made to control and to eliminate the fire without excessive damage to facilities. If, however, the result of an initiation is an explosion or detonation, then little, if anything, can be done to prevent damage to immediate facilities. Therefore, at an early stage in a process, all high energy liquids are examined to determine if propagation results from initiation for conditions such as thickness and energy input found in the process. If such is the case, the material or process may be rejected or modified to obtain an acceptable safety margin for processing.

2-16 DUST EXPLOSIBILITY TEST

2-16.1 OBJECTIVE. To determine whether a finely divided solid material will react explosively when dispersed in a gaseous medium and ignited.

2-16.2 OPERATING PRINCIPLE.

2-16.2.1 Threshold Dust Concentration. Finely divided explosive dusts are dispersed into an energy source in air to determine the threshold explosive dust concentration.

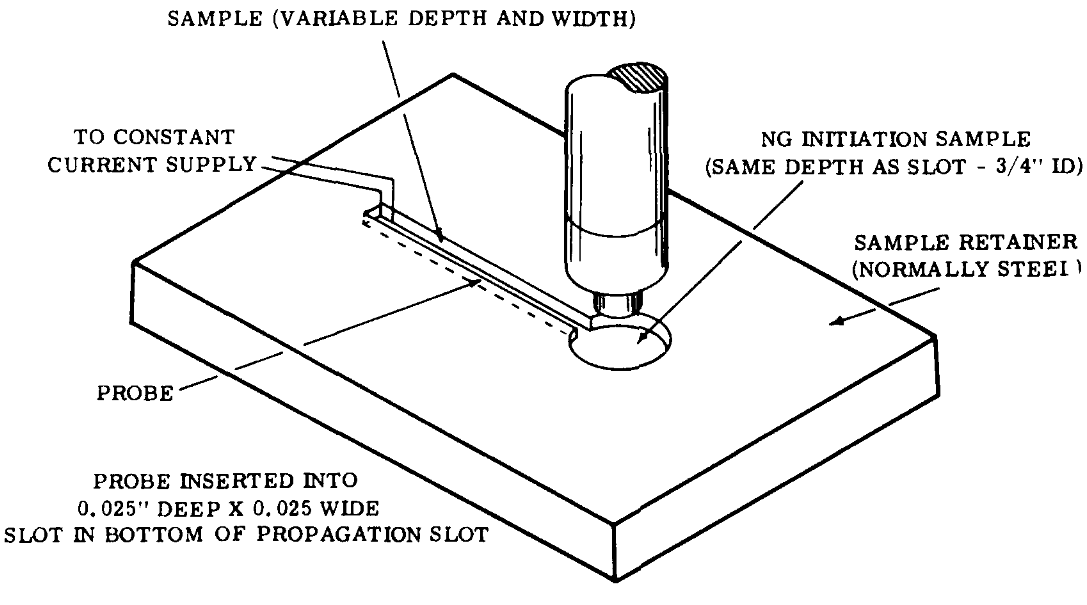

Figure 2-9 Thin Film Propagation Test

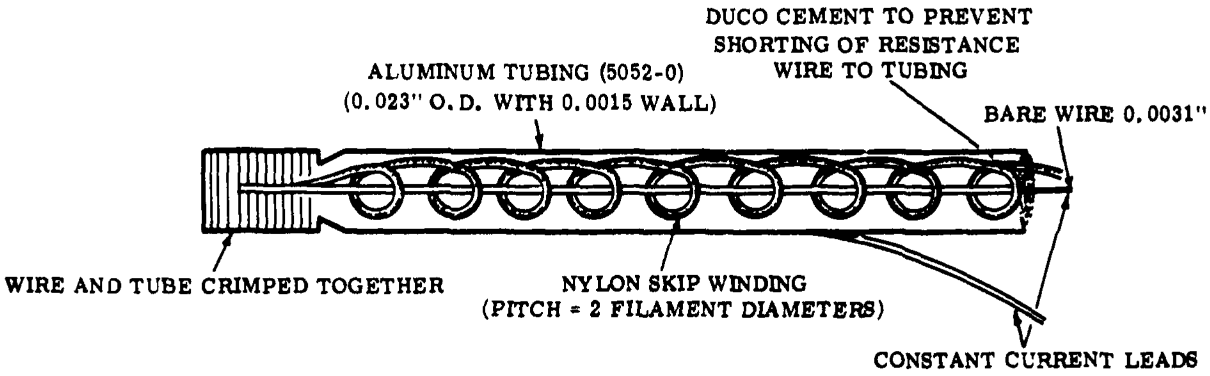

Figure 2-10 Resistance Wire Probe

2-16.2.2 Threshold Electrostatic Discharge. Dust air mixtures in the explosion range are subjected to condenser discharge sparks to determine the threshold electrostatic discharge energy for highly explosive dust-laden atmosphere.

2-16.3 TEST DESCRIPTION. The Bureau of Mines "Hartmann" Dust Explosibility Test Apparatus (reference 12) employed for defining the explosibility properties of finely divided materials is shown in Figure 2-11. Basically, momentary dust clouds are produced in a Lucite combustion tube by a single blast of air (or inert gas) and ignited. To determine the threshold explosion concentrations, known dust sample weights are dispersed into a continuous, high-voltage-spark, high-energy source. Sample material, physical and chemical properties and environmental conditions encountered during processing are duplicated during testing. Following dust ignition, an explosive reaction is detected if the pressure is sufficient to rupture a filter paper diaphragm. Sample weight is varied until a dust concentration is obtained for which no explosion occurs in four consecutive trials.

The test for threshold electrostatic discharge initiation is similar to the above except that a dust sample in excess of the explosive limit is utilized and an electronic timer controls the electrostatic discharge during dust dispersal. Electrostatic discharge energy is varied until an energy level is obtained for which no explosive reaction occurs in four consecutive trials.

2-16.4 TEST ANALYSIS AND LIMITATION. Data from this test are reported as threshold dust concentrations (oz/ft 3), meaning the concentration of dust must exceed this level to explode, and the threshold electrostatic discharge energy (joules), if exceeded, will initiate an explosive reaction in the dust/air mixture.

Test capabilities permit establishing explosibility characteristics of a material under different media for evaluating compositional effects, and the relative benefits of inert gases or additives for eliminating a dust explosion. The test yields relative values which are influenced by such factors as the chemical and physical properties of the dust, uniformity of the dust cloud, properties of the atmosphere, ignition source and environmental conditions.

2-16.5 APPLICATION OF PROCESS HAZARDS ANALYSIS. Establishment of dust concentrations in processing system is impractical because dust concentrations and composition change constantly. For this reason and the others cited above, conditions for potential dust explosion are assumed to be present when the process hazards observational survey shows that significant dust accumulation accompanies the processing material. Application of dust explosibility data to propellant manufacturing operations increases safety by establishing (a) the need and extent for dust removal systems and (b) the requirements for an inert atmosphere (type and concentration) when dust removal is ineffective or when materials are particularly sensitive.

2-17 CRITICAL HEIGHT TO EXPLOSION (TRANSITION) TEST

2-17.1 OBJECTIVE. To determine if a material will react explosively when initiated by flame.

2-17.2 OPERATING PRINCIPLE. Materials are submerged and surface flame initiation under environmental process conditions to determine if a material will react explosively for a specific operation.

2-17.3 TEST DESCRIPTION. The experimental test arrangement for determining the critical height to explosion or detonation for propellant materials is shown in Figure 2-12. A suitable container (generally schedule 40 pipe), charged with the sample material to be tested, is subjected to bottom flame initiation produced by a 12 gram bag igniter. Top or partially submerged ignition is also employed where applicable. Environmental processing conditions common to the sample material such as loading density, temperature, etc., are duplicated during testing.

Criteria for ascertaining that transition from slow burning to an explosion has occurred in a particular test trial can be established on the basis of container damage or interpretation of the reaction velocity profile. The container damage criterion consists of testing at different material heights until a material height is used at which no container damage results for three trials for a given material height and charge diameter. This approach, however, requires numerous trials (high cost), assumes no difference in rupture strength between test containers and fittings, and yields no in-

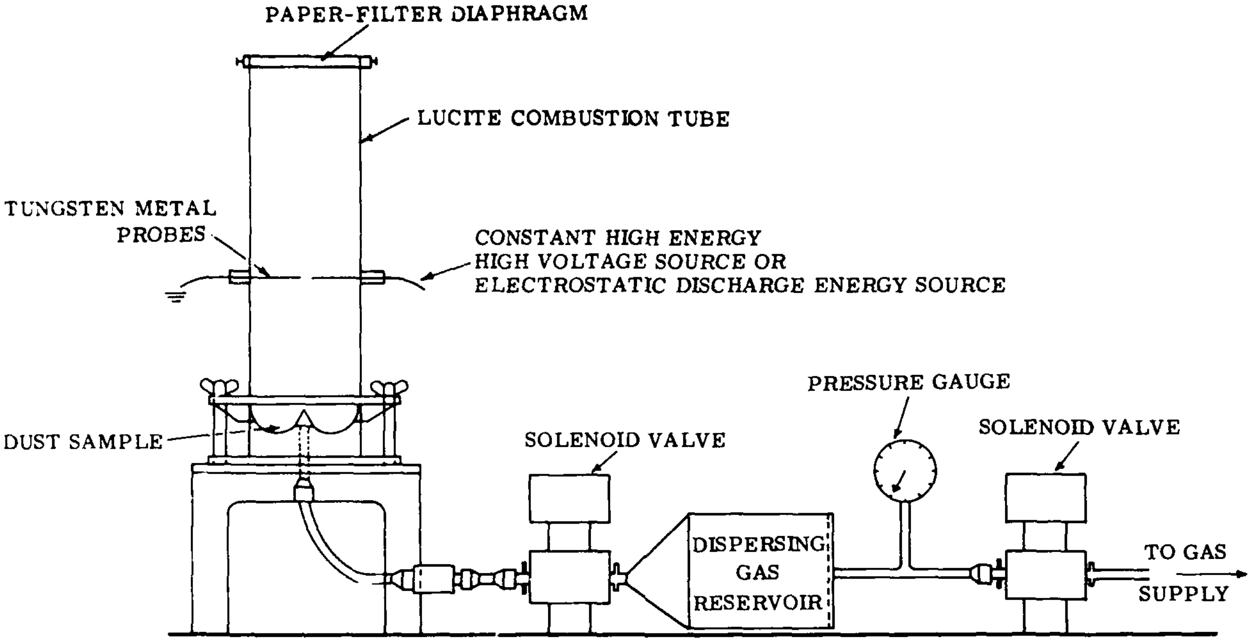

Figure 2-11 Hartman Dust Explosibility Test Apparatus

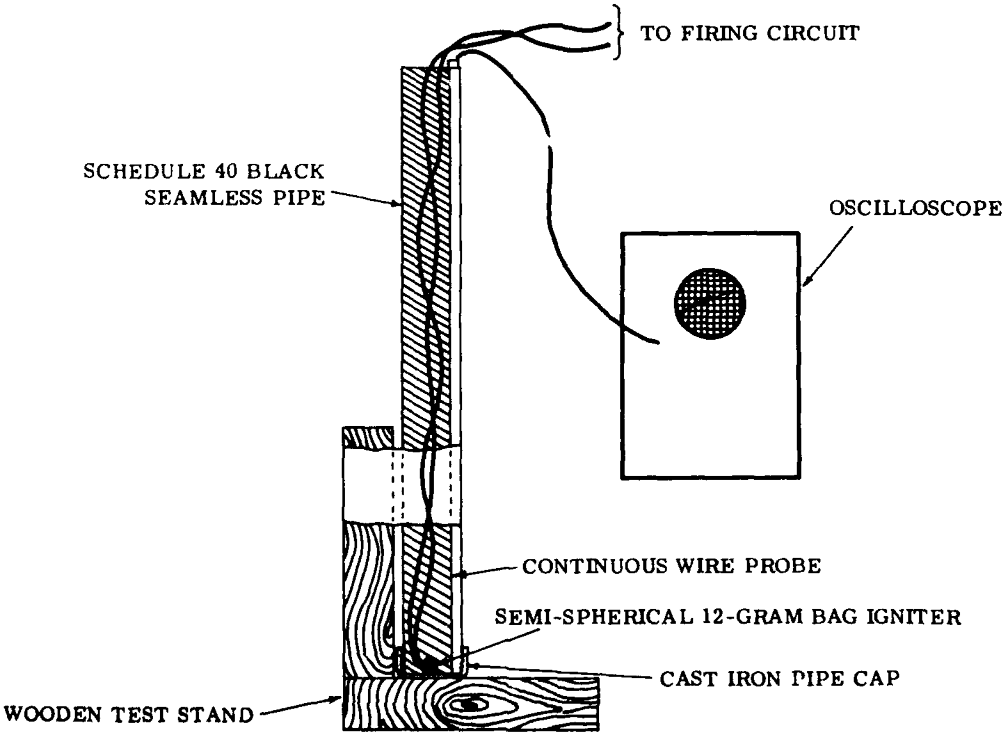

Figure 2-12 Experimental Test Setup for Critical Height to Explosion

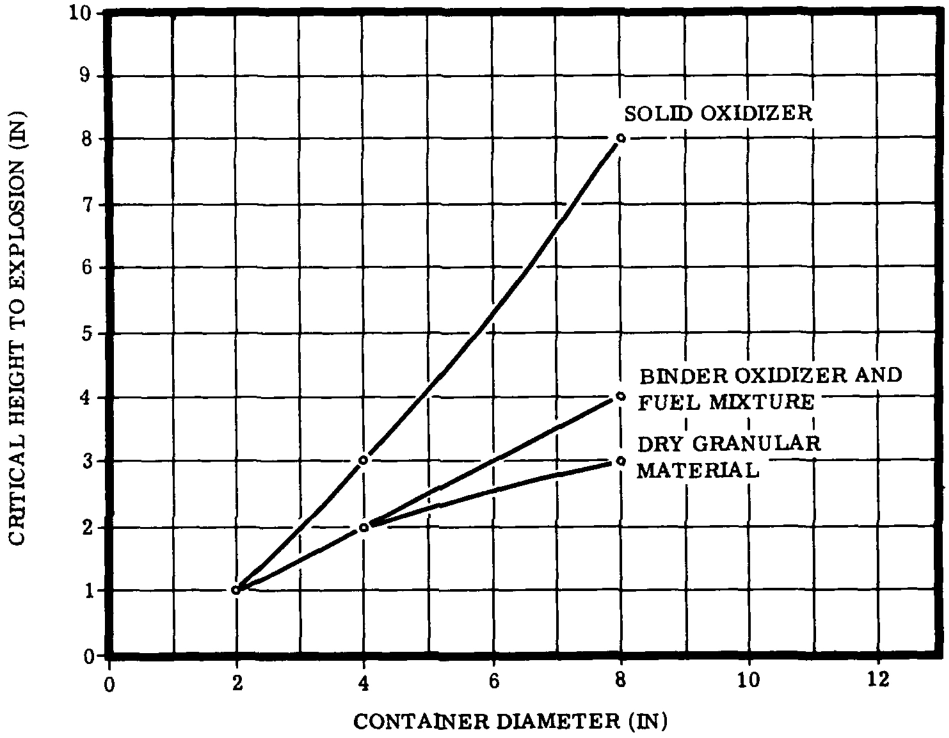

Figure 2-13 Critical Height to Explosion vs. Container Diameter

formation regarding reaction rates. Material height for transition to detonation was established in the same manner as the critical height to explosion test; however, the Primacord-lead plate witnessing technique is employed to detect detonation. Primacord is detonated by shock pressures in excess of 4 Kilobars and velocities $>$ 1500 m/s. One undesirable feature of this approach is that Primacord, if not sufficiently protected, can be initiated by container fragments, thus yielding erroneous results.

An alternate technique uses a resistance wire probe (reference 13), Figure 2-10, to monitor the reaction velocity during burning and the transition periods to explosion and detonation. In principle, the pressure front accompanying the reaction collapses a metal tube onto a resistance wire, producing a change in the circuit resistance and a corresponding change in the magnitude of the input voltage signal to an oscilloscope. The voltage signal, interpreted as distance (material height) and expressed as a function of time, provides a velocity profile of the reaction through the entire sample length. The material height at which the reaction rate exceeds that produced by the initiation source is the onset of the explosive reaction or the minimum material height necessary to produce an explosive reaction. Tests are normally performed at 2, 4, and 8 inch diameters to establish the materials transition characteristics as a function of test container diameter. Full scale tests are performed in special cases where critical height data cannot be used to predict the material transition hazard because of system design or confinement.

2-17.4 TEST ANALYSIS AND LIMITATIONS. Whether a material explodes or detonates is, for practical purposes, of little consequence since both reactions result in destruction of facilities and possibly personnel injury. Since material explosion heights are generally lower than detonation heights, the safety of personnel and protection of plant operations dictate that the no-explosion level be employed in process hazard evaluation studies.

Critical heights to explosion (CH_e) data are reported as the material height (inches) above which an explosive reaction can occur for a given container diameter. The data are valid within the limits of test container sizes and confinement and assuming submerged initiation, equal to a 12 gram bag igniter. Application to process hazard analysis studies assumes the critical height to explosion increases with the charge diameter, Figure 2-13. It is expected that the effect of diameter on CH_e will diminish as this dimension increases. These data are representative of a highly transient reaction and may not be indicative of a situation where the material continues to burn. In the latter case, cook-off data would be more applicable. The critical height test permits testing of solids, liquids, and mixtures used in propellant manufacture. The test can also be used to verify benefits of recommended material modifications to eliminate or minimize transition hazards. Further, the test can be used to implement investigations of influencing factors such as initiation energy, density, temperature, design configuration, and degree and material of confinement.

2-17.5 APPLICATION TO PROCESS HAZARDS ANALYSIS. Application of transition data to propellant manufacturing operations increases safety by establishing nonexplosive material heights in handling, mixing, transportation and storage containers. The explosion height for a particular material for any diameter is predicted by extrapolation from small-scale data plots for the container diameter in question. Process material heights exceeding the extrapolated critical height to explosion value are potential transition hazards. The latter are eliminated by lowering material heights below the height necessary for explosion, by modifying the material itself, or possibly by using a less confining container. In instances where none of these above approaches are feasible, the transition data establish the requirements and extent of remote operating conditions.

2-18 CRITICAL DIAMETER FOR PROPAGATION TEST

2-18.1 OBJECTIVE. To determine if a material will propagate an explosive reaction when subjected to induced shock and to establish the critical dimension for nonpropagation.

2-18.2 OPERATING PRINCIPLE. Materials are purposely shocked by pressures of a detonating high-energy donor to determine if a material dimension is capable of propagating an explosive reaction. The dimensions of the material are varied under specific environmental process conditions to establish the critical nonpropagating dimension.

2-18.3 TEST DESCRIPTION. A typical test arrangement for determining the critical non-propagating diameter for propellant materials is shown in Figure 2-14. A suitable container is charged with the material to be tested and subjected to induced shock produced by a high-energy donor material. Sample material environmental conditions encountered during processing are duplicated during testing. Confined tests are normally performed in schedule 40 steel containers, and unconfined processing tests are made in cardboard tubes. Other containment materials are employed when dictated by the process hazard survey. The explosive donor diameter is equal to that of the test specimen and has a minimum length over diameter ratio (L/D) equal to 3:1 plus one inch for the initiating cap. This minimum ratio presumably allows the donor-induced shock wave to achieve constant velocity and maximum radius of curvature at the sample interface.

Criteria for ascertaining that a material dimension propagated an explosion reaction in any particular test trial can be established using a Primacord-lead plate detection system and ionization or pressure instrumental techniques to monitor the rate of propagation through the entire sample length. The Primacord technique has the disadvantage of not detecting low reaction rates (1500 meters/sec), and neither the Primacord nor the ionization probe method provides continuous monitoring or accurate rates for decaying and accelerating reactions (pulsating reactions).

The pressure probe is capable of measuring both low and high reaction rates. A resistance wire probe (reference 13) is used to monitor the reaction velocity, Figure 2-10. In principle, the pressure front accompanying the reaction collapses a metal tube onto a resis-

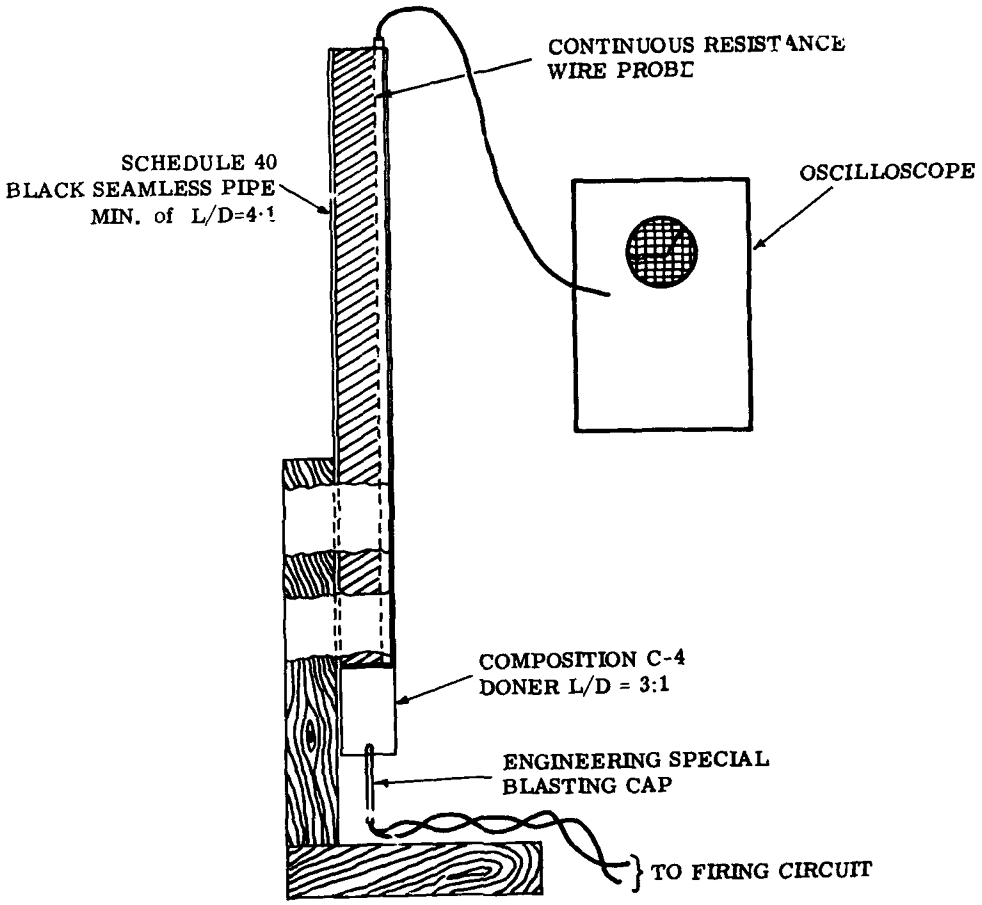

Figure 2-14 Critical Diameter Experimental Test Setup

tance wire, producing a change in the circuit resistance and a corresponding change in the magnitude of the input voltage signal to an oscilloscope. The voltage signal, interpreted as distance (material length) and expressed as a function of time, provides a continuous velocity profile for studying the reaction rate through the entire sample length. The test container diameter is normally varied in 0.25 inch increments until a diameter is reached at which the material fails to propagate an explosion reaction. A minimum of three trials is performed at this level to establish the nonpropagating material diameter.

2-18.4 TEST ANALYSIS AND LIMITATION. Critical diameter data are reported as the confined or unconfined material diameter (inches) at which an explosion reaction will not be propagated. Application to process hazard analysis studies assumes an initiation has occurred and has progressed to an explosion reaction equal to or greater than that characteristic of the Composition C-4 booster material. Degree of confinement can influence test results and thus must be considered in applying the data.

The critical diameter test permits testing of solids, liquids and mixtures that may occur during propellant manufacture. In addition, benefits of recommended material modification to eliminate potential propagation hazards are easily verified as are results of studies to investigate density, temperatures, confinement, and high and low-reaction rate phenomena with liquid and solid explosives.

2-18.5 APPLICATION TO PROCESS HAZARDS ANALYSIS. Application of critical diameter data to propellant manufacturing operations increases safety by establishing proper sizing and selection of equipment materials for transfer lines to prevent an explosive reaction from propagating through inter-connected equipment. In-process material diameters and confinement data from the process survey are compared with applicable critical diameter data to determine if the nonpropagating diameter is exceeded, in which case a propagation hazard would exist. This propagation hazard is usually eliminated by designing line sizes below the critical diameter, by using materials offering less confinement, or by modifying the material or process. In instances where none of the above approaches are feasible, the critical diameter data establish the requirements and extent of remote operating conditions.

2-19 BOTTLE DROP TEST

2-19.1 OBJECTIVE. To assess the hazards associated with the inadvertent dropping of explosive liquids during transport or sampling.

2-19.2 OPERATING PRINCIPLE. Containers of explosive liquids, preferable of the same geometry as those used in the process, are dropped from various heights to determine if the test liquid can be initiated in this type of environment.

2-19.3 TEST DESCRIPTION. One technique for this test employs plastic bottles partially filled with the test liquid. The bottles are dropped to failure or the limit of the test, 10 feet or so, from progressively increasing heights. A spinning rod and reel has been used as an inexpensive remote handling device which gives a reproducible, almost free fall for repeated testing of a plastic bottle. When a liquid has shown no reaction at the limit of the test, some evaluators have modified the test by adding glass or steel spheres to modify the drop to include shear and impact; the sample may be transferred (remotely) to a glass bottle and dropped from the maximum height as a final test.

2-19.4 TEST ANALYSIS AND LIMITATIONS. Some explosive liquids have been initiated into low velocity explosive reactions by relatively small energy inputs, stress concentrations and cavitation in the liquid being deemed the mechanism of initiation. At the stage of development of a new explosive liquid when two to five pound quantities are available, this test has been used to assess the hazard of an inadvertent drop of the material. The bottle drop test has the limitation, as does all sensitivity testing, of possibly finding only an unsafe condition while giving no information as to a safe condition.

2-19.5 APPLICATION TO PROCESS HAZARDS ANALYSIS. In the handling of explosive liquids, a major process hazard has been the occasional dropping of a container of the liquid during transport or sampling. The bottle drop test is useful in assessing this hazard, especially when the test item is identical to the item used in the process being evaluated.

2-20 CARD GAP TEST

2-20.1 OBJECTIVE. To determine the force required to initiate a detonable material (solid or powdered).

2-20.2 OPERATING PRINCIPLE. Materials are confined in steel tubing and subjected to the detonation of a booster explosive to measure the relative ease of initiation to a detonation.

2-20.3 TEST DESCRIPTION. The experimental test arrangement for determining the card gap value is shown in Figure 2-15. The test is usually known as the NOL Card Gap Test (reference 13) because in its present form it was developed by the Naval Ordnance Laboratory, White Oak, Maryland. The sample container is a cold-rolled steel tube, 1.44 inch ID x 1.875 inch OD x 5.5 inch long.

An explosive booster consisting of two inch diameter, 1 inch high pentolite pellets is used to provide the shock wave with which to initiate the sample. The result of the test is determined by the presence or absence of a hole in a steel plate 4 inch square x 3/8 inch thick. To avoid penetration of the plate due to the concentrated effect of reflected waves, an air gap of 1/16 inch is provided between the top of the sample and the witness plate. The test result can be given as three negative trials for a given gap height which is a standard interval greater than a smaller height which gives one positive result in three trials.

2-20.4 TEST ANALYSIS AND LIMITATIONS. The use of the witness plate limits the usefulness of the test because of the high pressure (95 kilobars) required to penetrate the steel plate. This difficulty is remedied by using one

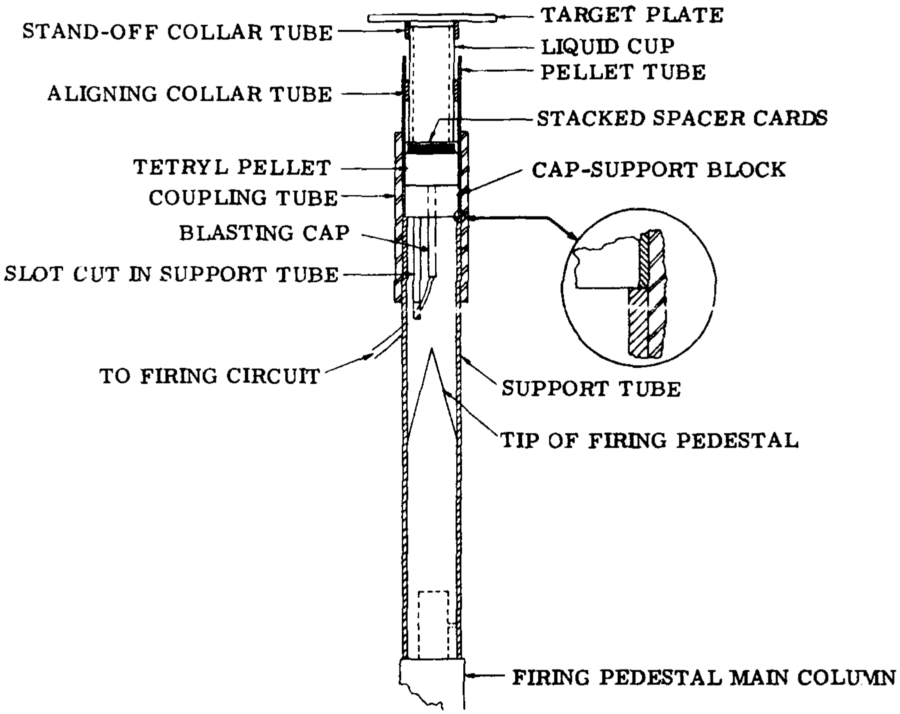

Figure 2-15 Card Gap Test Apparatus

of the following devices:

a. ionization probes which can establish a steady-state detonation velocity

b. a resistance wire probe which also serves to measure low velocity detonation

Although, generally, double-base propellants have critical diameters of less than two inches, most composite propellants have critical diameters greater than two inches and hence may give erroneous or misleading results.

The test is designed to measure a detonational response, but frequently a response of burning is just as important because of its potential for destruction.

As presently constituted, the test method employs a decaying pulse lasting in the order of microseconds. It is recognized that the pressure level and duration of the pulse as well as the confinement (in this case, in a steel pipe) have an important effect on the test results and their applicability. Consequently, a "zero" card gap does not preclude the ability of a material to react explosively.

2-20.5 APPLICATION TO PROCESS HAZARD ANALYSIS. The card gap test is applicable to process situations wherever a detonation can occur. Examples are liquid and powdered materials, either fluidized or stored. In such cases the card gap test will serve to compare the relative ease of initiation to detonation or even to estimate the actual force (in kilobars) required to cause detonation. Some approximate methods are available to extrapolate the required pressures to diameters larger than 2 inches.

In some instances it may be possible to avoid detonations by using either detonation traps or appropriate thicknesses of attenuating materials such as plastics or metals.

2-21 SUSAN TEST (Lawrence Radiation Laboratory)

2-21.1 OBJECTIVE. To assess the relative behavior of explosives under field conditions of impact.

2-21.2 OPERATING PRINCIPLE. In this test a specimen of confined explosive is subjected to a high velocity impact against a steel plate. The degree of reaction of the explosive sample is inferred and used as an index of sensitivity.

2-21.3 TEST DESCRIPTION. An explosive specimen of slightly less than 1 lbm is confined in a 12 lbm projectile and fired from a converted 3 inch naval gun at velocities from 100 to 1200 fps onto a massive steel target. The velocity of the projectile, the blast overpressures generated by the specimen, the light emitted from the reaction of the specimen, and the time between the impact of the specimen and the arrival of the blast wave at the pressure gauge are measured with appropriate transducers. High-speed cameras are also used to observe the impact phenomena. From these data, an energy of reaction, on an arbitrary scale, is determined and given as a *function of impact velocity.*

2-21.4 TEST ANALYSIS AND LIMITATIONS. The relative energy scale is set so that a violent deflagration or detonation which consumes all of the explosive will give a value of approximately 100. A milder deflagration, which also consumes all of the explosive, has been found to give only 30-40 on the same scale. The test, thus far, is limited to a fixed geometry and is designed for the testing of solid explosives.

2-21.5 APPLICATION TO PROCESS HAZARDS ANALYSIS. The test results could be applied to an analysis of process hazards where impact velocities are expected to exceed those normally encountered in dropping or other sudden deceleration of explosive items. Materials which have produced no catastrophic incidents ignite and burn during the test, while the more hazardous explosives burn to detonation under the same conditions. No solid propellants have demonstrated the latter results as yet.

2-22 SHEAR WATER HAMMER TEST

2-22.1 OBJECTIVE. To determine if a moving bed of an explosive mixture in a slurry form can be initiated by sudden deceleration.

2-22.2 OPERATING PRINCIPLE. Thus far, use-type tests, in which the material in question is dropped or shot onto a steel plate, have proved the most useful.

2-22.3 TEST DESCRIPTION. None.

2-22.4 TEST ANALYSIS AND LIMITATIONS. Test results on the slurries investigated thus far have shown a velocity requirement greatly exceeding that available in the process.

2-22.5 APPLICATION TO PROCESS HAZARDS ANALYSIS. During the processing of slurries, particularly those whose continuum is a detonable liquid, this mechanism of initiation must be taken into account. In newer systems which might contain a less desensitized liquid continuum than those heretofore examined, use-tests of this type should also be performed.

2-23 GLOSSARY OF TERMS EMPLOYED IN EXPLOSIVE TECHNOLOGY

Explosive	Any material which decomposes exothermically over a short time period to yield high gas pressure or shocks (impulse) in the immediate vicinity.
Primary Explosives	Explosives which can be detonated by thermal means (i.e., hot wire). Example: lead azide, mercury fulminate.
High Explosives	Explosives which can be detonated only when in contact with or in vicinity of, another detonating explosive. Examples: TNT, Pentolite, etc.
Propellants	High-energy materials which are employed in such an environment that they burn (sometimes at a high rate) but without the destructive force of an explosive. Under certain conditions these materials will also function as explosives.
Initiation	That stage in an explosive decomposition in which a stimulus (i.e., heat, shock, etc.) has initiated a decomposition but the decomposing substance has not released sufficient energy to proceed beyond the burning stage.
Deflagration	The second stage of the explosive decomposition process in which a self substained reaction is being carried out. Heat is transferred from the reacted to the unreacted material, causing further reaction. Generally, deflagration is a very slow process and dependent on ambient pressure.
Transition	The third stage of an explosive in which rate increases from a deflagration to a high velocity reaction usually called an explosion or detonation.
Detonation	A steady state-high rate consumption of the explosive in which energy liberated is transmitted to the unburned layers of explosive by means of shock waves. In most condensed explosives the rate at which the detonation passes through the explosive is 5 to 8 mm/μ sec.
Explosion	Often used synonymously with detonation. In most cases denotes any reaction in excess of burning.
Brisance	The shattering power or destructive energy release of an explosive. The brisance of the explosive can be measured by such techniques as the "sand test", Trauzl lead block and ballistics mortar tests. Results of these tests are generally dependent on the energy content and the reaction rate of the explosives involved.
Air Blast	The destructive energy imparted to the atmosphere surrounding an explosion.
Overpressure	The increase in pressure (above ambient) in the atmosphere surrounding an explosion. The damage potential of the overpressure (a quantitative assessment of air blast) depends on both magnitude and duration (i.e., impulse).

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CHAPTER 3

PROPELLANT MANUFACTURING

3-1 INTRODUCTION

3-1.1 PURPOSE. This section on propellants will alert the manager, supervisor, engineer, and operator to the many hazards encountered in the manufacture of solid propellant, its handling, and its utilization in the assembly of a rocket motor. It offers information on identification of hazards, how they might be eliminated or controlled, and what to expect if they are not. The text is not presented for use as an operating procedure nor does it present all the information that one needs to design a manufacturing facility or to operate one. It is believed that, having been alerted to the hazards, all possible sources of information will be sought to insure the safety of operations.

3-1.2 REGULATIONS. Many of the processes used in the manufacture of solid propellant evolved from those used in the explosives industry. Consequently, it inherited many of the governmental regulations and laws which had been established to control the hazards of that industry. The knowledge that other equally hazardous materials may not be so regulated tends to cause the newcomer to overlook the existence of the laws and regulations which do apply to the manufacture of solid propellants. Safety regulations concerning the production of hazardous materials demand increasing consideration. The safety office of every propellant manufacturing installation should maintain a complete library of all the Federal, State, County, and City regulations governing the handling of explosives and solid propellants.

The customer, particularly the military or other government department, will be concerned about a manufacturer's capability to perform the contract to its completion without delays in schedule. To increase the assurance that delays are not caused by accidents, specific safety requirements will be applied to operations. Therefore, knowledge of the safety requirements of the contract is essential. The military is particularly sensitive about the adverse publicity which it gets when a facility producing hazardous materials has an accident which injures employees, destroys facilities, or causes damage in the community. To assure that a cause for such publicity does not occur, the contractor will be required by contractual agreement to maintain controls over his operation which will lessen the probability of such an incident. The safety office should also have complete information on these provisions of the contract and be able to interpret their scope and application.

3-1.3 SAFETY CODES. Probably because of the detailed governmental regulations and the attitude of the customer, as explained above, the solid propellant manufacturing industry has not developed and adopted a set of safety codes, as most other hazardous industries have, through their industry associations. Many safety codes developed by other industry associations provide very useful guidance to those engaged in solid propellant manufacturing; indeed, many of them have been adopted as law by governmental agencies. Examples of the safety codes are the National Electrical Code, the ASME Pressure Vessel Code, the Uniform Building Code, and the National Fire Protection Code.

3-1.4 ACCIDENT REPORTS. The solid propellant industry, like many other industries, has learned from accidents. In fact, many of the design characteristics which exist today in operating equipment have evolved from deficiencies discovered as a result of accidents, many of them very costly in terms of personnel injury and death, property damage, and loss of production capability. Members of the explosive and solid propellant industry have cooperated in the dissemination of accident information so that the facts may be used in accident prevention. Much of this information should be available in the safety office. Anyone planning a new operation or the changing of an old one should be sure that safety personnel are actively participating in the plans.

3-1.5 CARDINAL PRINCIPLES. Certain cardinal principals should be used in establishing hazardous operations:

a. Where operationally feasible, separate each handling operation from all others in a manner that if an accident occurs in one it will not propagate by fire, blast overpressure, or fragments to another operation. This can be accomplished by isolation, by distance, or by shielding.

b. Do not use more personnel in the operation than is absolutely necessary for its accomplishment.

c. The propellant or other hazardous material at the point of operation must be maintained at the minimum necessary for an efficient operation.

3-2 INGREDIENTS

The ingredients used in the manufacture of solid propellants are many and varied. This section does not include all those available because every research effort results in new additions to the list. The hazardous properties of many of the ingredients in common use are given. Consideration of the properties will give an indication of the hazards involved in handling and processing the ingredients during manufacturing operations. If the necessary information for safe use is not found in this section, it is suggested that information be sought directly from the supplier.

3-2.1 ALUMINUM.

3-2.1.1 Properties.

a. Physical:

Density (solid)	2.699 g/cc at 20°C
Apparent Density	1.0 - 1.2 g/cc
Crystalline Form	cubic
Average Particle Size	13 - 32 microns
Color	silvery white
Melting Point	932 ± 1°K (659°C)
Boiling Point	2720°K (2447°C)

b. Chemical:

Symbol	Al
Molecular Weight	26.98

3-2.1.2 Hazards.

a. Fire:

Ignition Temperature of Aluminum Powders in °C.

	Dust Cloud	Dust Layer
Atomized	640	750
Stamped	550	470

Electrostatic Sensitivity. Minimum energy required for ignition of powder by electric sparks (millijoules)

	Dust Cloud	Dust Layer
Atomized	15	2.5
Stamped	10	1.5

Aluminum dust may be ignited when exposed to heat or flame or it may ignite by chemical action with oxidizers. The powder is easily ignited or initiated to explosion by the discharge of static electricity. Water should be kept away from aluminum dust because it reacts to form hydrogen gas. The hydrogen gas may ignite to cause an explosion.

Firefighting: In extinguishing fires anything which tends to disturb the burning mass may throw aluminum into the air and result in a violent explosion of tremendous destructive power. Dry sand if carefully placed over a pile of burning aluminum will smother the flame. Except for small fires the sand and the aluminum powder may be thrown into the air. It is usually better to try to isolate the fire and let the fire burn out.

b. Explosion:

When mixed with air, aluminum powder is an explosion hazard.

c. Health:

(1) Aluminum powder is systemically inert.

(2) Although a single case of pulmonary fibrosis has been reported, chronic exposure to aluminum dust is not considered hazardous to health.

(3) Provide adequate ventilation and perform physical examinations annually, including chest x-rays.

(4) The TLV is 15 mg/m^3 (ACGIH 1968).

(5) The combustion products of aluminum powder present no acute health hazard.

d. Shipping and Handling. Good housekeeping and dust control are important. Keep powder dry. Eliminate ignition sources in dusty area, i.e., no smoking, open lights, fires, sparks. Ground all equipment, especially containers being filled or emptied. Scoops should be conductive and of non-sparking material. No non-conductive material should be used to handle the powder. Powders should be handled gently and never allowed to fall any distance.

3-2.2 ALUMINUM HYDRIDE

3-2.2.1 Properties.

a. Physical:

Density	1.43 g/cc
Form	Fine gray powder
Particle size	2 micron avg.

b. Chemical:

Molecular Formula	AlH_3
Molecular Weight	29.97
Heat of Formation	-2.2 kcal/mol

3-2.2.2 Hazards.

a. Fire:

Ignition Temperature	120° to 140°C
Vacuum Stability (45°C)	Generates H_2 at 20 cc/g - 24 hr (STP).
Impact (5kg wt.)	Negative at 600 mm
Friction	55 lb
Electrostatic Sensitivity	.00125 joule
DTA	Decomposes slowly at room temperature. Exotherm about 50°C-70°C, strong exotherm at about 140°C.
Fire Fighting:	Aluminum hydride is a fire hazard because it will ignite when exposed to air. A fire cannot be extinguished with water, CO_2, carbon tetrachloride, or any foams. To extinguish - smother with dry sand or dry powder.

b. Explosion:

AlH_3 reacts explosively with water, acids, and oxidizers. In an inert atmosphere, it decomposes with heat to form hydrogen gas. It is also a dust hazard. In this condition it will pick up a charge and react explosively. Containers should be grounded when being filled or emptied to prevent an explosion.

c. Health:

There is practically no information on the toxicity of aluminum hydride. Exposure to this dust is limited by its flammability. The toxicity of metal hydrides generally is related to that of the metal. Aluminum is essentially non-toxic. Precautions to avoid its combustion will provide adequate health protection.

d. Shipping and Handling: Contact with the skin should be avoided as moisture from the hands could cause ignition. Respirators, goggles, and rubber gloves should be worn when handling. The material should be stored in a dry inert atmosphere at low temperatures. AlH_3 should not be stored with oxidizers.

3-2.3 AMMONIUM NITRATE

3-2.3.1 Properties:

a. Physical:

Density	1.73 g/cc
Color	colorless
Melting Point	170°C
Vapor Pressure	

°C	mm Hg
188	3.25
205	7.45
216	11.55
223	15.80
237	27.0
249	41.0

Soluble in water, alcohol, acetic acid, nitric acid, pyridine.

b. Chemical:

Molecular Formula	NH_4NO_3
Molecular Weight	80
Heat of Combustion	346 cal/g
Heat of Formation	1098 cal/g
Heat of Explosion	346 cal/g
Heat of Fusion	18.23 cal/g

3-2.3.2 Hazards.

a. Fire:

100°C Test	
loss in first 48 hrs	0.74%
loss in second 48 hrs	0.13%
explosion in 100 hrs	none

Explosion Temperature	
5 seconds	465°C

Vacuum Stability, cc/40 hrs.	
100°C	0.30
120°C	0.30
150°C	0.30

Impact, 2 Kg Wt.	
BM	100+cm
sample weight	20 mg
PA	31 in.
sample weight	17 mg

Firefighting. Use flooding amounts of water in early stages of the fire. When large quantities of ammonium nitrate are involved they should be fought by remote control.

b. Explosion:

Detonation Velocity is 1,250 - 4,650 m/sec. Card Gap - AN/fuel oil commercial mixtures is 0.73-1.85 inches. 98.9% TMD is 0.0 inches.

Ammonium nitrate is an oxidizing agent that supports combustion and is capable of undergoing detonation if heated under confinement that permits high pressure buildup, or if subjected to strong shocks.

c. Health:

Symptoms: Absorption of ammonium nitrate can cause diuresis, and aciduria. In large doses it can cause acidosis and methemoglobinemia. When used medicinally, doses as high as 6 to 12 grams per day have been prescribed.

Routes of Absorption: Ingestion or inhalation.

Local Action: It can cause irritation of eyes and mucous membranes and chemical burning of abraded skin.

Preventive Measures: Avoid skin and eye contact, inhalation, and ingestion. Prevent contamination of drinking water supplies.

TLV: None established.

EEL's: None established.

Combustion Products: Thermal decomposition at 210°C produces H_2O and N_2O. The latter has been used for anesthesia. May explode when heated with organic materials releasing irritant fumes.

d. Shipping and Handling: Classed as an oxidizing agent for shipping and storage. It is stable and can be stored at ambient conditions in large amounts without fear of self-ignition or spontaneous explosion. Hazard Class 2. In the presence of moisture, ammonium nitrate reacts with copper, iron, steel, brass, lead, and calcium.

3-2.4 AMMONIUM PERCHLORATE

3-2.4.1 Properties.

a. Physical:

Density	1.95 g/cc, Solid
Crystal Form	rhombic
Color	colorless (white appearance)
Melting Point	decomposes above 423°K (150°C)
Boiling Point	decomposes
Transition Point	rhombic to cubic 513°K (240°C)
Solubility in water	g/100 cc of saturated solution.

Temp. °C	0	25	60	100
Sol.	12	20	39	88

Solubility in Nonaqueous Solvents at 25°C.

Solvent	g/100 g solvent
Acetone	2.26
Ethyl Acetate	0.032
Ethyl Alcohol	9.908
Ethyl Ether	insol.
Methyl Alcohol	6.85

b. Chemical:

Molecular Formula	NH_4ClO_4
Molecular Weight	117.5
Heat of Formation at 25°C	-69.42 kcal/mol

3-2.4.2 Hazards.

a. Fire: Decomposition temperature and products. Decomposes at a maintained temperature of 150°C. Decomposition products are NO_2 and ClO_2. Decomposition begins at 130°C. On the DTA, a large exotherm occurs at 240-260°C resulting in decomposition.

100°C Heat Test:

loss in first 48 hours:	0.02%
loss in second 48 hours:	0.00%
explosion in 100 hours:	none

Vacuum Stability Test:

cc of gas evolved from 5g in 40 hours at:

100°C	0.13
120°C	0.20
150°C	0.32

Impact

NOS (5 kg wt)	600 mm
ABL (2 kg wt)	510 mm

Friction (8 ft/sec)	120 lb
ESD	0.625 joule

Firefighting. Fires involving Ammonium Perchlorate alone may be fought with water. If clothing catches fire, douse liberally with water, do not use fire blanket.

b. Explosion:

A powerful oxidizer, explodes violently when heated with sulphur, organic matter, or finely divided metals, particularly, magnesium and aluminum. Exploded by shock.

Card gap was not positive on standard test but damaged plate. The NOL extended gap test value using TNT explosive witness is 207 cards.

In Trauzl lead block tests, dry AP was detonated by a No. 6 cap, but when salt contained 5% moisture a No. 8 cap gave only a weak detonation.

Critical Diameter - AP (25, Dry), less than 1/4 inch.

Velocity of Detonation in 60 mm diameter iron tube and detonated with five No. 8 caps, 3800 m/sec.

Explosion temperature in 5 sec, 435°C

(AP in the size range of 5 to 15 microns reacts violently when exposed to flame or severe impact.)

c. Health:

Perchlorates are irritating to the skin and mucous membranes. Avoid skin and eye contact.

Combustion products include hydrogen chloride.

d. Shipping and Handling:

DOT: Oxidizing material; yellow label (16 micron and above).

(AP in the size range of 5 to 15 microns is DOT Class A. Shipments of this AP are made under the authority of ICC Special Permit No. 4041 in DOT 37A steel drums filled with conductive polyethylene liners of at least 3 mil thickness.

Navy: AP is received in metal drums with polyethylene liner. It is considered as an oxidizer and stored in fire-resistant buildings without regard for quantity distance. Precautions are taken to prevent contamination especially copper and brass. AP operations are considered as Class "B".

3-2.5 BERYLLIUM (BE)

3-2.5.1 Properties:

a. Physical:

Density	1.848 g/cc
Color	dull gray white (beryllium oxide)
Melting Point	2345°F
Boiling Point	5432°F at 760 mm

b. Chemical:

Molecular weight	9.0122
Heat of reaction	29,000 BTU/lb
Stability	Oxide coating develops on beryllium at room temperature which prevents further oxidation.

3-2.5.2 Hazards.

a. Fire:

Finely divided beryllium metal powder dispersed in air may ignite and burn producing beryllium oxide fumes. The degree of combustibility is a function of particle size. Fires have been started by sparks contacting an absolute filter loaded with beryllium metal powder, and by welding in ventilation ductwork used for transporting beryllium metal powder. It is standard practice for firefighters to wear protective respiratory apparatus when combating metal fires. Wind dispersal of fine beryllium oxide particles requires establishment of an exclusion area downwind. Note: Beryllium oxide particles from fires are toxic. Firefighters must wear self-contained breathing apparatus.

b. Explosion:

Does not offer an explosion hazard.

c. Health:

In the past industries have experienced serious disease problems arising from exposure to beryllium and some of its compounds. The etiology of the disease is not clearly understood, and consequently, the use of beryllium involves an unknown potential for serious public and employee health problems.

Acute beryllium disease may arise from pathological alterations to the skin, the respiratory tract, and the lungs. Chronic beryllium disease begins insidiously. The initial symptoms may include fatigue, weakness, exertional dyspepsia, cough, anorexia, and loss of weight. As the disease progresses, symptoms intensify. Onset of the disease may be delayed many years following the causative exposure.

The acceptable daily exposure limit is 0.002 milligrams Be per cubic meter averaged over an eight hour period. Beryllium process and test area shall be routinely monitored by qualified personnel for contamination. Records of air contamination shall be maintained. The static firing of rocket motors containing beryllium in the propellant offers additional problems. For protection of the surrounding community a prediction of downwind concentrations of beryllium exhaust products should be prepared prior to static testing. Personnel should be excluded from the test stand until the local beryllium oxide exhaust products are of a negligible concentration.

"Low-fired" beryllium oxide. For intermittent exposure to beryllium oxide arising from rocket motor firing, which has the physical and chemical characteristics of "low-fired" beryllium oxide comparable to a product calcined at temperatures around 400°C, a maximum exposure of 75 microgram-minutes of beryllium percubic meter may be tolerated within the limits of 10-60 minutes accumulated during any two consecutive weeks.

"High-fired" beryllium oxide. For intermittent exposures to beryllium oxide arising from rocket motor firing, which has the physical and chemical characteristics of "high-fired" beryllium oxide comparable to a product calcined at temperatures in excess of 1600°C, a maximum exposure of 1500 microgram-minutes of beryllium per cubic meter may be tolerated within the limits of 10-60 minutes during any two consecutive weeks.

In applying the above criteria for intermittent exposure to a rocket motor firing, it will be necessary to consider simultaneously the concentration of soluble beryllium compounds, the "low-fired" beryllium oxide, and the "high-fired" beryllium oxide and adjust the limits accordingly.

d. Shipping and Handling:

Beryllium compounds are shipped under a DOT Class B poison label. Beryllium in approved shipping or in-plant handling containers should be stored in fire proof buildings.

Waste water lines or drainage from a storage and handling building or area should be terminated into retention ponds or ditches in order that the contamination of final discharge water will not exceed the most stringent level established by local, state, or federal authority.

In event that accidental spills of beryllium powder occurs the following actions are recommended:

a. Evacuate exposed or affected persons from the contaminated area.

b. Insure that all persons suspected of being affected by the beryllium spill are sent for medical examination.

c. Establish an exclusion area.

d. Allow only essential personnel, protected by approved clothing and respiratory apparatus, into the exclusion area.

e. Collect spilled beryllium powder by vacuum, and place in suitable containers for removal to a pre-established disposal area.

f. After all powder is collected, flush area with water.

g. Perform tests for detection and analysis of surface and airborne contamination.

h. Retain the exclusion area until decontamination is completed.

3-2.6 BIS (2,2-DINITROPROPYL) FORMAL (BDNPF)

3-2.6.1 Properties.

a. Physical:

Refractive Index at 35°C	1.4614-1.4624
Volatility at 77°C	3.0×10^{-8} g/cm^2-sec (max)
Boiling Point (Approx.)	110°C at 0.02 mm Hg
Melting Point	33°-34°C
Density	1.415 g/cc

b. Chemical:

Moisture	0.95% (max)
Acid Number	0.10 mg KOH/g (max)
Hydroxyl ion	0.04 (max)
Formula	$[CH_3C(NO_2)_2CH_2O]_2CH_2$
Molecular Wt.	312.0

3-2.6.2 Hazards.

a. Fire:

Burns without detonating.

b. Explosion:

BDNPF is detonated by a tetryl booster but not by a No. 8 Cap.

Impact Test (50% Point)	50 cm/2kg
Critical Diameter	1.5-2.0 inches
Adiabatic Compression (Aerojet Test) at 120,000 psig/sec	negative
at 180,000 psig/sec	positive
at 280,000 psig/sec	positive

c. Health:

Avoid skin contact or vapor inhalation (may be irritating to the skin or mucous membranes.)

d. Shipping and Handling: Completely compatible with bis (2,2-dinitropropyl) acetal. Hydrolyzed by strong acids, unaffected by bases. Classed by DOT as Class "B" explosive. Keep away from heat or flame. Avoid shock. Extinguish fire with foam, dry chemical, or carbon dioxide.

3-2.7 BLACK POWDER

3-2.7.1 Properties.

a. Physical:

Density	approx. 1.6 g/cc
Crystalline Form	mechanical mixture
Color	Black

b. Chemical:

Heat of explosion	684 cal/g

3-2.7.2 Hazards.

a. Fire:

75°C International Heat Test

Loss in 48 hrs	0.31%

Explosion Temperature

seconds	°C
0.1 (no cap used)	510
1	490
5	427
10	356

Vacuum Stability,	cc/40 hr
100°C	0.5
120°C	0.9

ESD

Unconfined	greater than 12.5 joule
Confined	greater than 0.8 joule

Impact, 2 Kg Wt.

BM	32 cm
PA	16 in
Sample wt.	16 mg

Fire Fighting. A black powder fire proceeds with explosion-like violence. No attempt should be made to fight the fire.

b. Explosion:

Detonation Velocity 400 m/sec

Decomposition Temperature. Exotheral preignition reaction starts at about 250°C and culminates at approximately 300°C (DTA and TGA curves.)

c. Health:

Symptoms: Potassium nitrate is the chief toxic ingredient. It can cause gastric irritation, emaciation, anemia, methemaglobinemia, and respiratory paralysis.

Routes of Absorption: Ingestion.

Local Action: There are no reports of direct action on the skin or eyes.

Preventive Measures: Prevent accidental ingestion by proper labeling.

TLV: None established.

EEL's: None established.

Combustion Products: Oxides of nitrogen may be released which are lung irritants. The effect may be *delayed several hours.*

d. Shipping and Handling: Black powder is shipped according to the DOT Regulations for a Class "A' explosive. A container of black powder should never be opened in the presence of other explosives. No more than three persons will be allowed to work with or be in the immediate area of exposed black powder. Black powder absorbs moisture and deteriorates rapidly. If kept dry, it will retain its properties indefinitely. Military classification, Class 7, Group O.

3-2.8 COMPOSITION C-3

3-2.8.1 Properties.

a. Physical:

Density	1.60 g/cc
Color	yellow
Plasticity:	
Below 0°0	hard (-29°C)
0-40°C	plastic
Above 40°C	exudes (77°C)

Solubility - soluble in acetone

b. Chemical:

Composition:

RDX	77%
Tetryl	3%
TNT	4%
DNT	10%
MNT	5%
NC	1%

3-2.8.2 Hazards.

a. Fire:

100°C Heat Test	
loss in first 48 hrs.	3.20%
loss in second 48 hrs.	1.63%
explosion in 100 hrs.	none
Explosion Temperature:	
5 seconds	280°C decomposes)
Vacuum Stability - cc/40 hrs	
100°C	1.21
120°C	11+
Impact, 2kg wt.	
BM	100+ cm
PA	14 in
Sample wt.	33 mg
Friction (8 ft/sec)	290 lb
ESD	greater than 12.5 joules
Fire Fighting	

Do not fight a Composition C-3 fire: evacuate.

b. Explosion:

Detonation Velocity	7625 m/sec
Card Gap	1.86 in. (den 1.60)

C-3 is a high explosive more powerful than TNT and about the same impact sensitivity.

c. Health:

Symptoms: C-3 can cause reduced blood pressure. headache, dizziness, nausea, methemoglobinemia, cyanosis, convulsions.

Routes of Absorption: It may be absorbed in toxic amounts by inhalation, ingestion, or skin contact.

Local Action: Irritation of the skin or eyes from direct contact has not been reported. Allergic dermatitis may be observed from some components.

Preventive Measures: Reduce inhalation hazard by ventilation. Wear protective gloves and aprons (rubber can retain hazardous amounts of some ingredients) and wash exposed skin frequently with soap and water to reduce skin absorption. Do not use solvents for clean up.

TLV: 1.5 mg/m^3 has been recommended for most of the hazardous ingredients.

EEL's: No recommendations.

Combustion Products: Lung irritation, both immediate and/or delayed can be caused by the combustion products.

d. Shipping and Handling: C-3 is classified as Class 7, Group I. When shipped in bulk form, C-3 shall be packed in rectangular wooden boxes at 58 pounds net weight, lined with strong oil and moisture-proof paper bags. C-3 can be packed by hand to form explosive charges for demolition purposes.

3-2.9 COMPOSITION C-4

3-2.9.1 Properties.

a. Physical:

Density	1.59 g/cc
Color	light brown
Plasticity:	
below 0°0	plastic (-57°C)
0-40°C	plastic
above 40°C	plastic (77°C)
Solubility	soluble in acetone

b. Chemical:

Composition:

RDX	91%
Polyisobutylene	2.1%
Motor Oil	1.6%
Di-(2-ethyl-hexyl) Sebacate	5.3%

3-2.9.2 Hazards.

a. Fire:

100°C Heat Test	
loss in first 48 hrs	0.13%
loss in second 48 hrs	0.00%
explosion in 100 hrs	none
Explosion Temperature:	
5 seconds	290°C

Vacuum Stability, cc/40 hr	
100°C	0.26
Impact, 2 kg wt	
BM	100+cm
PA	19 in
sample wt.	27 mg

Fire Fighting: Do not fight a composition C-4 fire - evacuate.

b. Explosion:

Detonation Velocity	8040 m/sec
Card Gap	2.14 in (den 1.41)

C-4 is less sensitive to impact than C-3, but they are both of the same order of sensitivity to initiation.

c. Health:

Symptoms: C-4 can cause headache, insomnia, dizziness, restlessness, and convulsions resulting from central nervous system stimulation. The convulsions resemble grand mal or epileptic seizures. It does not exhibit pharmacological effects similar to nitrates or nitrites.

Routes of Absorption: It can be absorbed by ingestion and inhalation but does not penetrate the skin.

Local Action: No effects from skin or eye contacts have been reported.

Preventive Measures: Ventilation should be provided to reduce the inhalation hazard. Employees should be cautioned of the effects from accidental or intentional ingestion.

TLV: No recommendations.

EEL's: No recommendations.

Combustion Products: Oxides of nitrogen and of carbon will be in the combustion products. Lung irritation may be produced either promptly or delayed a few hours after exposure.

d. Shipping and Handling: C-4 is classified as Class 7, Group I. C-4 is packed by hand to form explosive charges for demolition. It is more powerful than TNT and detonates at a higher velocity than C-3. It erodes less than other plastics explosives when immersed underwater for long periods. C-4 may be detonated by the military blasting cap.

3-2.10 COPPER CHROMATE

3-2.10.1 Properties.

a. Physical:

Other names	Basic Cupric Chromate
Form	Light chocolate-brown powder
Solubility	Insoluble in water; soluble in nitric acid

b. Chemical:

Symbol	$CuCrO_4 \cdot 2CuO \cdot 2H_2O$ (loses water at 260°C)
Molecular weight	374.66

3-2.10.2 Hazards.

a. Health:

Copper chromate can cause dermatitis, skin ulcers, conjunctivitis with lacrimation, edema of the eyelids, ulceration and turbidity of the cornea, ulceration of the cartilaginous part of the nasal septum, erythematous macular rash of the skin, cancer of the lungs, gastrointestinal disturbances (spasms, gastritis, and ulcers of the stomach and intestine) and hepatitis. Absorption can occur by contact with the skin and eyes, inhalation or ingestion.

Ventilation should be used to reduce the inhalation hazard. Wear appropriate eye protective equipment and protective gloves. Wash exposed skin frequently with soap and water to reduce skin absorption.

TLV for chromates (as CrO_3 is 0.1 mg/m^3).

b. Shipping and Handling: No shipping regulation. Good housekeeping and dust control are important.

3-2.11 CYCLOTETRAMETHYLENE TETRANITRAMINE (BETA HMX)

3-2.11.1 Properties.

a. Physical:

Types - Four types of HMX are produced Alpha, Gamma, Delta, and Beta. With the Beta type being the most common type.

Crystal Density	1.903 g/cc
Color	white
Melting Point	280°C

Solubility Specifications:

(1) Total acetone insoluble material - 0.05% (max)

(2) Inorganic insoluble material - 0.03% (max)

b. Chemical:

Molecular Formula	$C_4H_8N_8O_8$
Molecular Weight	296.17
Heat of Combustion	2362 cal/g
Heat of Formation	-60.5 cal/g
Heat of Detonation	1222 cal/g
Heat of Explosion	1356 cal/g

3-2.11.2 Hazards.

a. Fire:

100°C Heat Test

loss in first 48 hours	0.05%
loss in second 48 hours	0.03%
Explosion in 100 hours	none

Explosion Temperature

Seconds	°C
1 (no cap used)	380
5	327
10	306

Vacuum Stability Test (cc/40 hr)

100°C	0.37
120°C	0.45
150°C	0.62

ESD

Dry HMX	0.075 joules
20% Acetone Wet	0.075 joules
Coated with NC and TA	75.0 joules

Impact, 5kg wt - 50% probability

25 micron	125 mm to 175 mm
60 micron	150 mm to 275 mm
200 micron	150 mm

Friction (8 ft/sec)	less than 1 lb.
Ignition	233.5°C to 241.5°C

Electrostatics:

Dry HMX creates an electrostatic hazard due to its ability to maintain a high potential with reference to ground. Therefore, all equipment used should be grounded.

Firefighting:

HMX is a high explosive; when a HMX fire occurs vacate the premises.

b. Explosion:

Critical Diameter	less than 1/4 in
Detonation Velocity 97% voidless density	9124 m/sec

Solutions of HMX should not be exposed to direct sunlight. There is a chance of instability in the presence of ultraviolet light. HMX is highly sensitive and will readily explode.

c. Health:

HMX has been reported to have caused symptoms of epilepsy; therefore, respirators should be worn. Otherwise it is considered non-toxic.

d. Shipping and Handling:

The stability of HMX is very good; therefore, storage does not present a hazard in this respect. HMX is shipped in water and isopropanol. The percent of water ranging from 30 to 60%. The isopropanol is used for an antifreeze. HMX is classed as a Class "A" explosive for shipping it wet. Shipping of dry material is prohibited. HMX is usually not handled in the dry state; where possible it is diluted with a solvent such as acetone.

3-2.12 CYCLOTRIMETHYLENE TRINITRAMINE (RDX)

3-2.12.1 Properties.

a. Physical:

Crystal Density	1.802 g/cc
Color	white
Melting Point	204°C

Solubility Spec.

(1) Total acetone insoluble material - 0.05% max.

(2) Inorganic insoluble material - 0.03% max.

b. Chemical:

Molecular Formula	$C_3H_6N_6O_6$
Molecular Weight	222
Heat of Combustion	2285 cal/g
Heat of Formation	-96 cal/g
Heat of Explosion	1280 cal/g

3-2.12.2 Hazards.

a. Fire:

100° Heat Test

loss in first 48 hours	0.04%
loss in second 48 hours	0.00%
Explosion in 100 hours	none

Explosion Temperature

Seconds	°C
0.1 (no cap used)	405
1	316
5	260 (decomposes)
10	240
15	235

Vacuum stability test (cc/40 hrs)

100°C	0.7
120°C	0.9
150°C	2.5

ESD

Dry RDX	0.07 joules

Impact, 5 kg wt

Dry recrystallized RDX	125mm to 150mm
Water wet recrystallized RDX	greater than 600 mm
Coarse RDX	175mm to 225 mm
Ammonium Perchlorate	about 375 mm
NG (100%)	60 mm to 75 mm

Friction (8 ft/sec)	less than 5 lb

Electrostatics:

Dry RDX can hold a large electrostatic charge therefore, it is important for all equipment to be grounded.

Firefighting:

RDX is a high explosive and a static hazard. Do not fight an RDX fire: vacate.

b. Explosion:

Detonation Velocity	8180 m/sec
Card Gap	3.36 in (den 1.53) 2.84 in (den 1.64)

RDX is one of the most powerful high explosives used today. It is considered too sensitive to be used alone.

c. Health:

RDX can cause headache, insomnia, dizziness, restlessness and convulsions resulting from central nervous system stimulation. The convulsions resemble grand mal or epileptic seizures. It does not exhibit pharmacological effects similar to nitrates or nitrites. It can be absored by ingestion and inhalation but does not penetrate the skin. No effects from skin or eye contacts have been reported. Ventilation should be provided to reduce the inhalation hazard. Employees should be cautioned of the effects from accidental or intentional ingestion. Oxides of nitrogen and of carbon will be in the combustion products. Lung irritation may be produced either promptly or delayed a few hours after exposure.

d. Shipping and Handling:

RDX has a high degree of stability, therefore, it should not present any storage hazard in this respect. For manufacturing use, RDX is purchased from Holsten Ordnance Works coated with wax. It is shipped in water and isopropanol, where the isopropanol is used only as an anti-freeze. RDX is stored wet.

Sensitivity classification is as follows:

Wet	Class 7	Group M(for storage)
Dry	Class 7	Group L (for storage)
Wet	Class A	(for shipping)
Dry	Prohibited	(for shipping)

3-2.13 DIETHYLENE GLYCOL DINITRATE (DEGN)

3-2.13.1 Properties.

a. Physical:

Density	1.38 g/cc
Color	colorless
Melting Point	2°C
Boiling Point	160°C
Vapor Pressure:	
20°C	0.0036 mm Hg
60°C	0.130 mm Hg
Solubility in Water,	g/100 g
25°C	0.40
60°C	0.60

b. Chemical.

Molecular Formula	$C_4H_8N_2O_7$
Molecular Weight	196
Heat of combustion	2792 cal/g
Heat of formation	2020 cal/g
Heat of explosion	841 cal/g

3-2.13.2 Hazards.

a. Fire:

100°C Heat Test

loss in first 48 hrs.	4.0%
loss in second 48 hrs.	3.0%
explosion in 100 hrs.	none
Explosion Temperature:	
5 seconds	237°C
Vacuum Stability,	cc/20 hr-g
Impact, 2kg wt.	
BM	100 cm
PA, 1 lb. wt.	9 in.

b. Explosion:

Detonation Velocity	6760 m/sec

DEGN is difficult to ignite, and in small quantity is not readily exploded by heat. It may explode when shocked.

c. *Health:*

In a manner similar to other nitrate containing aliphatic compounds, diethylene glycol dinitrate may cause dilation of blood vessels, headaches, nausea, methemoglobinemia, cyanosis, and reduced blood pressure. Extreme exposures may result in central nervous system depression, coma and respiratory paralysis. DEGN is absorbed by inhalation, skin contact or ingestion. Alcoholic beverages aggravate the health hazard. Severe headache shortly after exposure may diminish after development of tolerance. A break of a few days in the work pattern interrupts this tolerance. Ventilation should be used to reduce the inhalation hazard. Protective gloves and aprons are helpful in avoiding skin contact. Frequent washing with soap and water will minimize absorption. Do not use alcohol or other solvents on the skin. A Threshold Limit Value has not been established for DEGN, however, it appears appropriate to treat it like ethylene glycol dinitrate which has a TLV ceiling of 0.2 ppm, and a skin absorption warning. Headache may be encountered at levels below 0.2 ppm. Combustion products may include CO, CO_2, and oxides of nitrogen.

d. Shipping and Handling:

DEGN is classified as Class 7. Must be desensitized to meet DOT requirements given in paragraph 173.53(e) of DOT Tariff.

3-2.14 2,4-DINITROTOLUENE (DNT)

3-2.14.1 Properties.

a. Physical:

Density	1.521 g/cc
Color	Yellow
Melting Point	69.5°C
Boiling Point	300°C

b. Chemical:

Molecular Formula	$C_7H_6N_2O_4$
Molecular Weight	182.13
Heat of Combustion	1545 cal/g

3-2.14.2 Hazards.

a. Fire:

Explosion Temperature:	
5 seconds	310°C
Vacuum Stability - cc/40 hr	
120°C	0.04
Friction (8 ft/sec)	950 lb
ESD	greater than 12.5 joule

Firefighting

DNT can be extinguished by the use of water, carbon dioxide, dry chemical, or carbon tetrachloride. Firefighting should be done by remote control.

b. Explosion:

DNT can be detonated by a very strong initiator. It may be an explosion hazard when involved in fire.

c. Health:

Signs and symptoms attributed to dinitrotoluene toxicity are dermatitis, gastritis, methemoglobinemia, giving rise to characteristic patterns of cyanosis, aplastic anemia and toxic hepatitis. The chemical may be absorbed by inhalation and ingestion and, to a lesser extent, by skin contact. Skin contact may result in staining of the skin and can give rise to dermatitis in susceptible individuals. DNT may cause irritation of mucous membranes of the respiratory tract and the eyes. To prevent skin and eye contacts, inhalation and ingestion, personal protective clothing and eye protective gear should be provided. Personal cleanliness should be enforced. Dusty operations should be exhaust ventilated. Indicator soaps are valuable to insure complete removal of DNT from the skin. DNT has a TLV of 1.5 mg/m^3 with a skin warning, (ACGIH 1968).

d. Shipping and Handling:

DNT is shipped in bottles, cans, metal barrel, drums, and wooden barrels or kegs with liners. Class 2, Group D.

3-2.15 DIPHENYL GUANIDINE

3-2.15.1 Properties.

a. Physical:

Density	1.15 at 25°C
Form and Color	white crystalline powder
Melting Point	147°C, decomposes above 170°C
Solubility	Slightly soluble in water, soluble in ethyl alcohol, carbon tetrachloride and chloroform

b. Chemical:

Symbol	NHC $(C_6H_5NH)_2$
Molecular Weight	211.1

3-2.15.2 Hazards.

a. Fire:

When heated to decomposition, it gives off toxic fumes.

b. Health:

DPG is a moderately toxic compound which dissolves readily in aqueous media to give strongly alkaline solutions. Although its toxicology has not been adequately investigated, it may cause central nervous system stimulation. It is a strong irritant to the skin and mucous membranes. Skin and eye contact should be prevented by wearing protective clothing and chemical goggles.

c. Shipping and Handling:

No shipping regulations apply. Good housekeeping and dust control are important.

3-2.16 ETHYL ACRYLATE

3-2.16.1 Properties.

a. Physical:

Specific Gravity	0.9230 (20/20°C)
Vapor Pressure	29.3 mm (20°C)
Form and Color	colorless liquid
Melting Point	-72°C
Boiling Point	99.4°C

Note: Polymerizes. Usually contains an inhibitor to prevent polymerization.

b. Chemical:

Symbol	$CH_2CHCOOC_2H_5$
Molecular Weight	100.11

3-2.16.2 Hazards:

a. Fire:

Flash point is 15.6°C (O.C.); lower explosive limit is 1.8%. Vapor is heavier than air and may travel a considerable distance to a source of ignition and flash back. At elevated temperatures polymerization may take place. If polymerization takes place in a closed container, there is a possibility of violent rupture.

Firefighting. Only slightly soluble in water. Water spray may be used to control fire; carbon dioxide, foam or dry chemical are preferred. Cool tanks or drums containing this material with hose streams to prevent polymerization and rupture. Self-contained breathing apparatus should be used as toxic vapors are given off at elevated temperatures.

b. Explosion:

Vapors, when confined, will explode when exposed to sparks or flame.

c. Health:

Ethyl acrylate can be absorbed through the skin, the lungs, and the digestive tract. It can irritate the skin, eyes, and any internal surface. It may cause lethargy, convulsions, pulmonary edema, damage to the liver and kidneys, if absorbed in sufficient quantity. The odor and irritating properties of ethyl acrylate probably will prevent most humans from allowing themselves to be exposed to toxic concentrations. Eye, skin, and respiratory protective equipment should be worn by people working with ethyl acrylate. If exposure occurs, the area exposed should be washed with copious amounts of water after contaminated clothing is removed. The exposed person should be placed in the hands of a physician for evaluation and care. The TLV is 25 ppm or 100 mg/m^3, with a skin absorption warning (ACGIH 1968.)

d. Storage and Handling:

Classified as Class IA flammable liquid by National Fire Protection Association; storage should meet requirements of NFPA for flammable liquids (NFPA Code Nr. 30). Vapors are uninhibited and may form polymers in vents or flame arrestors, resulting in stoppage of vents. It is not listed in the DOT regulations. However, it should be shipped as a flammable liquid/red label.

3-2.17 FERROCENE

3-2.17.1 Properties.

a. Physical:

Other Names	Dicyclopentadienyliron
Form	orange crystals
Melting Point	174°C, sublimes at approximately 100°C
Solubility	Insoluble in water; soluble in alcohol and ether

b. Chemical:

Symbol	$C_{10}H_{10}Fe$
Molecular Weight	186.0

3-2.17.2 Hazards:

a. Fire:

When exposed to fire, it may emit highly toxic iron carbonyl.

b. Health:

Symptoms: Ferrocene is only moderately toxic by ingestion. Small amounts produce an increased count for leukocytes, erythrocytes, and thrombocytes. Larger amounts give increasing deposits of hemosiderin in the liver, spleen, adrenal cortex, lymph nodes, and kidneys. Excessive amounts give cellular changes resembling hemochromatosis.

Absorption: The compound is readily absorbed following ingestion and is retained longer than $FeSO_4$. It is primarily concentrated in the liver.

Local Action: There are no reports of adverse local reactions to applications of ferrocene.

Preventive Measures: No protective equipment needed.

TLV and/or EEL: None have been recommended.

Combustion Products: Decomposition of ferrocene produces iron carbonyl which is a highly toxic substance.

3-2.18 LEAD AZIDE

3-2.18.1 Properties.

a. Physical:

Density	4.80 g/cc
Form	crystalline compound
Color	cream-colored
Melting Point	decomposes
Solubility, g/100 g in water	
20°C	0.95%

b. Chemical:

Molecular Formula	N_6Pb
Molecular Weight	291
Heat of Combustion	630 cal/g
Heat of Formation	-346 cal/g
Heat of Explosion	367 cal/g

3-2.18.2 Hazards.

a. Fire:

100°C Heat Test

loss in first 48 hrs	0.34%
loss in second 48 hrs	0.05%
explosion in 100 hrs	none

Explosion Temperature

seconds	°C
0.1 (no cap used)	396
1	346
5	340 explosion
10	greater than 335

Vacuum Stability - (cc/40 hrs.)

100°C	1.0 dextrinated
120°C	0.07 dextrinated

NOS

(Pure)	0.007 joule
NOS (.39% water)	.00125 joule

Impact, 2 kg wt.

	Pure	Dextrinated
BM	10 cm	17 cm
PA	3 in.	5 in.
Sample Wt.	30 mg	28 mg

Initiating efficiency, grams required to cause ignition of the following:

TNT	0.25 g
Tetryl	0.10 g
RDX	0.05 g
PETN	0.02 g

Firefighting

Do not fight fire.

b. Explosion:

Detonation Velocity about 4630 m/sec

Lead Azide is very sensitive to friction, shock and electrostatic discharge. It is used as an initiating explosive.

c. Health:

Lead azide is moderately toxic by ingestion. Like most lead compounds, small amounts administered repeatedly may result in absorption of significant quantities. The acute toxicity of lead azide is due principally to the azide. Sodium azide is more toxic than lead azide. Symptoms of acute exposure may include reduced blood pressure, excessive sweating, vertigo, nausea, fatigue, or collapse. Continued exposure to lesser quantities may cause cough, fever, injury to the kidneys and spleen or fatal convulsions.

Absorption - Azides are rapidly absorbed following ingestion or inhalation.

Local Action - Lead azide may be irritating to mucous tissue.

Preventive Procedures - Avoid breathing dust. Use a dust respirator approved by the Bureau of Mines.

TLV or EEL - None have been recommended.

Combustion Products - Lead oxide and nitrogen oxides. May also form cyanides if organic matter is present.

d. Shipping and Handling:

Lead Azide is shipped with water. Where the water is used as a wetting agent, hydrazoic acid is present in containers of Lead Azide. It is very poisonous and easily exploded by flame. Caution should be used when opening these containers. Lead Azide should not be exposed to copper, zinc or alloys containing such metals because of the possible formation of other azides which are more sensitive than the lead. Water wet Lead Azide in 1-100 gm quantities are transferred behind shields with aluminum scoops. A wash bottle is used frequently to keep all surfaces wet. All working surfaces and floors are conductive and grounded. Lumps of dry Lead Azide are never broken up but are disposed of chemically. Lead Azide is classed: Class 7, Group M (wet).

3-2.19 MAPO

3-2.19.1 Properties.

a. Physical:

Other names	tris (2-methyl-1-aziridinyl)-Phosphine Oxide
Density	1.079 at 25/25°C
Form and color	Amber colored liquid
Boiling Point	118°-125°C at 1 mm Hg
Solubility	Miscible with water and organic solvents

b. Chemical:

Symbol	$N(CH_2)_2CH_3$ P: O
Molecular weight	215

3-2.19.2 Hazards

a. Fire:

When heated to decomposition it emits highly toxic fumes.

b. Health:

Up to the present time, symtomatology observed in humans has been limited to primary irritation and sensitization of the skin. Based on animal studies, acute toxicity of the chemical may be manifested in signs relating to the central and autonomic nervous system and the gastrointestinal and genitourinary systems and may be elicited by either skin contact or ingestion. This chemical may also be absorbed by inhalation. Delayed toxicity effects may occur in the liver and in hematopoietic tissues. On long term exposure, this chemical may have carcinogenic properties. Protective clothing (rubber gloves and aprons, goggles and face shields) should be worn when handling the chemical. In the case of accidental skin contact, the skin should first be flushed with copious amounts of water and then washed with soap and water. Contaminated clothing should be removed. The chemical should be used only in a well ventilated area and should be handled, stored and disposed of with care.

No TLV has been established.

The composition of the combustion products of this chemical are unknown, however, they are noxious and irritating and should be avoided.

3-2.20 MERCURY FULMINATE

3-2.20.1 Properties.

a. Physical:

Crystal Density	4.43 g/cc
Color	white to gray
Melting Point	decomposes
Solubility, g/100 g in water	
12°C	0.07%
49°C	0.18%

b. Chemical:

Molecular Formula	$C_2N_2O_2Hg$
Molecular Weight	285
Heat of Combustion	938 cal/g
Heat of Formation	-226 cal/g
Heat of Explosion	427 cal/g

3-2.20.2 Hazards.

a. Fire:

100°C Heat Test

Exploded in 16 hours.

Vacuum Stability Test, cc/40 hours

100°C - Explodes

Explosion Temperature

Seconds	°C
1 (no cap used)	263
1	239
5	210 explosion
10	199
15	194
20	190

ESD	0.025 joule
Impact, 2kg wt.	
BM	5 cm
PA	2 in.
Sample wt.	30 mg

Initiating efficiency, grams required to cause ignition.

TMT	0.25 g
Tetryl	0.20 g
RDX	0.19 g
PETN	0.17 g

Fire Fighting

Do not approach a fire in which this material is involved.

b. Explosion:

Detonation Velocity	about 5000 m/sec

It should be kept moist. Very sensitive to initiation by friction, shock, spark, or heat in dry state.

c. Health:

Mercury fulminate can cause severe dermatitis and irritation of the eyes and respiratory tract. Chronic exposure may lead to sensitization, leukopenia, albuminuria, and other symptoms of mercury poisoning. The Threshold Limit Value for organic mercury compounds is 0.01 mg/m^3, with a warning to avoid skin absorption (ACGIH 1968.) Avoid all routes of exposure. Wash exposed skin thoroughly with soap and water. Continued exposure to combustion products may lead to absorption of mercury.

d. Shipping and Handling:

Mercury fulminate is shipped with water. Where the water is used as a wetting agent, care should be taken when handling to prevent shock or friction. It should be kept away from flame or heat. It is not appreciably affected by long storage at moderate temperatures. At higher temperatures it will become inert after long periods, stored at 95°F about 3 years, at 122°F about 10 months. Classified as Class 7, Group M (wet).

3-2.21 METHYL ACRYLATE

3-2.21.1 Properties.

a. Physical:

Specific Gravity	0.9574 (20/20°C)
Vapor Pressure	65mm (20°C)
Form and Color	colorless volatile liquid
Melting Point	-76.5°C
Boiling Point	80.5°C

Note: Polymerizes. Usually contains an inhibitor to prevent polymerization.

b. Chemical:

Symbol	$CH_2CHCOOCH_3$
Molecular Weight	86.1

3-2.21.2 Hazards.

a. Fire:

Flash point is -3.0° (O.C.); autoignition temperature not established; flammable limits are 2.8% - 25%; vapors are heavier than air and may travel a considerable distance to a source of ignition and flash back. At elevated temperatures polymerization may take place. If polymerization takes place in a closed container, there is possibility of violent rupture of the container.

Firefighting:

Only slightly soluble in water. Water spray may be used to control fire; carbon dioxide, foam or dry chemical are preferred. Cool tanks or drums containing this material with hose streams to prevent polymerization. Self-contained breathing apparatus should be used as toxic vapors are given off at elevated temperature.

b. Explosion:

Vapors, when confined, will explode when exposed to sparks or flame.

c. Health:

Methyl acrylate can be absorbed through the skin, lungs and digestive tract. It can irritate the skin, eyes and any internal surface. It may cause lethargy, convulsions, pulmonary edema, damage to the liver and kidneys if absorbed in sufficient quantity. The odor and irritating properties of methyl acrylate probably will prevent most humans from allowing themselves to be exposed to toxic concentrations.

Eye, skin and respiratory protective equipment should be worn by people working with methyl acrylate. If exposure occurs, the person should be removed from the area, contaminated clothing removed, and exposed areas washed with copious amounts of water. The exposed person should be placed in the hands of a physician for evaluation and care.

The TLV is 10 ppm or 35 mg/m^3 with a skin absorption warning (ACGIH 1968).

d. Storage and Handling:

Classified as Class IA flammable liquid by National Fire Protection Association; storage should meet requirements of NFPA for flammable liquids (NFPA Code Nr. 30). Vapors are uninhibited and may form polymers in vents or flame arrestors, resulting in stoppage of vents. It is not listed in the DOT regulations. However, it should be shipped as a flammable liquid/red label.

3-2.22 METRIOL TRINITRATE (MTN); or TRI*METHYLOLETHANE* TRINITRATE (TMETN)

3-2.22.1 Properties.

a. Physical:

Density	1.47 gm/cc
Form	liquid
Color	water-white
Melting Point	-3°C
Solubility in water (gm/100 gm)	
25°C	less than 0.015
60°C	less than 0.015
Viscosity at 20°	156 cp

b. Chemical:

Molecular Formula	$C_5H_9N_3O_9$
Molecular Weight	255
Heat of Combustion	2624 cal/g

3-2.22.2 Hazards:

a. Fire:

100°C Heat Test

loss in first 48 hrs	2.5%
loss in second 48 hrs	1.8%
explosion in 100 hrs	none

Explosion Temperature:

seconds	°C
5	235 ignition

Vacuum Stability Test	
100°C	1.9 cc/g in 40 hours
Impact:	
PA	20 in. at 1 pound
BM	47 cm at 2kg
NOS	200 mm at 5kg
Friction (8ft/sec)	950 lb
ESD	0.025 joule

MTN is flammable material and will burn as well as explode. When unconfined and in small quantity, it will burn without explosion. If the material is confined or if local heating occurs an explosion will follow.

b. Explosion:

Detonation Velocity	7050 m/sec
Critical Diameter	0.27 in.

NOL Card Gap:

0.0 - 0.05 inch gap - 7000 m/sec
0.10 - 12.0 inch gap - 1500 m/sec

Will not propagate a detonation in less than 1 in. diameter. Detonates with No. 6 blasting cap but not with No. 5 blasting cap.

c. Health:

Absorption through the skin or lungs causes lowering of the blood pressure and increase in heart action.

d. Shipping and Handling:

Shipped under DOT Special Permit 2510 Class

"A" material. Shipped in Specification DOT-25 inside polyethylene drums of five gallons capacity each, which are overpacked in steel drums conforming to Specification DOT-6J. Although MTN has been shipped without desensitizers this is not recommended. MTN should be shipped in 25 percent desensitizer such as methylene chloride.

3-2.23 NITROCELLULOSE (12.6% N)

3-2.23-1 Properties.

a. Physical:

Other names: Cellulose Nitrate, Pyrocellulose, Nitro Cellulose, Nitrocotton (Gun) cotton is nitrocellulose containing 13% or more nitrogen, usually 13.2-13.4%)

Density, g/ml	(solid) about 1.66
Form	cotton-like fibrous solid or amorphous powder (when cut up or beaten)
Color	white
Melting Point	decomposes. In a vacuum melt at approximately 473°K (200°C)
Boiling Point	decomposes.
Solubility	Numerous substances dissolve or more accurately disperse pyrocellulose. Among these are acetone, ether-alcohol (2:1), fatty acid esters, nitro compounds, and organic nitrates. Insoluble in water, ether and benzene.

b. Chemical:

Molecular Formula: $\left[C_6H_{10-x}O_5xNO_2\right]n$ When x=No. of NO_2 groups and n=degree of polymerization. For 12.67%N, x=2.45.
Molecular Weight: Variable, approximately 0.42 to 0.78 x 10^6 or 272.3 (calculated from formula).
Heat of formation: -617 cal/g.

3-2.23.2 Hazards.

a. Fire:

Dry nitrocellulose is very sensitive to friction, heat and spark. Heat can cause deterioration of NC with resultant reduction in viscosity, decreased nitrogen content and increased acidity. NC wet with solvent is a dangerous fire hazard. NC wet with water is less dangerous. Dry NC is an explosive hazard.

Impact	
NOS (3+, 5 kg)	225mm
ABL (2kg)	210mm
Friction (8 ft/sec)	520 lb
ESD	0.0125 joule

b. Explosive:

Critical Diameter - Type A, dry less than 1/4 inch
Card Gap - 1.97 in (den. 1.45)

Nitrocellulose can be detonated even when wet, when confined, and when initiated with a strong booster. A nitrocellulose water mixture (containing 40% water) confined in a steel barrel sometimes is detonated by a stick of dynamite, and frequency of detonation is greater when the water is frozen. Tests have shown that NC (uniformly wetted with 35% ethanol) will also detonate on occasions when initiated with a strong booster dynamite. Dry NC when confined in pipe closed at one end will transit to a detonation. NCW 35% ethanol 20-40% is safely handled routinely in manufacturing of Single Base and Double Base propellants.

c. Toxicity:

The vapors of most solvents are heavier than air and tend to flow downward. Therefore, the ventilation should be designed to exhaust from floor level.

d. Health:

The ingredients of nitrocellulose present no toxic hazard. However, when handling nitrocellulose wet, the alcohol preservative is denatured with 1/2 per cent benzene which, by nature of its high vapor pressure, presents a benzene inhalation hazard.

Precautions:
When handling wet nitrocellulose, it will be necessary to observe adequate ventilation measures and wear rubber gloves for prolonged exposures.
The TLV for benzene is 25 ppm or 80 mg/m^3 with a skin warning (ACGIH 1968).
The combustion products may include CO and oxides of nitrogen.

e. Shipping and Handling:

Dry NC is DOT Class A. The weight limits per container is 10 pounds of dry, uncompressed NC.
Wet NC (colloided, granular or flake) with 20% water is classed as a flammable solid.
Wet NC (colloided, granular or flake) with 20% alcohol or solvent is classed by ICC as a flammable liquid. The alcohol is usually denatured ethanol, isopropanol or normal butanol.
At no time is the alcohol entirely uniform throughout the drum. On standing the alcohol gradually settles leaving the NC on top with a lower alcohol content. NC appears to undergo very slow decomposition even at ordinary temperatures. Because of deterioration NC should be used within a few months after manufacture. Frequent stability checks should be made if stored over 1 year. As a rule of thumb

NC should not be stored over 2 years. The lids of NC drums should fit tightly to prevent evaporation of alcohol and subsequent drying of NC should be protected from direct rays of sun. Keep away from heat, open flame and ignition sources.

3-2.24 NITROGYLCERIN

3-2.24.1 Properties.

a. Physical:

Density	1.596 g/cc
Crystal Form	Labile - Rhombic Stable - Triclinic
Color	Colorless to light yellow
Freezing Point	Labile form - 2.2°C Stable form - 13.2°C
Vapor Pressure	0.00025 mm at 20°C.
Viscosity:	1.0 mm at 127°C

°C	Centiposes
10	69.2
20	36.0
30	21.0
40	13.6
50	9.4
60	6.8

Solubility:
g/100 g (%) at room temperature

Water	Alcohol	Trichlorethylene	Carbon Tet.
0.18%	54%	22%	2%

Nitroglycerin is only very slightly soluble in water (0.25% at 50°C) but it is miscible in all proportions in methyl alcohol, acetone, ether and benzene, as well as several other organic solvents. Being a chemical ester, it is saponified in alkaline solutions.

b. Chemical:

Molecular Formula	$C_3H_5(ONO_2)$
Molecular Weight	227.07
Heat of Combustion	1616 cal/g
Heat of Explosion	1600 cal/g
Heat of Formation	400 cal/g
Heat of Detonation	1486 cal/g
Autoignition Temperature	180°C
explodes	222°C

Heat of Transition

liquid to labile	5.2 cal/g
labile to stable	28.0 cal/g
liquid to stable	33.2 cal/g

At ambient temperatures nitroglycerin, free of acid, is, from a chemical point of view, a very stable compound. The presence of even a small amount of acid causes decomposition to occur, which increases with increase in temperature. For this reason, viz. removal of all traces of acid, is imperative for safe storage of the material.

The chemical stability of nitroglycerin used in smokeless powder manufacture is monitored to assure that it meets a KI (potassium iodide-starch) test requirement of 10 or more minutes as performed by the Abel heat test.

3-2.24.2 Hazards.

a. Fire:

100°C Heat Test

loss in first 48 hours	3.6%
loss in second 48 hours	3.5%
explosion in 100 hours	none

Explosion Temperature

5 seconds	222°C

Vacuum Stability Test

90°C - cc/g - 6 hr	1.6
100°C - cc/g - 16 hr	11+

ESD

Unconfined - greater than 12.15 joule

Impact Sensitivity, 2kg wt.

BM apparatus	15 cm
NOS	75 mm
Friction (8 ft/sec)	less than 1 lb

Fire Fighting

Nitroglycerin being a high explosive with low auto-ignition temperature, the best procedure to follow when a fire occurs is to vacate.

b. Explosion:

Detonation Velocity

Confinement

Glass	1600-1900 m/sec
Steel	7700 m/sec
Card Gap	0.91 in. (small scale card gap)

c. Sensitivity Properties:

Nitroglycerin is a powerful, brisant explosive. Its decomposition into gaseous products is accompanied by the evolution of large quantities of heat and the oxygen content of the molecule is sufficient for complete combustion of the carbon and hydrogen to carbon dioxide and water. Nitroglycerin is not readily flammable, however, it will ignite at 150-160°C and will explode unless burned in the manner described in Section 3-8.6. It is very sensitive to mechanical shock or impact undergoing detonation under a falling weight of 2 kg. at a heighth of 4 cm. Frozen nitroglycerin is less sensitive to impact than liquid nitroglycerin, in the order of about 1/3 as sensitive, however, a half-thawed mixture of liquid and solid nitroglycerin is more sensitive than the liquid alone, ascribed to the friction of the crystals against each other. For this reason, care must be exercised to avoid the frozen state condition and if freezing does occur, extreme care must be used in thawing same.

d. Health:

Nitroglycerin can cause dilation of blood vessels, headaches, nausea, vomiting, methemoglobinemia, cyanosis, reduced blood pressure, central nervous system depression, coma and respiratory paralysis through inhalation, ingestion or skin absorption. Alcoholic beverages aggravate the health hazard.
Ventilation should be used to reduce the inhalation hazard. Wear protective gloves and aprons (rubber can retain nitroglycerin) and wash exposed skin frequently with soap and water to reduce skin absorption. Do not use alcohol or other solvents on the skin.
The TLV is a ceiling value of 0.2 ppm with a skin absorption warning (ACGIH 1967).
Combustion products are lung irritants and may cause delayed lung injury.

3-2.25 NITROGUANIDINE

3-2.25.1 Properties.

a. Physical:

Crystal Density		1.72 gm/cc
Form		white solid
Melting Point		232°C
Solubility, g/100 g (%) in		
	°C	%
Water	25	0.44
	100	9.0
1.0 n-Potassium Hydroxide	25	1.2
40% Sulfuric Acid	0	3.40*
	25	8.00*

* - g/100 cc saturated solution

b. Chemical:

Molecular Formula	$CH_4N_4O_2$
Molecular Weight	104
Heat of Combustion	1995 cal/g
Heat of Formation	227 cal/g
Heat of Explosion	721 cal/g

3-2.25.2 Hazards.

a. Fire:

100°C Heat Test

loss in first 48 hrs	0.18%
loss in second 48 hrs	0.09%
explosion in 100 hrs	none

Explosion Temperature:

seconds	°C
5	275 decomposes

Vacuum Stability Test (cc/40 hrs)

100°C	0.37
120°C	0.44

Impact, 2kg wt.

BM	47 cm
PA	26 in
sample wt.	7 mg

Fire Fighting:

Nitroguanidine is dangerous when exposed to heat or flame or by chemical reaction with oxidizers. It can be extinguished with water.

b. Explosion:

Detonation Velocity	7650 m/sec
Card Gap	1.96 in. (den. 1.16) 32 in. (den. 1.64)

Nitroguanidine is known as a flashless or cool explosive.

c. Health:

Unknown

d. Shipping and Handling:

When handling, do not expose to flame, shock or extreme heat. It should be stored in a cool dry place and away from oxidizers. Classed as a Class "A" explosive.

3-2.26 PENTAERYTHRITOL TETRANITRATE (PETN)

3-2.26.1 Properties.

a. Physical:

Crystal Density	1.77 gm/cc
Color	white
Melting Point	141°C

Solubility:
Soluble in: acetone, benzene, tolune, methyl acetate, B-ethoxy-ethyl-acetate, chlorbenzene, TNT

b. Chemical:

Molecular Formula	$C_5H_8N_4O_{12}$
Molecular Weight	316
Heat of Combustion	1960 cal/g
Heat of Formation	383 cal/g
Heat of Explosion	1385 cal/g

3-2.26.2 Hazards.

a. Fire:

100°C Heat Test

loss in first 48 hrs	0.1%
loss in second 48 hrs	0.0%
explosion in 100 hrs	none

Explosion Temperature

seconds	°C
0.1	272
1	244
5	225 decomposes
1.0	211

Vacuum Stability, cc/40 hrs.	
100°C	0.5
120°C	11+
ESD	
Unconfined	0.06 joule
Confined	0.21 joule
Impact, 2kg, wt.	
BM	17 cm
PA	6 in.
Sample wt	16 mg.

Fire Fighting

PETN is very similar to RDX. It is somewhat more sensitive, but almost equal in power. Do not fight a PETN fire: Vacate.

b. Explosion:

Detonation Velocity	8300m/sec

PETN is one of the most powerful explosives. It is sensitive to shock or when it is exposed to heat. When decomposing it emits highly toxic fumes of oxides of nitrogen which will react vigorously with oxidizable materials.

c. Health:

Symptoms are similar to those produced by other aliphatic nitrates. It can cause dilation of blood vessels, headaches, nausea, vomiting, methemoglobinemia, cyanosis, reduced blood pressure, and central nervous systems depression. Alcoholic beverages aggravate the health hazard.
It is absorbed slowly from the gastrointestinal tract and the lung, but not to any appreciable extent from the skin. Rare instances of dermatitis have been attributed to this chemical.
Safety measures to prevent explosions of the chemical are sufficient to prevent undue health effects among workers.
No TLV has been established for PETN.
Combustion products include carbon monoxide, oxides of nitrogen and carbon dioxide.

d. Shipping and Handling:

PETN must be shipped wet with at least 40% by weight of water in metal barrels, drums, or kegs in which the material is packed in cloth or rubber bags.
PETN is stored in a wet form.
PETN is classified as Class 7, Group M.

3-2.27 PLASTISOL NITROCELLULOSE (PNC)

3-2.27.1 Properties.

a. Physical:

Form	Solid white powder made up of spherical particles or aggregates of spherical particles.
Density	Approximately 1.55 g/ml
Melting Point	Has no melting point; ignites at 160°C.

b. Chemical.

Symbol	Varies from $C_{12}H_{17}(ONO_2)_3O_7$ to $C_{12}H_{14}(ONO_2)_6O_4$
Molecular Weight	Variable, 459.28 to 594.28

3-2.27-2 Hazards.

a. Impact

Drop test (dry): greater than 600 mm with a 5kg wt.

Sliding Rod Friction Sensitivity (NOS)	
	45° angle - 5 burn
	75° angle - 5 detonations
ESD	0.00125 to 0.00525 joule

b. Fire:

Dry plastisol nitrocellulose is an extreme fire hazard and proper care should be taken in its handling and storage. Plastisol nitrocellulose is less dangerous when stored wet with solvents and water, but due to the flammability of many of the solvents, a fire hazard still exists. Generally, the same precautions should be taken for PNC as for NC.

c. Explosive:

Plastisol nitrocellulose should be considered similar in explosive hazard to nitrocellulose and the same precautions taken.

d. Shipping and Handling:

PNC is not listed by DOT. It probably should take same classification as NC. Shipping classifications for various forms should be requested from DOT.
Dry plastisol should be handled or transported in conductive containers, which are grounded at all times. Stainless steel is recommended for this purpose, since PNC may be affected by some metals. For storage and handling, it should be treated the same as nitrocellulose with the exception that the solvents utilized may require different handling.

e. Health:

Plastisol Nitrocellulose is not generally considered toxic as such, or reactive with the skin. In a wet condition, however, the solvents used in its manufacture or for shipping may be dangerous and present the greatest toxicity hazard in its handling. For example, PNC made at the Naval Ordnance Station contains nitromethane because of its method of manufacture. It is also shipped containing this solvent in addition to water. Nitromethane is a toxic and flammable material which can cause serious damage to the nervous system, liver and kidneys. It has a strong odor which should

serve as a warning to those working with the material that the concentration is too high and steps should be taken to remedy the situation. Although there is no evidence of significant skin absorption of it into the body, contact with the skin should be avoided.
In working with the dry plastisol nitrocellulose inhalation of the fine dry powder should be avoided through the use of respirators or equivalent equipment to protect the respiratory system.

3-2.28 POTASSIUM CHLORATE

3-2.28.1 Properties.

a. Physical:

Density g/ml	(solid) 2.32
Crystalline Form	Monoclinic
Color	transparent colorless crystals or white powder
Melting Point	641.5°K (368.4°C)
Boiling Point	decomposes at 673°K (400°C) giving off oxygen

Solubility: Soluble in water and alkalies; slightly soluble in alcohol.

b. Chemical:

Symbol	$KClO_3$
Molecular Weight	122.55
Percent Oxygen	39.17

3-2.28.2 Hazards.

a. Fire and Explosion:

Potassium chlorate is exploded by shock, or heat, and when rubbed in the presence of organic or reducing material. $KClO_3$ is sensitive to friction.

Firefighting:

Chlorate fires should be fought with large streams of water or with water fog.

b. Health:

Potassium chlorate can irritate the gastrointestinal tract. If absorbed, it can cause hemolysis of red blood cells, methemaglobinemia, liver, and kidney damage.
Avoid ingestion. Five grams is considered a toxic dose for adults.
One of the significant combustion products is hydrogen chloride.

c. Shipping and Handling:

Potassium chlorate is classified as an oxidizing material by DOT and requires a yellow label.

3-2.29 RESORCINOL (m-DIHYDROXYBENZENE)

3-2.29.1 Properties.

a. Physical:

Density	1.285 at 15°C
Form and color	very white crystals, becoming pink on exposure to light when not perfectly pure.
Melting Point	110.7°C
Boiling Point	281°C
Solubility	Soluble in water, alcohol, ether and glycerol

b. Chemical:

Symbol	$C_6H_4(OH)_2$
Molecular Weight	110.11

3-2.29.2 Hazards.

a. Fire:

Slight, when exposed to heat or flame; can react with oxidizing material.
Fire Fighting:
Use water, carbon dioxide or dry chemical.

b. Health:

Resorcinol (meta dihydroxy benzene) can cause dermatitis, edema, and corrosion of the skin. In addition, it may cause restlessness, methemoglobin formation, convulsions, tachycardia, and death from respiratory failure if absorbed through the skin, inhaled or ingested. It may cause injury to the eyes.
Gloves and aprons should be worn to prevent skin contact. Use local exhaust ventilation to reduce inhalation of dusts. Wash skin frequently to prevent skin absorption.

c. Shipping and Handling:

Good housekeeping and dust control are important.

3-2.30 SULFUR

3-2.30.1 Properties.

a. Physical:

Density g/ml	(solid) rhombic 2.07 monoclinic 1.96 amorphous 1.92
Crystalline Form	Sulfur exists in two stable crystalline forms and also in an amorphous form. a. Rhombic sulfur (below 94.5°C), octahedral yellow crystals b. Monoclinic (above 94.5°C), prismatic pale yellow crystals

Melting Point	Rhombic form, 112.8°C (rapid heating) Monoclinic form, 119.3°C
Boiling Point	444.6°C
Solubility	Insoluble in water; slightly soluble in alcohol and ether; soluble in carbon disulfide, carbon tetrachloride, and benzene, toluene and benzyl chloride.

b. Chemical:

Symbol	S_8
Molecular Weight	256.48

3-2.30.2 Hazards.

a. Fire:

Slight, when exposed to heat or flame. When ignited molten sulfur will burn in air producing acrid fumes of SO_2, which are irritating and suffocating. Burning sulfur can react vigorously with oxidizing material. When mixed with such materials as carbon, lamp black, fats and oils, sulfur forms mixtures which can ignite spontaneously.

Firefighting.

Use water.

b. Explosion:

Moderate, in the form of dust when exposed to flame. When compounded with chlorate and some other oxidizing agents, it forms sensitive explosive mixtures.

c. Health:

Sulfur dust is a very mild irritant and may produce conjunctivitis and irritate sensitive skin. There are a few reported cases of thiopneumoconiosis and bronchitis with emphysema resulting from prolonged dust inhalation.
TLV: None. Treat sulfur as a nuisance dust.

Preventive Measures: Provide adequate local exhaust ventilation, mechanical filter respirator, and protective clothing. Maintain work atmosphere below 15 mg/m^3.

Decomposition Products: Sulfur dioxide causes irritation and discomfort. Maintain work atmosphere below 5 ppm, SO_2 to avoid discomfort. Chronic effects are not known to occur from intermittent exposures.

d. Shipping and Handling:

DOT does not classify as hazardous material. Coast Guard classifies it as hazardous. It should be stored away from oxidizing material.

3-2.31 TETRYL

3-2.31.1 Properties.

a. Physical:

Crystal Density	1.73 gm/cc
Color	light yellow
Melting Point	130°C
Solubility, g/100 g	

Water		Acetone	
0°C	0.0050%	20°C	75%
20°C	0.0075%	30°C	95%
40°C	0.0110%	40°C	116%
80°C	0.0810%	50°C	138%

b. Chemical:

Molecular Formular	$C_7H_5N_5O_8$
Molecular Weight	287
Heat of Combustion	2925 cal/g
Heat of Formation	-14 cal/g
Heat of Explosion	1080 cal/g

3-2.31.2 Hazards.

a. Fire:

100°C Heat Test

loss in first 48 hrs	0.1%
loss in second 48 hrs	0.0%
explosion in 100 hrs	none

Explosion Temperature

seconds	°C
0.1 (no cap used)	340
1	314
5	257
10	238
15	236
20	234

Vacuum Stability, cc/40 hrs

100°C	0.3
120°C	1.0
150°C, 12 hrs	11+

ESD

thru 100 mesh

Unconfined	0.007 joule
Confined	4.40 joule

Impact, 2kg wt.

BM	26 cm
PA	8 in.
Sample wt.	18 mg

Firefighting

No attempt should be made to fight fires except from remote controls.

b. Explosion:

Detonation Velocity - 7850 m/sec
Card Gap - 2.94 in. (den. 1.43)

A powerful explosive sensitive to percussion and more sensitive to shock and friction than TNT.

c. Health:

Symptoms: This compound may cause irritation of the skin and mucous membranes which may become dermatitis with yellow discoloration of the skin after prolonged exposure. The toxic signs are anorexia, fever, vomiting, and other gastrointestinal disorders. The compound is a strong skin sensitizer. Liver and kidney damage have been suggested.

Routes of Absorption: Inhalation, percutaneous.

Preventive Measures: Protective clothing should include respirators, chemical goggles, impervious gloves and skin protection. Indicator soaps are valuable to insure complete removal of tetryl from the skin.
TLV is 1.5 mg/m^3 (ACGIH - 1968.)

d. Shipping and Handling:

Tetryl should be shipped in siftproof cloth or paper bags; metal containers enclosed in wooden boxes, fiberboard boxes, or wooden barrels; usually not over 60 pounds net weight. It can be stored dry.
Protective clothing should be worn when handling to avoid skin contact, and safety goggles for protection of the eyes.
Classed as Class 7, Group L.

3-2.32 TRIETHYLENEGLYCOL DINITRATE (TEGDN)

3-2.32.1 Properties.

a. Physical:

Clear colorless liquid
Density 1.33 g/cc at 60°C
Vapor Pressure 0.001 mm Hg
Solubility: ether
alcohol
water 0.55 gm/100 gm at 25°C
Melting Point: TEGDN supercools and resists crystallization to temperatures as low as -55°C

b. Chemical:

Formula $O_2NOCH_2CH_2OCH_2CH_2OCH_2CH_2ONO_2$
Molecular Wt. 240.172

3-2.32.2 Hazards.

a. Fire:

Moderate fire hazard by spontaneous chemical reaction. Extinguish fire with carbon dioxide or dry chemical.

b. Explosion:

Decomposes on heating; explodes at 223°C in five seconds. Keep away from heat or flame; avoid shock.

PA Impact Sensitivity 43 in.
BM Impact Sensitivity 100 cm
Card Gap 0.19 in.
Critical Diameter 1.05 in.

c. Health:

The toxicological properties of aliphatic nitrates are similar, differing only in intensity, onset and duration of effect. The symptoms include headache, nausea, methemoglobinemia cyanosis, dilated blood vessels, and reduced blood pressure sure. Extreme exposures may result in central nervous system depression, coma, and respiratory paralysis. TEGDN is absorbed by inhalation, skin contact or ingestion. The use of alcoholic beverages may aggravate the health hazard. Severe headache shortly after exposure may diminish upon development of tolerance. A break of a few days in the work pattern interrupts this tolerance. Ventilation should be used to reduce the inhalation hazard. Protective gloves and aprons are helpful in avoiding skin contact. Since aliphatic nitrates may permeate rubber, gloves should be changed frequently. Thorough washing with soap and water will minimize skin absorption. Do not use alcohol or other solvents as skin cleaners. A Threshold Limit Value has not been established for TEGDN. However, it appears appropriate to treat it like ethylene glycol dinitrate which has a ceiling TLV of 0.2 ppm and a skin absorption warning. Headache may be encountered at levels below the TLV. Combustion products may include CO, CO_2 and oxides of nitrogen.

d. Shipping and Handling:

Shipped without diluent as Class "A" in DOT Type 2A polyethylene drums encased in specification DOT 6-J metal drums each outer packed in an open-head steel drum.

3-2.33 TRINITROTOLUENE (TNT)

3-2.33.1 Properties.

a. Physical:

Crystal density	1.65g/cc
Color	light yellow
Melting point	81°C

Solubility:
Soluble in: acetone, benzene, toluene, carbon tetrachloride, chloroform, pyridine, methyl acetate, ethylene dichloride, aniline, chlorbenzene

Vapor Pressure

°C	mm hg
80	0.042
85	0.053
90	0.067
95	0.085
100	0.106

b. Chemical:

Molecular Formula	$C_7H_5N_3O_6$
Molecular Weight	227
Heat of Combustion	3620
Heat of Formation	78.5 cal/g
Heat of Explosion	1080 cal/g

3-2.33.2 Hazards:

a. Fire:

100°C Heat Test

loss in first 48 hrs	0.2%
loss in second 48 hrs	9.2%
explosion in 100 hrs	none

Explosion Temperature

seconds	°C
0.0 (no cap used)	570
1	520
5	475
10	465

130°C for 100 hrs. - no decomposition
Gaseous decomposition production starts at 160°C
240°C: ignites in 0.5 hr.

Vacuum Stability, cc/40 hours

100°C	0.10
120°C	0.23
135°C	0.44
150°C	0.65

ESD

Unconfined	0.06 joule
Confined	4.4 joule

Impact

BM	95 - 100+cm
PA	14 - 15 in.
Sample wt.	17 mg

Firefighting

TNT fires should be fought by remote control The surrounding area should be evacuated.

b. Explosion:

Detonation Velocity	6825 m/sec
Card Gap	2.82 in. (den 1.07)
	0.73 in. (den 1.62)

Small amounts will burn quietly if not confined but sudden rapid heating will cause detonation. Strong shock or high temperature will cause explosion. Combustion of large quantities may proceed vigorously and even cause detonation.

c. Health:

Signs and symptoms attributed to trinitrotoluene toxicity are dermatitis, gastritis, methemoglobinemia, giving rise to characteristic pattern of cyanosis, aplastic anemia, and toxic hepatitis. The chemical may be absorbed by inhalation and ingestion and to a lesser extent by skin contact. Skin contact may result in staining of the skin and can give rise to dermatitis in susceptible individuals. TNT may cause irritation of mucous membranes of the respiratory system and the eyes. To prevent skin and eye contact, inhalation and ingestion, personal protective clothing and eye protective gear should be provided. Personal cleanliness should be enforced. Dusty operations should be exhaust ventilated. Indicator soaps are valuable to insure complete removal of TNT for skin.

TNT has a TLV of 1.5 mg/m^3 with skin warning. Combustion products of TNT are CO, oxides of nitrogen, and CO_2, and proper precautions to prevent harmful effects should be taken.

d. Shipping and Handling:

TNT is shipped as Class "A" explosive, it represents an explosion hazard in case of fire but not in case of accident without fire. TNT can be stored in a dry condition. When handling TNT, confined areas should have a ventilation system depending on the type of work being done. For infrequent exposures respirators will suffice. Classification, Class 7, Group I.

3-2.34 ZIRCONIUM POWDER

3-2.34.1 Properties.

a. Physical:

Density (solid) g/ml	6.49
Crystalline Form	cubic
Melting Point	2125°K (1852°C)
Transition Point	1135°K

Solubility Data: Insoluble in water. Soluble in HF, agua regia, hot phosphoric acid. Slightly soluble in acids.

b. Chemical:

Symbol Zirconium	Zr
Molecular Weight	91.22

3-2.34.2 Hazards.

a. Fire:

Autoignition Temperature 500°F (260°C)
Minimum energy required for ignition of powder by electrostatic spark, millijoules:

(dust cloud)	5
(dust layer)	0.001

Impact

NOS (3+, 5 kg, wt.)	greater than 600 mm
Friction (8 ft/sec)	165 lb

Dry Zirconium powder is very susceptible to ignition and explosion in air by static electricity. Sufficient static electricity to cause ignition can be generated by aspiration of this dust into air, or by pouring the powder in air. Particles below 10 microns in size are especially susceptible to spontaneous combustion.

Powder containing 5-15% water is extremely dangerous; when once ignited it burns even more violently than dry powder. The reason for this is that high temperatures break down the water, releasing oxygen which supports the combustion. The susceptibility of dry Zirconium powder to ignition is practically eliminated if the powder is blanketed with a completely inert atmosphere such as argon or helium. Zirconium can burn in nitrogen and also carbon dioxide.

Firefighting:

Do not fight fire with water or regular fire extinguishers. Use special mixtures of dry chemical salt, dry sand or powdered talc. It is usually better to isolate the fire and let it burn out because in fighting the fire the Zirconium may be thrown into the air and explode.

b. Explosion:

The explosive range of dust clouds of Zirconium powder in air is about 45 to 300 mg/l. The range of fire and explosion is increased by heat and presence of oxidizers. It may explode in the presence of water due to evolution of hydrogen.

c. Health:

Zirconium presents no significant health hazard unless the metal becomes imbedded in the skin in which case, skin lesions (granulomas) may develop. The TLV for zirconium compounds is 5 mg/m^3 (ACGIH 1968). This TLV appears to have been set on the basis of the toxicity of zirconium tetrachloride and may be overly conservative for the oxide or powdered metal. The nuisance dust level of 15 mg/m^3 seems more realistic for zirconium oxide or powder.

d. Shipping and Handling:

Do not form a dust cloud; for example do not pour in air. Use grounded scoop to transfer small amounts. Separate storage is recommended, especially isolate zirconium from oxidizing materials. Prevent electrostatic discharge by grounding equipment and zirconium and by use of conductive shoes and floors. Eliminate electrostatic generators such as use of polyethylene equipment. Any zirconium powder that may accumulate on benches and floors should be gathered up immediately, stored underwater, and later destroyed by burning. In handling zirconium, the relative humidity should be kept above 25% to reduce electrostatic hazard. Zirconium is shipped submerged under water.
DOT Classification: Flammable Solid.

3-3 SOLVENTLESS SOLID PROPELLANT MANUFACTURE

3-3.1 GENERAL DESCRIPTION. If propellant is to be used in the form of sheets, strips, and discs, or is extruded into grains, it may be suitably made by a rolled sheet process, or a rolled sheet-extrusion process. When propellant grains with burning distances (webs) greater than 12-inch are required, the solvent process is usually unsuitable because of difficulty in removing residual solvent from the processed grains. The solventless process uses plasticizers but no volatile solvents and was developed to replace the solvent process for the large web grains. It was also developed to produce thin sheets requiring even thickness control. This process for manufacturing nitrocellulose base propellants for use as mortar increments, disc charges, or extruded grains consists of several of the following steps, depending upon the end item desired; paste preparation, drying and blending, differential and even-speed rolling, slitting and carpet roll winding, increment and disc manufacture, and grain extrusion and inhibiting.

Where processing is considered especially hazardous the operation is carried out by remote control. In the case of rolling double-base sheet propellant, where fires are not uncommon, use is made of special deluge type sprinkler systems which automatically trip by a photo-cell or a heat-sensitive detector.

3-3.2 PROCESS AND SAFETY DISCUSSION. The process steps for manufacturing solventless propellant are described in detail in following paragraphs.

3-3.2.1 Paste Preparation. Considerable flexibility is possible in the processing of solventless propellant. However, it is necessary that the relatively sensitive ingredients, nitroglycerin and nitrocellulose, be initially premixed into a workable matrix prior to subsequent processing, and this can be accomplished in small quantities in a Schraeder mixer or in larger quantities in water-slurry tanks. The required amount of nitroglycerin is added slowly as a steady stream or sometimes by aspiring with water when the water-slurry method is employed. Extreme care is used in adding the nitroglycerin to insure that it is mixed directly with the nitrocellulose and is thoroughly absorbed. Provision is made to assure that any spills of nitroglycerin or nitrocellulose are immediately removed. In this operation, the same precautions are taken as those used for nitroglycerin handling, i.e., use of lead floors and catch tanks, and avoidance of extremes of heat and cold, and direct rays of the sun.

3-3.2.2 Slurry Mixing. If slurry mixing is used, the fibrous mixture or paste is pumped to a lead-lined centrifuge and dewatered to about 18% moisture. For some applications, the paste can be drained off through a filter. Following the dewatering operation, the dewatered paste is placed in cloth bags, about 30 pounds to the bag. As a safety precaution, the bagged material is passed under a metal detector in order that any metallic objects which may have inadvertently fallen into the matrix during earlier processing can be detected, and these bags of material removed.

3-3.2.3 Equipment. Buildings for preparing paste are usually barricaded and spaced farther apart than quantity-distance stipulations. In the case of Schraeder mixers, which are constructed of brass, the lead-lining of the floor is extended up the side of the mixer to avoid openings where nitroglycerin can permeate to form pockets. Slurry-mix tanks and process water tanks are made of spark resistant copper alloy, and their stirrers or agitators are of the conical-pump-action type to prevent severe impact during mixing.

3-3.2.4 Drying and Blending. After bagging and metal detection of the paste, further elimination of water is accomplished in a forced hot-air dry building where the forced air prevents any build-up of vaporized nitroglycerin. Doors and walls are checked frequently for nitroglycerin condensate, and door hinges are oiled at the completion of each drying cycle. Care is exercised to maintain the temperature well above freezing during off-heat periods or when the material is in storage. Drying racks to hold the paste are so constructed as to allow free movement of heated air; and during storage, care is taken to avoid stacking of the paste bags to heights which could result in mass effects leading to possible detonation in case of fire. As the moisture content of the paste is reduced, 7-9% moisture, the paste becomes more sensitive to static electricity, impact, and friction. Therefore, it is important that the necessary precautions be taken to prevent the presence of paste dust on floors, particularly during the manual unloading operation at the dry house. Bags are either carried or wheeled, never dragged or skidded.

For reasons of economy, dry houses are usually of light-weight, wood-frame construction. The inside walls are made of plywood sheets, which are taped at the joints. The surface is further sealed by applying several coats of chemically resistant paint. The heat source (blower and steam coils) for the dry houses is located in a separate building outside the main barricade. All heating controls are of the fail-safe type and are equipped with remote control monitoring devices.

Following drying, the paste is manually dumped into a rotary blender, which is then operated remotely and subsequently manually discharged. It is here that any water soluble or ballistics modifying salts are added. During the loading and unloading operation, care is taken in providing masks or protective clothing to insure that dust from any toxic salts are neither inhaled nor absorbed through the skin. A routine plan for medical examination and blood tests is necessary to prevent the detrimental effect of prolonged lead exposure. Further, the building housing the blender is equipped with a wet scrubber to sweep out explosives contaminants in the air.

3-3.3 DIFFERENTIAL AND EVENSPEED ROLLING. The initial paste mixing steps bring about the intimate mixing of all ingredients and initiate the formation of the desired colloidal structures, but they do not yet have the desired low moisture and plastic consistency. The transformation of this fibrous material to a less sensitive rubbery plastic is accomplished by working on differential and evenspeed rolls. The remotely operated differential mill is a conventional mill having two horizontal, parallel cylindrical rolls which are heated (210°F) and which rotate at different circumferential speeds. The paste can be inspected as it is added to the rolls, where it soon adheres to the faster of the two rolls. The other roll subjects the propellant to a shearing action which works the material and changes it to a colloided sheet. In case of fire, a quick-acting deluge system protects the equipment. The elevated temperature of the rolls causes the moisture content to be reduced to about 0.6%. Rolling time is relatively short, usually requiring 1-1/2 to 3 minutes to produce a sheet approximately 0.040-inch thick. A doctor blade removes the sheet in preparation for the final rolling process.

In order to obtain a homogeneous sheet of propellant free from air bubbles and surface irregularities by a less harsh rolling action, the differentially rolled sheets are subjected to evenspeed rolling. These rolls are similar to the differential rolls except that both rotate at the same speed, and are set at a lower temperature (150°F). These evenspeed or final roll mills are operated manually and operators wear fire-protective clothing, including head gear and heavy gloves. Rolls are constructed of hardened steel to provide long wearing surfaces and to give positive rolling action without undue slippage of propellant. All rolls are periodically inspected for cracks, pits, wear and gap settings. Fire prevention is further aided by the use of preheated rolls and preheated propellant; otherwise, the high viscosity material slips in the bite of the rolls and undergoes undue friction. For very thin sheets, progressive decreases in roll gaps are employed to obtain desired sheet thicknesses and to prevent flash fires from too heavy bites.

Roll buildings are usually built without barricades, since flash fires rather than explosions are to be expected. As a further precaution to lessen the chance of an explosion, pressure relief blow-out panels are installed in the roof sections. These buildings are also force ventilated to prevent accumulation of vapors from the nitroglycerin and lead salts and to provide as much comfort to the operator as possible.

3-3.4 SLITTING AND CARPET ROLL WINDING. For increment or disc propellant processing, or for horizontal, extrusion pressing, the evenspeed sheets are cut into strips with a slitting machine. Where extrusion processing is required, the strips are fed into a winding machine, which coils them into rolls having a diameter slightly less than the inside diameter of the extrusion press. Safety glasses and safety clothing suffice for handling the propellant through the slitting and carpet roll operations, since at this stage, the propellant is well colloided and relatively insensitive. The same type building construction is employed for these operations as that for the roll houses. Quick-acting deluge systems protect both the operator and the equipment.

3-3.5 INCREMENT AND DISC MANUFACTURE. When trench mortar increments or discs are desired as the final product, the propellant is usually rolled to thicknesses of about 0.005 to 0.025 inch on evenspeed, final roll machines. The final rolled sheets are stacked and cut or punched, depending upon the end item desired. For trench mortar increment production a number of trimmed sheets are sewn together prior to cutting and punching. In this area, the operations are controlled remotely, and the operator observes the process through a viewing port. Under no condition is the equipment (shear press, sewing machine, slitter, punch press, etc.) left unattended while in operation. The operator is in position to stop the machine if malfunction should occur. Only the minimum tools, equipment, and material necessary for the immediate operations is permitted inside the operating bay. All equipment is grounded, and building and equipment is protected by quick-acting deluge systems. The individual machining operations are divided with reinforced concrete walls.

3-3.6 GRAIN EXTRUSION AND INHIBITING. The hydraulically operated extrusion presses for solventless propellant differ from those used for solvent type propellant by having (a) a means for heating both the jacket and die, and (b) a vacuum system for removing the air from the propellant cylinder. The type of press commonly used is mounted horizontally and has a piston diameter of about 15 inches. The feed for the press consists of a number of carpet rolls stacked one next to the other. A 15-inch press will require a charge of approximately 135 pounds of propellant. The propellant is heated to about 100-120°F before it is placed in the press. Barrel and die temperatures are adjusted from 130 to 160°F depending upon the properties of the propellant. Extrusion is effected at a hydraulic pressure of about 1500-2500 psig. In a 15-inch press, a hydraulic pressure of 1500 psig is equivalent to 7,275 psi on the propellant. Because of this confinement, the quantities involved, and the possibility of press shorts, all operations are controlled remotely.

The outside diameter of the grain produced by extrusion is usually considered limited to a maximum equivalent to 1/3 the diameter of the pistol of the press. Larger grains tend to be poorly consolidated. This is why extruded grains are seldom made with diameters of much over seven inches. Since no volatiles are present during extrusion, the press dies have nearly the same shape and dimensions as the strand, and close sectional details including sharp angles can be reproduced with fidelity. Only single dies are used, since work with multiple dies showed uncontrolled flow rates among the several strands and a high heat build-up in some of them. Also, to prevent the generation of excess heat, strand rates are kept under 90 inches per minute. The press is housed in steel lined, concrete butted, earth-covered structures, which must be strong enough to withstand an explosion of the entire contents. Press buildings are usually located to allow the die end of the press to face an earth abutment, in case of a press shot and the ejection of the die or die block.

Following extrusion, the grains are sawed to length and annealed to remove any stress concentrations. Most grains are tubular in design with burning initiated on the inner surface and advancing to the outer surface. When outside inhibiting is required, the outer surface is machined to provide a constant diameter both along the length of the individual grains and among different grains. Machining is accomplished with a lathe or with a hollow-head mill used for machining wooden dowels. Cutting and doweling are continuous remote operations with the operator being protected by heavy shields. Following machining, the grains are X-rayed for voids and then spiral-wrapped with three or four overlapping layers of plastic tape which are cemented in place with a solvent. An automatic tube winding machine is used for this operation. An operator can work immediately adjacent to this machine, and the main safety requirements are good grounding of the equipment and forced air ventilation to prevent continuous inhalation of solvent fumes from the cementing process. Following inhibiting and after a short drying period, the final product is ready for loading into rockets.

3-4 CASTING AND CURING OF COMPOSITE-MODIFIED DOUBLE-BASE PROPELLANTS

3-4.1 GENERAL TECHNIQUE. In its simplest form, the casting and curing of composite-modified double-base (CMDB) propellants involves combining casting powder and casting liquid (solvent) in a mold, and subjecting them to conditions of time and elevated temperatures to form a high quality rocket propellant. The granules of casting powder are small right-cylinders which contain the solid ingredients of what will be the finished propellant including plasticizers, oxidizers, fuels, stabilizers and principal binder-forming material. The casting liquid normally consists of a high-energy liquid, such as nitroglycerin, inert plasticizers and stabilizers.

3-4.2 HAZARDS (GENERAL). Potential hazards exist during casting and curing of CMDB propellants because both the casting powder and casting liquid can be initiated under abnormal conditions and because both components, under some conditions of use, can explode if initiated.

The in-process hazards of CMDB propellant system are related to the same energy sources as any other propellant system: impact, friction, electrostatic, high levels of shock, chemical contamination, and degradation at temperature levels beyond acceptable safety limits. Each ingredient or combination of ingredients must be evaluated for its sensitivity to these potentials. Factors of safety must be established and production processes and production equipment must be carefully analyzed in detail to ensure that the required factors of safety are not compromised, either intentionally or accidentally.

3-4.3 GENERAL SAFETY PRECAUTIONS. All safety maxims for process operations involving initiation-sensitive materials have two principal objectives: to eliminate the possibility of initiation of any of the materials, and to minimize the consequences of any accidents should they happen. Many of the safety regulations used are general in nature. The level of application should be directly related to the sensitivity of the materials being processed. It cannot be emphasized too strongly that careful studies must be made to establish these levels of sensitivity. After the sensitivity of a particular formula is known, the effectiveness of preventive precautions will be directly related to the force of the rules established by management to control the operation. Following the guidelines listed below will considerably reduce critical conditions:

a. Explosives should be handled and processed in a way to avoid or minimize spilling, jarring, impact, friction, electrostatic buildup and generation of dust.

b. While an operation involving explosives is being performed, objects not being used in that operation and not permanently fastened to the building should not be permitted within the immediate working area.

c. Flammable solvents used for cleaning should not be permitted in the operating bay while explosives are present.

d. Sampling of explosives should be the minimum, in both size and number, to adequately control the process.

e. All explosive spills should be immediately reported to supervisory personnel and cleaned up under their direction. One drop of liquid explosives out of place should be considered a spill.

f. Materials (propellant materials and process ingredients) which may come into contact with explosives should be tested for compatibility.

g. During the performance of an explosives processing operation involving personnel, no other operation which involves manual or remote manipulation of tools or equipment or movement of explosives should be performed.

h. Personnel and explosive limits and quantity-distance requirements to be observed should be determined on the basis of minimum requirements.

i. Explosives should be shielded from the direct rays of the sun.

j. High standards of housekeeping should be mandatory.

k. Tools, equipment and facilities should be inspected each shift before beginning operations in a particular bay or building.

l. No operation should be performed without approved written procedures and specific authority from operating supervision.

m. The approved procedures should be carefully developed in sequential steps and should be explicitly followed for all operations.

n. Conductive safety shoes, flameproof coveralls, and approved eye protection should be worn by operating personnel.

o. All containers for explosives or sensitive ingredients should be thoroughly inspected for defects and foreign material before each use.

p. Hand tools, when not in immediate use, should be placed in designated locations and secured to prevent falling.

q. Personnel should not ride in the cargo hauling space of transportation equipment when explosives, propellants or sensitive ingredients are present.

r. All manually operated electrical switches or equipment controls should be properly identified.

3-4.4 SAFETY PRECAUTIONS DURING MOLD LOADING. Failure to determine the force necessary to ignite base grains has caused great loss in recent years. It is imperative to assure that the forces necessary for ignition do not exist in the process. Specific cautions are given in the following paragraphs.

a. If pneumatic transport to the loading device or pneumatic dispersers are employed, the initiation hazard of impinging particles of base grain should be considered.

b. The free-fall impingement hazard also should be considered as some base grain particles may be initiated at less than their terminal velocity.

c. Dust generated during mold loading operations may be explosive and should be removed or minimized; inert atmosphere can be used to minimize dust.

d. Actual loading should be performed remotely.

3-4.5 SAFETY PRECAUTIONS DURING CASTING. Do not be fooled by the simplicity of the rules given in the following paragraphs. Everyone who is familiar with the use of casting solvents can relate stories of the costliness of failure to follow these rules.

a. The casting sequences should be controlled from a remote location. If remote control is not possible, only one manually controlled casting station should be in operation in a processing building at any one time.

b. During casting, movement of desiccators or cast molds, or charging of desiccators, there should be no concurrent manual or remote manipulation of tools or equipment or movement of explosives.

c. Waste liquid explosive accumulation should be limited within the operating bay to that generated by the operation being immediately performed.

d. Desiccators for casting liquids should be protected from falling or moving objects by a cushioning material that is resistant to liquid absorption.

e. Adjustments in composition of the casting liquid in the casting building should not be permitted.

f. Sampling of casting liquids within the confines of a casting building should be minimized or eliminated, if possible.

g. Precautions, such as the use of traps or other devices, should be taken to prevent explosives from entering air or vacuum lines.

h. All tooling used should be designed for simplicity and ease of cleaning, and to prevent entrapment of explosive materials in threads, blind holes, etc.

i. When remotely controlled operations are in progress, a visual signal of their occurrence such as warning lights, should be given.

j. In the movement of liquid explosives, each hose connection and hose plug installation should be secured with double retainers.

3-4.6 SAFETY PRECAUTIONS DURING CURING. Having successfully filled the mold with base grains and solvents does not relieve the necessity for continued care during the final cure process. Some of the specific rules follow.

a. Operations should not be performed simultaneously on more than one case unit at a time.

b. Casting liquid sampling, or manual addition to or removal of explosives from a unit on cure should not be permitted.

c. The minimum solvent pressure needed to assure a quality product should be used on the casting liquid.

d. Cure bay temperature control mechanisms should contain fail-safe devices as well as dual controls.

e. Units being placed on cure should be electrically grounded immediately after being positioned in the cure bay.

f. No work should be performed on hot pressurized units.

3-4.7 DISASSEMBLY OF CASTING MOLDS FOR DOUBLE-BASE PROPELLANTS.

3-4.7.1 General. Basically two types of motors are involved in the disassembly of cured motors:

a. Cartridge loaded motors

b. Case-bonded motors

The propellant grain for cartridge-loaded motors is cast in a beaker, made for example of cellulose acetate sheet, mounted in a "corset" to withstand the vacuum and pressure at various stages of the casting operation.

Two general levels of hazard can be distinguished depending on the hazard classification of the propellant; Class "B" propellants made with casting solvent containing 75% NG or less; and Class "A" propellant made with casting solvent containing more than 75% NG.

3-4.7.2 Technique. The cured propellant is transported to the disassembly area in a frame constructed so as not to permit tilting or falling over. The molds for high energy propellant (Class A) are provided with slots where the threaded bolts pass through end flanges, and the threaded bolt ends are covered with plastic thimbles. The pressure on the grain and on the solvent is released before the propellant is allowed to cool. Excess solvent is either syphoned off, removed with an aspirator, or absorbed in cotton waste. The propellant is allowed to cool before any other operation is begun. In the case of Class A propellant, the 90% NG on top of the grain will have been replaced by Triacetin.

Casting solvent hoses are removed from the top and bottom of the mold. After the disassembly of the top closure plate and the pressure plate, additional solvent is removed as described above.

The core is broken loose with an air or hydraulically operated jack. All parts are promptly cleaned. Excess propellant is trimmed. Finally the propellant is covered with conductive plastic or with metal foil.

3-4.7.3 Safety Precautions.

a. Reference is made to 3-4.5 and 3-4.6 Safety Precautions for Casting and Curing of Double-Base Propellants. The precautions listed in these sections also apply to the disassembly of molds or motors.

b. Only one motor is to be disassembled at one time.

c. Wait till the motor has cooled below the specified temperature before casting solvent is removed.

d. All contaminated parts must be promptly removed to the cleaning area.

e. The jacking or "breaking-loose" of cores must be done remotely. Do not exceed the specified limiting force.

f. Wear goggles when removing solvent hoses from the mold or when removing solvent from the top of grains.

3-4.8 CORE REMOVAL.

a. Once the core, or mandrel, in a rocket motor has served its purpose of molding the internal propellant configuration during cure, the core must be removed. Since most cores are constructed with a tapered and smaller diameter dimension at the forward end than at the aft end, the critical portion of the core removal process is the freeing of the cores, or "core popping."

b. "Core popping" is the application of a force on the core to overcome the adhesive force existing between the propellant and the mold release material on the core. During this part of the removal process, the propellant is subjected to stresses, some of which could be highly localized or concentrated; therefore, the movement of core at moment of initial release shall be controlled. Tears in the propellant or separation at bonded interfaces could result if the operation is not accomplished judiciously.

c. During "core popping" operation personnel shall be afforded adequate protection. Once the core has been "popped" final removal from the motor should be routine. Care must be exercised, however, to prevent propellant damage, dropping of the core or damage to the mold release agent through mishandling. After removal, the core should be inspected for propellant contamination and any contaminant removed. In no case shall cores be cleaned as they are removed from the motor.

3-5 INERT DILUENT PROCESSING

3-5.1 CONTINUOUS, REMOTE PROPELLANT PROCESSING. Research in the propellant industry has resulted in several generations of high-performance double-base-type propellants. The cast double-base compositions have been extensively used in solid propellants. Further development resulted in a generation of propellants which possess a higher order of energy by the incorporation of solid ingredients such as ammonium perchlorate, aluminum, and HMX. These systems are characterized by low binder and high solid-oxidizer levels and have been termed composite modified double-base (CMDB). Normally they are manufactured as described in Sections 3-3 and 3-4 of this chapter.

Recently, however CMDB propellants have been produced by a process involving the mechanical mixing of all of the propellant ingredients, liquids and solids, to form a slurry and subsequent casting of this material into a rocket chamber. This method may offer advantages of greater safety and economy through reduction in the number of operations, reduction in work input to the material processed, and reduction of personnel exposure. Other advantages that may result are shorter lead-time requirements for composition changes and greater versatility in the types of processible ingredients. Despite any safety advantages of slurry processing, mechanical mixing remains as an integral part of the process, and several accidents occurred during its development. With the advent of higher energy propellants, it is increasingly evident that a departure from this practice may be necessary. A very real demand for an efficient, safe, and preferably nonmechanical mixing process evolved. In 1962 the Navy entered into negotiations with industry on an extension of the Quickmix process to include double-base and CMDB processing. This subsequently became known as the inert-diluent process.

3-5.2 PROCESS DESCRIPTION. The inert-diluent process fulfills the processing need by providing a system that is both continuous and nonmechanical in mode of mixing. In this process, all of the propellant ingredients are handled in the desensitized form of a slurry, which is made with a process carrier liquid. Process carrier liquid is basically an ingredient transfer and processing medium and is optimumly characterized by the following criteria:

Low specific gravity (<1.0)

High vapor pressure

Nonflammability

Compatibility with formulation ingredients

Low or nonsolubility of process ingredients

Low viscosity (<10 cps)

Low surface tension (<20 dynes/cm)

Low toxicity

Low cost

The carrier liquid commonly used meets all these criteria except flammability and marginal toxicity.

The process is an adaptation of pipeline mixing to the manufacture of propellants. As shown in Figure 3-1, the liquid and solid propellant ingredients are metered into a high-velocity liquid carrier to form dispersions of the propellant ingredients in the carrier. The resultant individual slurry streams meet and are intimately contacted in a nonmechanical mixer. The mixer utilizes stream turbulence to effect uniform particulate distribution in the process stream. Primary separation of the mixed propellant from the carrier is performed in a gravity separator with tangential entry. This is followed by removal of entrained carrier during degassing and vacuum casting of the propellant. The propellant can be cast into either a retaining vessel or directly into a rocket motor casing. The separated carrier is recycled. Where double-base and CMDB processing is concerned, contamination of the recycled carrier liquid stream with potentially hazardous ingredients is a concern. Continuous monitoring is conducted on a sampling stream from the carrier overflow line. The stream is scanned for dissolved nitroglycerin by infrared spectrophotometry. The detection sensitivity of the MSA system is 0.04% when the absorption band of 1667 cm^{-1} is used. The nitroglycerin content has always been below the established safe upper limit of 0.8% during processing operations. Curing of the cast motor may be accomplished by standard techniques in another location or may be done by temperature controlling of the motor in the place where cast.

3-5.3 PLANT DESIGN. In order to use the processing advantages and capabilities available in the inert-diluent system, it is necessary to characterize the system from processing feasibility, safety and engineering design standpoints.

3-5.3.1 Equipment. Safety tests must be conducted to determine that ingredient slurry sensitivites, compatibilities, settling rates and densities are within the realm of processing reality. Suspect potential hazards in carrier wetted equipment are basically pump and valve items. These must be modified and hazard tested to satisfaction, respectively, for suitability to a safe processing system. The remotely operated pneumatically actuated, diverter valves (Durco plug type) should be dynamically tested while wetted with representative explosive/carrier mixtures. Usually a Teflon seat incorporated in the valve configuration will provide sufficient lubricity to insure against friction initiations. (Example: A Durco 3-way valve was actuated through 100 complete cycles in both the horizontal and vertical positions while loaded with (a) 25/75 Olin Ball "A" nitrocellulose/carrier liquid, (b) same on a 70/30 weight ratio, and (c) 25/75 uncured double-base propellant/carrier liquid. No signs of damage to equipment or degradation of process materials were encountered.) The centrifugal process pumps, which provide the motive force necessary to transfer the slurry streams through the process lines and maintain the process streams in dispersion, should be modified in several ways to insure safe operation. Specially cooled seals should be adapted and a throat flush system be provided to prevent the migration of solids into these potentially sensitive areas. In addition the pump heads should be turned 180° so that open impeller pump and housing can drain free, each time it is stopped. The pumps should be tested for incidence of cavitation by operation under a variety of head and process fluid conditions while maintaining photographic surveillance of the pump, specially modified with a plexiglass face plate to permit internal viewing under dynamic load conditions. Cavitation, if any, should occur only at the eye of the impeller and under severe restrictions of flow to the inlet of the pump. The safe operating conditions thus defined should be well within the realm of normal processing limits. An interlock system must be provided so that the pump cannot be operated under severe inlet flow restriction conditions. Numerous critical diameter tests (both detonation propagation and deflagration-to-detonation-transition tests) on various concentrations and combinations of ingredient and propellant slurries must be conducted to verify the safety of the processing approach. (Example: Piping must be maintained below the critical diameter found in test mentioned above.)

3-5.3.2 Design Features. In order to build a safe full-scale processing facility, certain design and equipment features must be reviewed. This design development includes the following:

a. Establish standby flush systems to insure purging of process lines with clear carrier liquid to prevent solids from settling out in the system, in the event of a power failure.

b. Establish safe operational conditions for pumps as described above.

c. Collect operational data on pressure drop, flow rates, component performance data, etc., on which to improve component design or plant configuration. (Example: (1) Disperser redesign to improve pump performance, solids slurry delivery rate and uniformity of solids distribution. (2) Design of optimum configuration vessel to effect the best propellant/carrier gravitational separation. (3) Dynamic testing of turbine flowmeters and check valves to determine suitability and surface finish requirements for use in contaminated carrier service.)

3-5.3.3 Controls. To obtain safe operations continuous controls are necessary. In order to accomplish this in the continuous processing of the inert-diluent system, three related processing phenomena have to be considered:

a. Precision of ingredient introduction into the system

b. Detection and control around the establishment of processing equipment

c. Special provision for material processed in a nonequilibrium state of operations (diversion of out-of-spec. material to separate accumulation).

3-5.3.4 Engineering. Translating this into plant operation and process design means that extra engineering effort must be provided to assure:

a. The design and provision of ingredient feeders which demonstrated a high degree of accuracy (by weight) in delivery rate

b. An accurate system equilibrium sensing and signal feedback with the quick response necessary to effective system control and equilibrium maintenance

c. The capable instruments to distinguish poor product from good product during processing and to segregate the poor material by timely stream diversion and separate accumulation.

3-5.3.5 Feeders. A solids ingredient feeder must accurately weigh increments of material, and by computer control, deliver each increment in a time that is precisely in proportion to the weight of the increment with an accuracy of better than ± 0.25% (by weight).

The liquid ingredient feed (usually an explosive material) must meter into the system with an accuracy of ± 0.10% (by weight) by a liquid volumetric displacement principle capitalizing on the precise methods of measuring insensitive liquids, and transposing them into sensitive processing capability without the hazards.

3-5.3.6 Blending. Blending of the solids slurry and liquid emulsion streams should be effected in an in-line nonmechanical mixer which contains no more than four ounces of material at a given instant. (This coupled with the fact that piping carrying the streams are smaller than the critical diameter of the involved material is an outstanding safety improvement.) Following the mixer a diversion valve is positioned to enable the segregation of mixed material that does not meet the established quality, as determined by instrumented measurements of system equilibrium.

3-5.3.7 Separation. Acceptable material is separated from carrier liquid by difference in specific gravity and vacuum cast (removing residual carrier liquid and any entrained gases) into a mold of the desired configuration.

3-5.3.8 Safety in Design. A well instrumented plant is capable of remote and therefore safer operation:

a. Major areas of function (separate ingredient feeds, control, casting, etc.) are physically separated by direct line distances from 250 feet to 1200 feet. (This allows for isolation of bulk explosive ingredients and limits the extent of attendant damage, in the event that an incident should initiate in one of the functional areas.)

b. Processing lines sized below the critical diameters to insure the real advantages of functional area isolation.

c. Below grade structures housing functional areas of ingredient feeding and dispersing, casting and control.

d. Cure-in-place and remote vacuum casting capability in the cast-cure pits.

e. Specially designed (solids feeders, dispersers, etc.) and modified (processing pumps, valves, etc.) processing equipment to enable gentle material handling yet deliver performance to operational objectives.

f. Inert gas blanketing in areas of usual process material exposure to air (transfer points and feeding locations). This suppresses or eliminates dusting and/or vapor accumulation to a point where the lower explosive level could be reached.

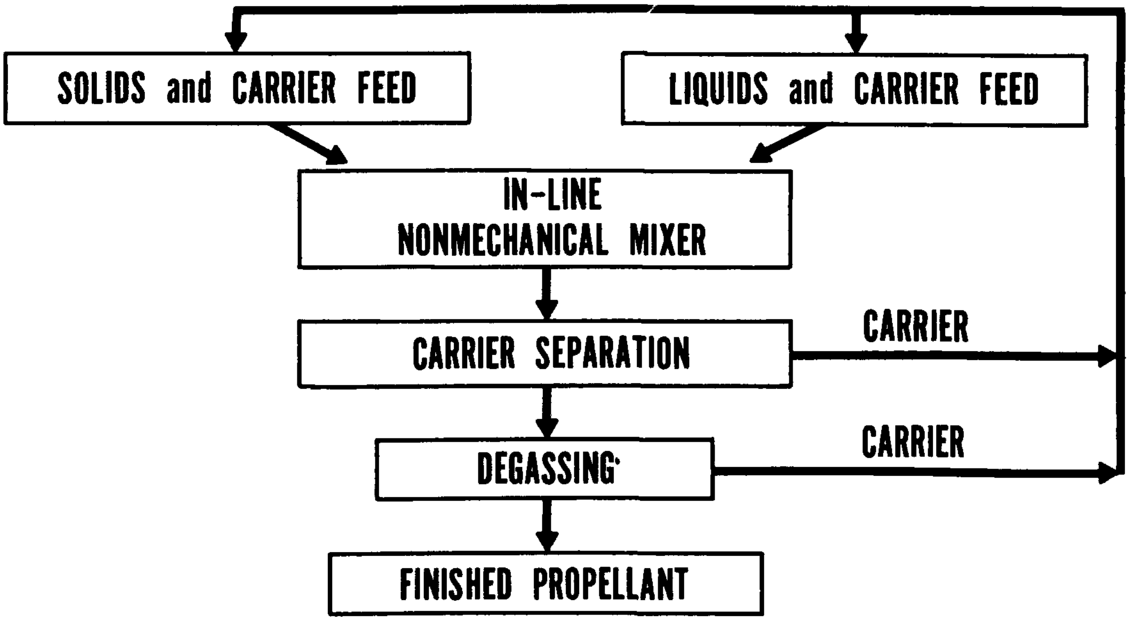
g. Frangible and low mass coefficient materials (plastics) used for operational area coverings.

h. Processing and transfer of ingredients dispersed in a carrier liquid, providing a state of lesser sensitivity and more fluid mobility.

i. Incorporation of the inherent safety features of the system, nonmechanical, low hold, high throughout capability mixer.

Figure 3-1

INERT DILUENT PROCESSING CONCEPT



3-6 MANUFACTURING COMPOSITE PROPELLANTS

3-6.1 INGREDIENTS IN GENERAL.

3-6.1.1 Raw Materials. The manufacturing, processing and handling of the wide variety of raw materials now utilized in the formulation of composite propellants is one of the most significant safety problems associated with composite propellant manufacturing. The advent of high energy oxidizers, improved burning rate catalysts and toxic fuels demands full attention to what has formerly been regarded as a simple support function.

3-6.1.2 Characterization. The complete safety characterization of each candidate ingredient is essential prior to its incorporation into a composite formulation. In addition to ingredient characterization and normal compatibility tests, tests should be conducted to determine the probability of ingredient changes under process conditions which may lead to the formulation of intermediate compositions more hazardous than the final product. Example: Ingredient changes from solid to gaseous state or sublimation and recrystallization in the presence of other unincorporated ingredients during mixing.

3-6.1.3 Laboratory Tests. Extensive laboratory tests and operations should be conducted on ingredients and formulations to project their behavior and processability before scale-up to larger quantities or production batches is made.

3-6.1.4 Oxidizers. Selection of solid oxidizers is presently limited to those commercially available from the chemical industry. Ammonium perchlorate is the workhorse oxidizer of composite solid propellants. Several other non-metallic perchlorates are being studied actively because of their potential energy contributions. Two such perchlorates are Hydrazinium Diperchlorate and Nitronium Perchlorate.

a. Ammonium Perchlorate. AP does not present significant handling problems. It can be a strong irritant to the skin and mucous membranes, but is relatively harmless for short term contact. It is not particularly sensitive to impact. Industry data show a 50 percent fire value of 114 kg-cm; no explosions were noted in a Bureau of Mines friction testing apparatus. AP is most sensitive to ignition at a moisture content of 6.02 to 0.5 percent, particularly when contaminated by copper or other metallic salts. Tests have shown that clothing contaminated with AP is extremely flammable. Recent tests (susceptibility to detonation) show that small (15 microns or less) size AP is more susceptible to shock initiation than the large particle sizes. The present trend in industry is to process small particle AP as a Class 7 military explosive item.

b. Hydrazinium Diperchlorate. HP_2 is more shock sensitive than AP. The presence of free acid (perchloric acid) on or in the crystals makes the material more susceptible to decomposition than the acid free material. Contact with or contamination by organic materials should be avoided. During processing, HP_2 should be regarded as a Class 7 military explosive. Friction and impact data show HP_2 to be more sensitive than AP.

c. Nitronium Perchlorate. NP is processed for use with compatible binders in conventional equipment but under stringent moisture control. Dehumidification and/or blanketing with dry gas is a necessity. Neoprene gloves may be used, but will react slowly, become soft, and eventually inflame. They must, therefore, be changed regularly. Clothing may inflame on contact with NO_2ClO_4, and, therefore, protective clothing made of Armalon or polyethylene should be used when handling large quantities. Skin contact results in nitric acid burns, which should be treated accordingly. Spills or dust must be cleaned promptly with large quantities of water. Toxicological problems are essentially those of nitric acid, nitrogen oxides, perchloric acid, and chlorine oxide, all of which may result when NP is exposed to contaminants, heat, moisture, or the atmosphere. Pure NP does not detonate at 300 kg-cm, but may become shock sensitive if contaminated.

3-6.1.5 Oxidizer Facilities and Equipment.

a. The use of wood or other porous organic materials in the construction of oxidizer facilities should be minimized. When the use of wood or other organic materials is required, such materials should be coated with impervious flame retardant paint.

b. Copper and copper alloys may form highly sensitive compounds when exposed to oxidizers such as ammonium perchlorate. They should not be used in oxidizer-processing facilities.

c. Oxidizer preparation should be conducted in closed systems where possible. Local exhaust and other dust control measures should be employed to prevent dust suspension and accumulation.

d. Flanged pipe and equipment connections should be incorporated to facilitate and insure safe dismantling of systems. Such systems should be provided with fire retardant, compatible, flexible connections.

e. Since oxidizing materials will cause oxidation of many metals used in process equipment, rigid preventive maintenance schedules should be established and followed.

f. All oxidizer process equipment should be electrically bonded and grounded.

g. Organic lubricants composed mainly of carbon and hydrogen are incompatible with strong oxidizing materials such as ammonium perchlorate and may form sensitive or explosive compounds when allowed to mix. Oxidizer preparation operations should be carefully studied to avoid inadvertent contamination of oxidizer with organic lubricants.

3-6.1.6 Oxidizer Preparation.

a. Oxidizers and fuels preparation should be conducted in separate facilities and with separate equipment. If facilities and equipment used to process oxidizers must be used for fuel processing, precautions should be taken to preclude contamination of one by the other.

b. Maximum safe temperatures should be established and employed when drying oxidizers. Equipment should be provided with dual temperature controls. Certain high energy oxidizers may, during drying, evolve products capable of starting a decomposition of the oxidizer. The decomposition may become autocatalytic. In such cases, adequate purging, venting and warning systems must be designed into the drying equipment.

c. Oxidizers should be screened before particle reduction operations are conducted. Reduction equipment should be designed to preclude pinching or metal-to-metal contact. Where possible, systems should be designed for continuous removal of reduced particles. This is particularly important when the reduced particles are extremely small, i.e., 15 microns or less.

d. Many oxidizers are hygroscopic. This is especially true of certain recently developed high energy oxidizers. Design of systems and equipment should incorporate provisions for exclusion of moisture. Moisture entrapped in oxidizer particles or surface moisture may create process difficulties or, as with some oxidizing agents, may increase the sensitivity of the material.

e. Oxidizers should be stored in facilities used only for that purpose. The separation of oxidizers from fuels and other organic materials is essential.

f. Objects such as jewelry, pens, pencils, rings and other items that may accidentally be introduced into oxidizer process equipment should not be permitted in oxidizer preparation facilities. All tools should be accounted for by use of tool check lists or shadow boards.

g. Oxidizer screening, blending, grinding and mechanized drying operations should be conducted remotely. Careful consideration should be given to the type of shielding required when remote operations (intraline distance) cannot be accomplished. Recently developed high energy oxidizers have undergone explosive reactions during drying and blending operations.

3-6.1.7 Fuels.

a. The discovery that metallic fuels could be incorporated into the binder-oxidizer mixture to provide higher energy, and high density propellants without affecting the mechanical properties of the system (i.e., without decreasing the allowable binder level), was a major advance in propellant technology.

b. Aluminum powder is the most commonly used metal fuel in castable composite propellants. Normally, aluminum powder is composed of irregularly shaped particles with an average particle size of 14 to 21 microns. In addition, spherical aluminum is used to lower propellant viscosity during processing. These particles are more nearly spherical, and the average particle size is between 9 and 15 microns.

c. Powdered aluminum and air form a flammable and explosive mixture. Water or moisture in contact with this metal will cause a rise in temperature that may lead to ignition. Hazardous condensation of moisture may occur when aluminum powder is transferred from a cold storage area to warm, humid air. The powder temperature should be brought to, or near, room temperature prior to processing.

d. Aluminum powder can be ignited by sparks discharged from an accumulation of static electricity. Static electrical charges most generally are produced by the flow or movement of the particles.

e. Beryllium and beryllium hydride also are being studied as high energy fuels. The beryllium supplier normally has slurried this metal (after passivation) with the binder and then shipped it to the propellant manufacturer. This precludes beryllium dust, which causes toxicity problems, in any process operation.

f. Generally, polymers (binding agents) are purchased and stored in metal drums. As they are normally nonhazardous, no special precautions other than fire prevention are required. They should be temperature conditioned before use to prevent moisture condensation. Moisture in the polymers often has a degrading effect on the propellants made from them. Manufacturing processes usually are designed to eliminate as much water as possible from the polymers.

3-6.1.8 Facilities and Equipment.

a. Facilities utilized for processing aluminum or other metal powders should be designed to provide the maximum vent area consistent with structural integrity.

b. Metal powder processing facilities should be equipped with conductive floors. All electrically conductive equipment should be bonded and grounded.

c. The humidity in facilities used for metal powders processing should be controlled. Generally between 50 and 55 percent relative humidity is recommended.

d. Local exhaust systems should be employed where necessary to preclude powder suspension or accumulation. High-volume, low-velocity exhaust systems are preferred. High-velocity movement of metal powders may create an electrostatic hazard within the system.

e. Belt drivers to power equipment should be avoided. Where required, belts should be conductive and/or provided with electrostatic dissipators. Sealed anti-friction bearings are recommended.

f. Vacuum collectors should be located outside *the process facility.*

3-6.1.9 Fuel Preparation

a. Because of the susceptibility of metal powders to ignition from electrostatic discharge, all dumping, screening, weighing, and handling equipment should be electrically bonded and grounded. Personnel engaged in metal powder operations should wear nonsparking conductive safety shoes and cotton underwear.

b. Spatulas, scoops and other tools used to dump, measure or stir metal powders should be grounded.

c. All possible measures should be taken to prevent moisture in metal powder operations. Waste metal powders should be immersed in a compatible oil until destroyed.

d. Vacuum cleaning of dust accumulations is preferred. Soft brooms or brushes may be used safely except where dust brushed from ledges may become suspended in air. Blowing down dust with compressed air may create dangerous dust conditions.

e. The introduction of an inert gas into process equipment to reduce the oxygen content has proved effective in prevention of dust explosions. This method is recommended where applicable.

f. Where possible, metal powders should be incorporated with binder systems prior to mixing with oxidizing agents. Since oxidizing agents are normally dry when added to mixers, a dust environment is present. To premix metal powder and binder systems and add to the mixer as a slurry precludes an oxidizer and metal powder dust environment in the mixer.

g. Since premixing creates a dust hazard in the premix container, provisions for blade-to-bowl clearances and preventive maintenance to insure safe premix performance are necessary.

h. All premix ingredients should be screened before premixing. If screening is not possible because of the physical characteristics of some of the ingredients, then X-ray or other techniques should be used to insure that foreign objects do not enter the premixer.

i. Objects such as jewelry, pens, pencils, rings and other items that may accidentally be introduced into mixers should not be permitted into fuel preparation facilities. All tools should be accounted for by use of tool check lists or shadow boards.

j. Recently, beryllium has been used as a fuel in composite propellants. The normal precautions used to prevent ignition of aluminum powder are effective where processing beryllium. However, beryllium is considered toxic. The threshold values now recognized by responsible authorities are extremely low. The processing of beryllium powder requires rigid industrial hygiene controls. A complete study of this problem in conjunction with local, state and federal health agencies should be conducted before its use is employed.

3-6.1.10 Burning Rate Catalysts. Burning rate catalysts (and accelerators) should be carefully examined to determine compatibility with other propellant ingredients. Certain catalysts sublime or vaporize at process temperatures and may present problems if allowed to mix with other unincorporated ingredients in a mixer environment. Other burning rate catalysts are known to increase the sensitivity of composite propellants to ignition from friction and other ignition potentials.

3-6.1.11 Curing Agents. Curing agents should be carefully examined to determine compatibility with other mix ingredients. MAPO (Methyl Aziridinyl Phosphine Oxide) is known to react with ammonium perchlorate. This reaction has resulted in propellant fires. MAPO has been safely used in propellant systems with AP where the MAPO was first incorporated in the fuel binder premix. Dilution ratios should be carefully examined when this method is used. MAPO is toxic by ingestion, inhalation and absorption. It is also an irritant. The hazardous (toxic) characteristics of MAPO are increased at elevated temperatures. Appropriate respirators, chemical goggles and non-absorbent gloves should be worn by personnel engaged in operations involving MAPO.

3-6.2 MIXING IN GENERAL. The proportioning and mixing of propellant ingredients are the most important steps in the solid propellant manufacturing process. Safe and efficient production of a high quality composite propellant demands that raw materials be proportioned precisely and mixed completely. The two operations are equal in importance. Several mixing techniques are now employed. Two such systems are discussed below.

3-6.2.1 Batch Mixing. Batch mixing has been the standard method for castable composite processing for years. Recently, sophisticated automatic weighing has led to the automated batch mixing system. Automated batch mixing provides integrated propellant batching. The ABM system is an addition to the current horizontal mixing process system which utilizes highly accurate digital output force measuring devices, modern materials handling techniques, and fully interlocked control circuitry. The proportioning and addition of all materials to mixing is controlled from a remote control station.

a. Both horizontal and vertical batch mixers currently are in common use in the industry. Either of these batch mixers can be modified for use with automated proportioning systems.

b. Horizontal mixers with either sigma blades or modified sigma (multiwing overlap) blades have been used successfully to process hundreds of millions of pounds of propellant at many solid propellant manufacturing facilities. The submerged glands on these mixer blade shafts are a potential hazard; however, studies have shown that exceptionally safe packing glands can be developed. The use of braided cotton as a packing material has proved satisfactory. However, other materials are in use and are considered to be as safe, economical and effective as cotton. Jute and braided asbestos are two such materials. Packing glands (housing) may be modified to incorporate liquid purge and temperature monitoring systems. A liquid polymer, or other compatible lubricant, may be used as a purge medium.

c. Mixer blade-to-blade and blade-to-bowl clearances have been studied. Larger blade clearance reduces attrition of coarse oxidizer and increases the safety margin in operating the mixers.

d. More recently, vertical mixers have become popular for solid propellant blending. The two primary types of vertical propellant mixers are the two blade vertical planetary type (typically manufactured by Baker-Perkins Co.), and the three blade helical planetary vertical mixer (typically manufactured by the J. H. Day Co.). Some of the reasons for the use of vertical mixers are elimination of submerged packing glands, increased clearances of blade-to-bowl, removable bowls that allow thorough cleaning and inspection, shorter mixing times, and less maintenance.

3-6.2.2 Continuous Mixing. Several years ago, continuous propellant mixing was under active investigation by many different companies. Since that time, this concept has been subjected to extensive investigation since it offers certain advantages over the batch mixing process. In addition, new, exotic propellants, which may be quick curing, pyrophoric, or toxic, perhaps could be processed efficiently and safely since the propellant remains in the mixer for only a short time and the entire process is operated remotely. Indeed, the most significant safety advantage of the continuous mixer is the minimal amount of material susceptible to ignition during mixing operations. When compared with the amount of material in the conventional mixer, this amount is relatively small. The most perplexing problem associated with the continuous mixer has been the effective elimination of the inadvertent introduction of foreign materials into the mixer housing.

3-6.2.3 Facilities and Equipment.

a. Mixer facilities should be designed to provide the maximum vent space consistent with the structural integrity of the facility. Lightweight construction is preferred. The use of wood or other porous organic materials in the construction of mixer facilities should be minimized. When the use of these materials is required, they should be coated with an impervious flame retardant paint.

b. Copper or copper alloys may form highly sensitive compounds when exposed to oxidizers such as ammonium perchlorate. They should not be used in mixer facilities.

c. Mixer facilities should be designed so that operating personnel will have unblocked escape routes at all times. Escape chutes to the outside should be provided from work platforms.

d. Quick-acting detection and delivery deluge systems should be included in the facilities to cover points of operation. These systems should be interlocked with the control circuits to the mixer to prevent operation of the mixer when the deluge system is inoperative.

e. Where possible, propellant mixers should be provided with a lightweight cover to prevent materials from dropping into the mixer. Care should be exercised in the design of the cover to allow adequate venting in the event of an ignition in the mixer.

f. Vertical mixers have proved difficult to vent adequately. Some success has been achieved by installing vent ports in the free board area above the mix level. Another method has been to design the hydraulic lift cylinders such that they relieve to "drop" the mixer bowl when force is exerted (pressure inside the bowl).

g. The gear housing of vertical mixers should be sealed or purged with inert gas during mixing operations to prevent contamination by dust from the mixer bowl.

h. Blades and other moving parts of new mixers and replacement parts for mixers should be nondestructively tested for cracks, crevices and other imperfections before being put into operation. Provisions should be made for periodic inspections required thereafter.

i. Nuts, bolts, and other hardware on mixers, monorail systems or in other locations that could loosen and fall into a mixer should be effectively secured.

3-6.2.4 Mixing Operations.

a. When possible, all materials should be introduced into the mixer remotely. The mixing cycles should be completely remote controlled.

Materials should be screened directly into the mixer. Where the physical characteristics of the material preclude screening, other methods such as magnetic separators or nondestructive testing should be employed to segregate foreign material and preclude their entry into the mixer. Where toxic or flammable vapors are involved, exhaust systems should be incorporated.

b. Oxidizing material should be introduced into mixers after fuel-binder compositions. It has been demonstrated that materials in the high oxidizer to fuel ratios, experienced when oxidizers are added prior to fuels, may undergo deflagration to detonation where ignition occurs. The time phase wherein this type of reaction can take place is shorter when fuels are added to the mixer first and oxidizers are added while the mixer is running.

c. Clearances between blades and mixer bowls should be established as the maximum clearance consistent with quality and process requirements. Blade clearances should provide for deflection of shafts and wear in journal and bearing areas. Blade clearance should be checked at sufficient intervals to assure a continuing condition of adequate clearances. The openings in screens (measured diagonally) should always be smaller than the blade-to-bowl clearances.

d. Special considerations should be given to the packing gland areas (shaft to mixer bowl juncture) of horizontal mixers. Frequent inspections and changing of packing gland materials should be accomplished to preclude a build up of oxidizer and fuel in those areas. Packing materials and lubricants should be compatible with the propellants being mixed. Thermocouples or other heat sensing devices should be connected to a recorder to alert operating personnel of any significant increase in heat in that area. Temperature readings of the propellant mass should also be recorded to warn against heat increase due to exothermic reaction.

e. Objects such as jewelry, pens, pencils, rings, and other items that may accidentally be introduced into mixers should not be permitted in mixer facilities. All tools should be accounted for by use of tool check lists or shadow boards. Shadow boards and tool boxes or racks should be located in a separate location away from the mixing room or facility.

f. Only non-sparking spatulas and devices should be employed to scrape down mixer sidewalls and blades. Friction and impact sensitivities of the propellants should be considered when determining such tools.

g. Vertical mixer bowls used as casting cans or transport hoppers should be nondestructively tested at frequent intervals to detect possible damage of weldments between the bowl and bowl jacket. Diameter and circumferential measurements should be made to determine if warping has resulted from handling.

h. Spilling or splashing of propellant during discharge of mixers should be avoided. Operators shall be so positioned during discharge operations that direct, unblocked routes of rapid exit will exist for emergency use.

3-6.3 CASTING AND CURING. The three most common methods of casting are bayonet casting, in which the propellant is introduced into the top of the motor through hoses; bottom casting, in which the propellant is forced by pressure up through an opening in the bottom of the rocket case; and vacuum casting, in which the propellant is passed through a slit plate into a rocket motor case enclosed in a vacuum bell. High strength cases are also used as vacuum bells.

3-6.3.1 Bayonet Casting. Bayonet casting has been used to load rocket motors for many years. This method requires equipment to either lower the motor or to remove the bayonets as the propellant level rises. This method also requires a pressure vessel to push propellant, using air or inert gas, through the bayonets. This process is relatively inexpensive and, although it allows introduction of more voids in the grain than vacuum casting, it also eliminates the necessity of expensive vacuum bells and related tooling. However, safety considerations are more complex and propellant losses with this process are higher than with other casting techniques. Bayonet casting introduces an additional hazard inasmuch as the casting tubing and accessories are contaminated. The removal of bayonets and connecting and disconnecting casting tubing from pressure vessels (casting cans) must be accomplished with extreme care to avoid friction or impact of contaminated surfaces.

The use of casting cans introduces the problem of working with pressure vessels. The integrity of mating detachable lids and vessels requires extensive testing and preventive maintenance. Pressures of up to 150 psia may be employed during bayonet casting operations.

3-6.3.2 Bottom Casting. This method of loading propellant into motors also is one of the earliest, but still advantageous with certain motor designs. The bottom casting process indicates only that the propellant will be pushed up into the motor from the bottom usually by a bayonet or piping connection between a pressure casting can, an impeller pump, and the motor.

Bottom casting is particularly advantageous with motor designs which do not allow adequate clearance from the top to drop in vacuum cast propellants or to accept a bayonet. However, the introduction of an impeller pump to move the propellant creates an additional ignition media not experienced in bayonet or vacuum casting.

3-6.3.3 Vacuum Casting. Casting the undeaerated propellant into a motor under vacuum is an extremely effective process. Since the deaeration is part of the loading, handling of propellant loaded transfer containers is minimized. Propellant impingement considerations are minimized because the only driving forces enabling the propellant to flow into the vacuum chamber through the narrow slits are gravity and the differential pressure, which, of course, cannot exceed atmospheric pressure. Vacuum casting has the advantage also of eliminating deaeration as a separate, time consuming step after mixing. Comparative data clearly have established the improvement in safety (i.e., reduction of exposure) of vacuum cast propellant. This process is the most widely used today.

3-6.3.4 Casting Facilities. Casting facilities should be constructed of lightweight materials and should be designed to provide the maximum vent area consistent with the structural integrity of the facility. They should be designed so that operating personnel will have unblocked escape routes at all times. Escape chutes to the outside should be provided from work platforms.

3-6.3.5 Casting Equipment.

a. All casting vessel assemblies should be designed to preclude internal cracks, crevices, corners, pockets, and any internal configurations which could subject propellant to initiation from impact, friction, or compression.

b. Lids should be secured to pressurized casting vessels in a manner which will withstand the rated pressures of the vessels. Frequest tests and inspections should be made to assure that the clamp or assembly device is functioning properly.

c. Pressurized casting vessels should be capable of withstanding at least twice the maximum allowable working pressure to which the can will be subjected. Periodic hydrostatic tests (1-1/2 times the working pressure) of such casting vessels should be performed at maximum intervals of six months. At five-year intervals, vessels should be hydrostatically tested at twice the working pressure. Hydrostatic tests (at twice the working pressure) should be made after any alteration, report of incident of abuse, mishandling, or dropping. A log of tests performed should be maintained for each vessel.

d. A blowout disk, designed to blowout at 120 percent of the vessel's maximum allowable working pressure, should be provided.

e. The internal welded seams should be inspected frequently by approved methods of weld examination.

f. Lifting fixtures designed as an integral part of the casting vessel should be proof load tested prior to use with a load equal to 111 percent of the total weight of the vessel, all attachments and equipment, and the maximum pounds of propellant to be handled. A regular program of load test and inspection should be performed and records of such tests and inspections maintained.

g. Casting vessel support fixtures, such as legs, should be attached in such a manner that routine handling will not cause damage to the casting vessel internal surface. They should be proof tested initially at 111 percent of the total weight to be supported and periodic tests and inspections conducted thereafter.

h. Vibration equipment attachments should be designed to prevent frictional heat generation on the vessel structure which contains propellant.

i. Threaded connections should not be permitted in piping, valve connection, or any part of the casting vessel charging or discharging system. Casting piping and manifold systems should not be joined with threaded connections. They should be pressure tested at 1-1/2 times the maximum casting pressures. Methods of attachment for casting piping and manifold joints should provide positive fastening to eliminate failure of mating parts when pressurized.

j. Valves through which uncured propellant flows should be designed to prevent compression of propellant between two metal surfaces (e.g., rubber diaphragm-type valves). These valves should be cleaned and inspected at regular intervals suitable to the process and a log of these inspections should be maintained. A positive system to prevent propellant flow should be incorporated into the casting system which will stop the flow should the primary valve fail. Valve attachment to the casting vessel should be equipped with flanged mating surfaces and secured by positive methods. The use of "quick release" type fittings should not be permitted.

k. Vacuum casting bells should be designed to withstand the conditions under which casting is accomplished.

l. Deaeration assemblies should be designed to prevent pinching or impacting propellant and for ease of assembly and disassembly.

3-6.3.6 Casting Operations.

a. Pressurization of casting vessels should be performed remotely. Line pressure for pressurizing the casting vessel should not exceed the working pressure of the vessel. Filters should be installed in air lines to remove water and oil. Use of safety links is recommended for pressurized casting vessels to restrain the casting vessel lid should the attachment device fail when pressurized.

b. Casting vessel handling methods and equipment should be designed and operated in accordance with approved standards. The vessels should be restrained to prevent movement should a rupture of the relief valve occur.

c. Mechanical insertion of cores in motors containing propellant should be accomplished remotely or personnel should be protected by adequate shields or barricades. When cores are to be inserted mechanically, the equipment should be designed to prevent metal-to-metal contact between the core and motor case below the propellant surface.

d. Loaded motor cases or casting molds should be secured during casting and handled in a manner which will prevent overturning or spillage of propellant. Casting cores should be secured to prevent movement when loaded motors are transported in any manner.

3-6.4 CURING FACILITIES AND EQUIPMENT.

a. Curing facilities should be constructed of lightweight materials and should be designed to provide the maximum vent area consistent with the structural integrity of the facility.

b. The safe temperature for curing the propellant should be established and dual heat controls should be installed to prevent that temperature from being exceeded.

c. Heating units or elements should be designed to eliminate any direct contact between the heating unit or element and the propellant or rocket motor.

d. Mold supports and other casting and curing fixtures should be designed to avoid rubbing or pinching of thin layers of propellant between metal surfaces.

e. Means of pressure relief should be provided on closed pressurized vessels into which motors are placed for curing.

3-6.5 CURING OPERATIONS.

a. Loaded motor cases or casting molds should be handled or secured so that overturning will be prevented.

b. Loaded or partially loaded rocket motors should be raised or suspended at minimum distances above floor level. If tests or experience indicate that rocket motors may ignite upon dropping, protective measures should be employed to minimize the possibility of such ignition.

c. Core popping (initial release or separation of case from propellant) should be done remotely or from a shielded location. When the core has been "released", final extraction of the core may be accomplished attendantly. When cores are not designed with a taper (smaller dimension at forward end), extraction of the core from the motor cavity should be done remotely or from a shielded position.

d. Numerous fires have occurred during curing operations involving castable composite propellants. A significant number of these incidents has been related to either mechanical failure of oven control devices or exothermic reaction of propellant formulations. Complete characterization of formulations should be conducted to establish safe curing temperatures. The probability of exothermic reaction should be identified. Design considerations should include positive temperature controls. Steam is the most common source of heat used in curing castable composite propellants.

3-6.6 MACHINING AND TRIMMING FACILITIES AND EQUIPMENT.

a. Machining and trimming facilities should be constructed of lightweight materials and designed to provide the maximum vent area consistent with the structural integrity of the facility.

b. Rocket motors should be secured to prevent movement should an ignition occur during machining operations.

c. Machining equipment should be designed to prevent contact with motor cases and other metal objects. Design should incorporate features for the removal of dust and chips.

d. Gasketing and sealing materials such as Teflon should be provided between shafts and bushings to provide a low coefficient of friction.

e. Compatible lubricants should be used to lubricate moving parts of machines.

f. Maximum vent area should be provided in the cutter (machines) housing to allow venting in event of a fire.

g. Positive methods should be incorporated to preclude loosening of bolts and screws exposed to the machining process.

h. Consideration should be given to quick acting deluge systems for machining operations. Such systems have been designed and have proved feasible.

3-6.7 MACHINING AND TRIMMING OPERATIONS.

a. Mechanical cutting, contouring or trimming operations of case bonded composite propellants should be done remotely or from a shielded location. Where possible, monitoring by TV should be provided.

b. The rate of feed and cutting speed of the tool should be consistent with the physical characteristics of the propellant. Feed rates and speeds should be controlled so as to preclude heat build up.

c. Hand trimming or cutting should be discouraged. Additional shielding and personal protective clothing should be provided.

d. Frequent and complete cleaning of machining equipment exposed to propellant or propellant dust should be accomplished.

e. Propellant dust and shavings should be immediately immersed in a compatible wetting agent.

3-7 FINISHING MOTORS AND TEST SAMPLES

3-7.1 PRINCIPAL HAZARDS. The principal source of hazard during the machining and trimming of cured propellant is the possibility of metal-to-metal contact within the tool or machine itself or between the tool and metal mold or chamber containing the propellant. Such contact may result in friction that ultimately produces highly concentrated heat or sparking. Exposed propellant surfaces are then subjected to fire danger. With a semi-confined configuration, the burning propellant gases may stream and result in a directed flame.

a. Spark ignition of most conventional cured propellants is unlikely, but finely divided and suspended propellant dust, chips, and shavings are sensitive to spark ignition or electrostatic discharge. Pyrophoric materials, such as many igniter ingredients, as well as vapors and other fumes, are especially susceptible to spark ignition. In these cases, the possibility of explosion and fire exists. Dry air promotes such hazards.

b. The confinement of propellant in a mold or a chamber aggravates the fire hazard and can provide a condition for explosion, with the subsequent danger of fragmentation and further spreading of the fire.

c. Propellant pinched or squeezed within a small area by tooling also presents the situation of containment and heat generation, which will act as an ignition source for the propagation of flame under favorable conditions. Clean-up operations after machining and trimming are less hazardous because less propellant is exposed. However, the danger from trapped propellant or scrap propellant is still present, especially in hardware or tool configurations that permit such entrapment or collection.

3-7.2 CHARACTERIZATION OF CURED PROPELLANT.

3-7.2.1 Explosive Classification. It is vital to the consideration of hazards in machining and trimming to understand the hazards of the propellant. Basic characterizations, such as whether the explosive hazard classification is one of detonation or deflagration, should be a guide to whether a particular operation must be remote. The explosive hazard classification also governs the quantity limits allowable for the operation. Compatibility with all materials with which the propellant will come into contact during operations must be determined. Other sensitivity and hazards characterizations, including spark testing and determination of sensitivity to impact, auto-ignition and sliding friction, must be completed before machining and trimming larger samples of propellant can be done safely.

3-7.2.2 Confinement. The degree and nature of the confinement will also determine the extent of caution. Light, flightweight, thin-walled chambers present less danger of fragmentation and flying missiles than heavy-walled chambers or thick-walled specimen molds.

3-7.2.3 Toxicity. For certain propellants, toxicity characterization is also required, and hazard precautions are accordingly observed in trimming and machining operations. Mechanical and physical properties must be characterized so that operational and safety considerations can be allowed for.

3-7.3 MOTOR RESTRAINT.

a. Operations on a loaded motor should always be done with the motor secured in fixtures designed to retain the motor if it should be accidentally ignited. The initiation hazard can be reduced by not having installed the initiator, the ignition system (including any safe/arm device), or the nozzle prior to trimming or machining operations. The design load for the tie-down fixture should be:

(1) Four times the nominal thrust applied axially forward or aft

(2) Two times the nominal thrust applied in any direction in the transverse plane at any retainer. Resulting stresses should not exceed ultimate material allowables

b. For operating flexibility, it is desirable to have an adaptor which is permanently attached to the motor during final processing and assembly and which is, in turn, tied down to the floor or to some fixture.

c. A mold containing propellant specimens, although not likely to be propulsive if ignited, should also be restrained.

3-7.4 DUST AND CHIP REMOVAL.

3-7.4.1 Vacuum System. The generally accepted method for the removal of propellant dust, chips, and scrap during machining and trimming is to pick them up with a vacuum pump. The pump and collector, being hazardous themselves, should be located in a properly barricaded area or cubicle separate from the machinery being served by the vacuum system. The pump should be of ample capacity and be explosion proof and nonsparking. The collector should be of the "wet" type, using water unless it is incompatible with the propellant being collected. No mixing of types of propellant must be permitted in the collector, and frequent and periodic cleaning procedures must be observed. If collection is dry, precautions against hazard from electrostatic ignition must be observed. Adequate grounding must be provided.

a. The vacuum hose should be located as close to the source of the dust and chips as possible. The hose should be flexible and flanged to allow ease of cleaning and to prevent the excessive buildup of pressure in the event of an explosion. No threaded connections are allowed. The interconnection of manually operated hose to the machinery should be avoided.

b. It is desirable to have safety lock-outs such that the machinery for trimming or sawing cannot be operated without the operation of the vacuum system. Conversely, it should not be possible to turn off the vacuum system without the knowledge of the operator performing the machining.

3-7.4.2 Scrap Storage. Only the minimum amount of scrap should be allowed to accumulate from propellant-trimming operations where it is not collected by a vacuum system. The scrap box, which should be outside the operational area, should not contain mixtures of combustible material, specifically items like oily rags or Kimwipes. The container should be metallic, properly marked and painted, and protected from sources of heat, including sunlight. Some scrap propellants must be wetted, and the compatibility of the propellant with the wetting agent must be checked. Scrap pyrotechnic, tracer, flare, and similar mixtures should be collected in number 10 mineral oil.

3-7.5 ENVIRONMENT AND GENERAL PRACTICE.

3-7.5.1 Tool Design. Where propellant may come into contact, all metal-to-metal surfaces shall be avoided. There must be no threads, cracks, crevices, or blind holes in metal parts that may contact the propellant during casting or finishing operations. Teflon-coating metal surfaces such as molds and mandrels will generally prevent the propellant from adhering. Surface roughness, including that of welded surfaces, shall not exceed 125 RHR.

a. Recommended materials for hand tools include the following: aluminum, oil-resistant Neoprene, beryllium-copper alloy, AISI 300 stainless steel, and tempered steel (for cutting tools such as X-acto knives). Steel cutting tools shall not be allowed to cut through the propellant to the metal. For example, during small sample cutting, the cutting board should be covered with a Teflon sheet.

b. Tool design or selection shall incorporate all safety considerations. Design points include tool speed control, use of nonsparking metals in contact with or close proximity to the propellant, provision of limit switches to prevent over-ranging of tool and possible metal-to-metal contact, explosion proofing of machinery, use of effective coolant where necessary, effective dust and chip removal, ease of cleaning, and provision for adequate grounding. The deluge system should provide for the quenching water to be directed at the area where the cutting or machining is occurring, since it is the likely source for the spread of flame.

3-7.5.2 Manual Operations. Manual operations are allowed only when the propellant is well characterized and when adequate experience with the specific operation is on hand to permit a complete description of the operation in a standard operating procedure.

a. Safety goggles shall be worn. Additional shielding shall be provided where feasible. Whenever possible, the operator shall not face the hazardous source directly. Fire-retardant clothes shall be worn, avoiding any type of encumbrances. Protective gloves shall be used when they do not hinder the manual operation.

b. Tooling should be designed so that cutting pressure can be applied to the tool with only one hand. For certain operations, it may be necessary to use the other hand to assist in guiding the tool to regulate the depth of cut, prevent pinching of the propellant, or avoid metal-to-metal contact.

c. Such tools as specifically designed cutters can be used for manual trimming, although final trimming may be more safely done by hand when the operation is closer to metal walls.

d. Parts shall never be forced to fit, as this may indicate that propellant has worked in between mating parts. Threads and bolt holes should be protected against propellant contamination by such measures as covering with masking tape when operations on them are not being done.

e. Where the hazard from static electricity discharge is high, as when propellant dust or igniter mixtures are present, personnel as well as equipment shall be properly grounded.

f. For highly hazardous manual operations such as hand trimming of motors, a second, standby operator shall be present or very available in the event of an accident. The second operator can also be used to verify the adequacy or completion of a safety measure prescribed in the standard operating procedure.

3-7.5.3 Remotely Controlled Operations.

a. For extremely hazardous propellants and/or configurations of motors, remotely controlled operations using power-driven equipment shall be required. Standard operating procedures with appropriate checklists shall be provided.

b. The safety warning system provided by human operators must be replaced by less discriminating automatic equipment. Television monitors should be provided for viewing potential trouble spots. Direct line-of-sight viewing shall not be allowed. The sound of the operation should also be monitored to catch unusual sounds that might forewarn of difficulty.

c. Normal considerations for dust and chip removal, desirable design features, observance of quantity-distance limits allowable, and proper grounding shall be maintained. Clean-up shall include thorough inspection and cleaning of areas that have accumulated dust or scrap, such as wheels, rollers, or guiding rails, as well as the general working area.

d. More than one operation at one time in one protected location is not to be allowed. Idle machinery should be properly protected from dust and chips during operations.

3-7.5.4 Facility and Storage.

a. Machining and trimming are hazardous operations, to be undertaken only with the complete restriction of personnel to allowed limits.

b. Propellant grains that are not case-bonded should be machined and trimmed to the extent necessary before they are cartridge-loaded into motors. Proper facilities for the grounding of equipment and personnel shall be provided where static electricity discharge is a high hazard. This includes provision for discharge from the person before entering the hazardous area, as well as provision for preventing charge build-up during operations.

c. Deluge systems shall be provided to minimize damage to equipment and to minimize the severity or spreading of accidental fires. General good housekeeping shall be observed. No unneeded or unrelated equipment shall be in the operating area. If feasible, no more than one charge of propellant should be in the operating area. Specimens and samples shall be restricted to reasonable amounts. Scrap shall not be allowed to accumulate excessively.

3-7.6 PREPARATION OF TEST SAMPLES.

3-7.6.1 Small Test Samples. The preparation of very small samples of propellant for such tests as density checks, calorimetric tests, impact, tensile or strand burning should follow general procedures of safe practice described in preceding paragraphs, relating to design, handling, cleaning of molts, hand-cutting pieces of propellant, and storage.

3-7.6.2 Milled Samples. The milling operation is usually employed to prepare mechanical property specimens. The general discussions on characterization of the propellant (3-7.2) and dust and chip removal (3-7.4) are applicable. Tool design and selection (3-7.5.1) should govern the milling machine. Spindle speed and table feed speed should be optimized for safety and efficiency for the particular propellant being operated on. Unless the propellant is well characterized and experience has been built up in milling it, milling operations in general should be accomplished remotely.

3-7.7 MACHINING.

3-7.7.1 Sawing. Sawing operations should always be done remotely. Paragraphs 3-7.2, 3-7.4, 3-7.5.1 and 3-7.5.2 are applicable. The fall-and-feed control should be optimized for safety and cleanliness of cut. Blades should be Teflon-coated. Extremely hazardous propellant may be sawed while immersed in a coolant. Otherwise, adequate coolant must be provided near the cutting operation. Continuous cleaning of the blade is desirable, possibly with a set of brushes with air attachment.

3-7.7.2 Turning. Lathe operations should always be done remotely. Paragraphs 3-7.2, 3-7.4, 3-7.5.1 and 3-7.5.2 are applicable. The lathe may be fitted for horizontal drilling. Exposed guide rails, especially those involving screwed rails, must be watched for collection of propellant dust and scrap.

3-7.8 TRIMMING OF CASE-BONDED CHARGES.

3-7.8.1 Manual Operations. Final trimming, chamfering and facing is probably safer if done manually. Paragraph 3-7.5.2 applies.

3-7.8.2 Remote Operations. For large case-bonded motors, trimming to finish a grain or cutting to produce the desired configuration, especially for "production" items, should be done remotely. Residual dust in the motor should be carefully removed by a vacuum system. Paragraphs 3-7.5.1 and 3-7.5.3 apply.

3-7.9 MOTOR ASSEMBLY. Assembly of motors, igniters, initiators, nozzles, etc., are discussed in detail in Chapter 5.

3-8 NITROGLYCERIN MANUFACTURE

3-8.1 INTRODUCTION. In certain propellant manufacturing facilities, nitroglycerin is made in the plant instead of procuring the desensitized raw material. An objective overview of the general safety instructions which applies to the Biazzi continuous process for the manufacture of R-nitroglycerin is given here. Detailed step-by-step safety instructions, which may be found in the Standard Operating Procedures for the installed plant, are not given.

3-8.2 PERSONNEL SELECTION. It is imperative that personnel used in nitroglycerin manufacturing operations be carefully selected. Selection criteria should consist principally of emotional stability and the ability to think clearly and react logically under emergency circumstances. Educational requirements are not of primary consideration. Personnel used in the nitroglycerin manufacturing line must display at all times an acute consciousness of the hazards inherent to the process and a constant surveillance must be made of the safety attitude and habits of these people to assure that familiarity with the process does not breed complacency. Mental alertness, job interest, job contentment, even temperment and ready acceptance of instruction are attributes which mark a potentially satisfactory nitroglycerin worker.

3-8.3 PLANT LAYOUT AND MATERIALS OF CONSTRUCTION. The cardinal principles found in 3-1 are stressed in the design and layout of a nitroglycerin plant. Extensive use is made of fail-safe remote operations and the employees are exposed to the smallest practical quantity of material for the shortest possible length of time.

The type of construction ordinarily used in commercial nitroglycerin manufacturing buildings is similar to that used in smokeless powder manufacturing construction. The quantity-distance tables cited in the AMC Safety Manual for Class 7 explosives are applicable to nitroglycerin; however, traditionally the unbarricaded distances are used for barricaded structures. This practice has been arbitrarily established and it is not meant to be set forth in this document as a firm regulation; however, this option should be considered when new construction of nitroglycerin facilities is planned. Because of the liquid nature of the raw materials and finished product, gravity transportation methods are generally employed between operating buildings. The floors of manufacturing buildings are generally lead-covered, and provided with either a rim or gutter around the outside perimeter to facilitate cleanup or to divert spilled material into outside catch tanks. Connecting conduits between operating buildings are usually provided with an interrupter station to provide discontinuity and to prevent explosion propagation between buildings. The processing equipment in the Biazzi system is constructed of stainless steel material; batch process equipment has traditionally been constructed from lead material, but the current trend is to use stainless steel for much nitroglycerin equipment. In the past, storage tanks for the finished product have been, almost invariably, constructed from lead. However, the current trend in selection of materials is toward stainless steel. Inter-connecting lines are generally fabricated from gum rubber hose or approved plastic materials. Valving is generally accomplished by use of pinch clamps, or specially designed ceramic block cocks. Extreme care must be exercised in the fabrication of processing vessels and inter-connecting conduit to avoid low points or pockets which do not have free drainage, wherein nitroglycerin might stagnate and possibly decompose. The interior of processing equipment is carefully inspected, prior to use, to ascertain that no pits or voids exist which might trap nitroglycerin. Threaded fittings are not used at any point where they would be exposed to the material. Rigid specifications for weldments are required in any piece of nitroglycerin equipment where fabrication requires welding techniques. Also, the building, equipment, and conduit arrangement is planned to prevent exposure of nitroglycerin to rays of direct sunlight. All major operating buildings are provided with catch tanks to capture and recover nitroglycerin spilled or washed from floors in these buildings. These catch tanks are housed separately outside the operating buildings with gutters connecting them to the buildings.

3-8.4 MANUFACTURING FLOW DESCRIPTION. R-nitroglycerin is manufactured by the nitration of glycerin with a composition of mixed sulphuric-nitric acids. After nitration, the nitroglycerin is separated from the spent acid by stratification, washed with soda water and then with water to neutralize residual acid, and it is finally stored under water containing dissolved soda ash. In the Biazzi process, the operations are carried on in a continuous fashion in stainless steel vessels of relatively small volume. The process is provided with a security system which monitors process temperatures and sets off visual and audible alarms when abnormal levels are reached in the critical areas. The nitrator and separator are equipped with dial thermometers having electrical contacts which close at 90°F, thereby automatically discharging the contents of these vessels into a drowning tank containing a large quantity of water. The security system in the Biazzi unit provides dependable protection but additional instruments are used as checks and balances to make it difficult for personnel to attempt to operate the unit in an unsafe manner without warning. Therefore, it is imperative that this security system be maintained in good working order at all times and periodically checked for functional operation.

3-8.5 PROCESS CONTROL. Nitroglycerin should not be allowed to remain in an acidic state, i.e., in a pH region below 6.8 longer than necessary for processing purposes. Of necessity, it is in this sour state during the nitration and separation steps. However, it is neutralized and brought into the pH region of 6.8 to 7.2 as soon as possible thereafter. During storage, pH of nitroglycerin is checked daily.

a. Nitroglycerin freezes at approximately 54°F and care must be taken to prevent it from freezing during manufacture and storage. While nitroglycerin is less shock-sensitive in the frozen state, it is more sensitive during the transition period from the solid to liquid state experienced during thawing. Frozen nitroglycerin can be gradually thawed by use of warm water; however, the possibility of freezing is avoided as much as possible and seldom occurs in well supervised manufacturing processes.

b. The decomposition of nitroglycerin is accelerated by high temperatures and therefore processing temperatures are carefully controlled, especially in the nitrating, separating, and washing operations. The nitrating reaction is exothermic and the heat liberated during this step of the operation must be removed by refrigerant techniques. The Biazzi process has provision for consistently monitoring the temperatures and producing visible and audible alarm in the nitrating, separating and washing operation should the temperature approach unsafe levels. The rapid decomposition of nitroglycerin is accompanied by a temperature rise and an evolution of red or brown fumes having a strong nitric acid odor. Should nitroglycerin reach this state of decomposition during manufacture, provision has been made to discharge the decomposing nitroglycerin into a drowning tank containing water and agitated with compressed air. This will also be done automatically if the operator should fail to detect the need to drown the charge.

c. Nitroglycerin has a lower specific gravity than the spent acid which is present after nitration and hence it is possible to separate the nitroglycerin from the acid by gravity techniques. This natural separation generally occurs in a short period of time after nitration; however, the separation is impeded by traces of certain foreign materials in the glycerin or acid; therefore, it is necessary to ascertain that these ingredients are free of foreign material prior to use. Poor separation not only causes inefficiency in the process, but can cause undesirable conditions in the subsequent neutralizing and washing operations. Nitroglycerin operations are normally closed down in an orderly manner prior to the occurrence of an electrical storm according to standard shutdown procedures. Auxiliary power and air sources are provided for nitroglycerin operations to protect against general utility failures.

3-8.6 DISPOSITION OF WASTE NITROGLYCERIN. Special filters are provided so that clean nitroglycerin, containing no solvents or impurities can be recovered and used from catch tanks, etc. Nitroglycerin which is collected from spills, catch tanks, etc., and which is not considered suitable for use, is disposed of by absorption in sawdust, wood pulp and fullers earth, after which it is carefully burned. In the event that nitroglycerin is spilled on the ground, the contaminated soil is carefully removed with low impact tools and is burned in the same manner as above. The burning is performed in a remote area, generally called a "Burning Ground", by special procedures and trained personnel. (Chapter 7)

Alkali sulfides are useful as nitroglycerin removers (destroyers-decomposers), and a sodium sulfide-water; methanol-acetone solution is commonly used for this purpose. The nitroglycerin is saponified by this material to insensitive compounds. Its use, however, is restricted to contact with minute quantities of nitroglycerin only since saponification reaction is exothermic. Equipment and devices which are to undergo maintenance repair are thoroughly cleaned, treated with NG remover, rewashed and then heated in an oven at 450°F for four hours. When feasible, this method of decontamination provides the greatest assurance of removing all traces of nitroglycerin from pockets, pores and other hidden areas. However, when this method is not practical, careful cleaning plus washing with nitroglycerin remover, followed later by a thorough water wash is an acceptable method of decontamination. Only freshly prepared solutions of remover are used, and because of the flammability of this material, only small amounts are stored in the nitroglycerin area. The nitroglycerin remover solution emits a very disagreeable, pungent odor and when it is used in enclosed areas, good ventilation should be provided. Personnel using it must wear goggles and impervious gloves.

3-8.7 ACID HANDLING. The nitroglycerin process utilizes a mixture of concentrated nitric and sulfuric acids for the nitration of glycerin and spent acid (which has a composition of nitric, sulfuric and water) is generated as a by-product. The usual safeguards for handling corrosive acids should be practiced, such as the use of rubber gloves, protective clothing, eye protection, emergency protective equipment (disaster suits and cartridge type and fresh air type gas masks), for the avoidance of inhalation of oxides of nitrogen and acid fumes, etc. Inasmuch as the spent acid contains small quantities of nitroglycerin, assurance that this nitroglycerin content is in solution with the acid should be maintained. This is usually accomplished by diluting the spent acid with 2 to 3 percent water to increase the solubility of nitroglycerin in the acid. Should free nitroglycerin be observed to be present in the spent acid during storage, removal and proper disposition of the free nitroglycerin should be made prior to processing the acid for recovery.

3-9 GLOSSARY OF TERMS

3-9.1 COMPOSITE PROPELLANTS. Composite propellants are formulations containing no nitrocellulose or nitroglycerin; are uncolloidal heterogeneous mixtures consisting generally of an organic fuel, an inorganic oxidizer, organic binding agents and curing agents.

3-9.2 CASTABLE. Having the physical characteristic of a semi-solid, pourable matter. Capable of being cast.

3-9.3 AUTOCATALYTIC. A process by which the catalyst may be produced as a by-product of the process. This by-product then catalyses and produces more catalyst which sustains and, in some cases, increases the catalytic action.

3-9.4 PURGING.

a. Introduction of an inert gas as agent to purge (cause removal) of potentially hazardous by-products of the process.

b. Introduction of an inert gas as agent to render the environment safer for the process.

3-9.5 HYGROSCOPIC. The characteristics of readily absorbing or retaining moisture.

3-9.6 BURNING RATE CATALYSTS. Substances introduced into composite propellant formulations to change the rate of burning.

3-9.7 CURING AGENTS. Substances introduced into composite propellant formulations to induce the conversion of the material from a semi-solid to a solid by causing a continued polymerization of the prepolymer.

3-9.8 SUBLIMATION. The direct passage of a substance from the solid state to the gaseous state and back into the solid form without at any time appearing in the liquid state.

3-9.9 VISCOSITY. The resistance to flow exhibited by a liquid (semi-solid) resulting from the combined efforts of cohesion and adhesion.

3-9.10 PASSIVATION. Chemical action or physical action to render a material inactive.

3-9.11 PYROPHORIC. Igniting spontaneously on exposure to air.

3-9.12 EXOTHERMIC REACTION. Chemical reaction which gives off heat.

3-9.13 DEAERATION. The removal of air.

3-9.14 ABBREVIATIONS

ABL	Allegany Ballistics Laboratory
ACGIH	American Congress of Governmental Industrial Hygienists
AMC	Army Material Command
BM	Bureau of Mines
DTA	Differential Thermal Analysis
EEL	Emergency Exposure Limit
ESD	Electrostatic Discharge
NOL	Naval Ordnance Laboratory
NOS	Naval Ordnance Station
PA	Picatinny Arsenal
TLV	Threshold Limit Value

CHAPTER 4

PACKAGING, STORAGE, SURVEILLANCE, AND TRANSPORTATION

4-1 INTRODUCTION

4-1.1 IMPORTANCE. Safe and efficient transportation is as inherently necessary to a missile system's reliability as any of its components. Regardless of the sophistication of a missile system and its associated components, and irrespective of the millions of man-hours expended in its design and manufacture, if a missile cannot be safely transported to its point of ultimate use without damage, then the cost of the missile is essentially wasted.

4-1.2 MATERIALS INCLUDED. The entry of guided missiles, heavy rockets, and spacecraft and their attendant problems in the field of transportation has caused considerable revision in the packaging, loading, blocking, and bracing, as well as the storage of hazardous materials. It would be noted that the regulatory bodies which govern the shipment of hazardous materials stipulate the manner in which these items will be packaged. The classification "Hazardous Materials" within the connotation of these instructions includes all dangerous articles listed in the commodity lists of the regulatory bodies from acetaldehyde to zirconium.

4-2 SCOPE AND RESPONSIBILITY

4-2.1 GUIDELINES. The guidelines to be followed in the shipment and storage of ammunition, explosives, and other dangerous articles are prescribed by the applicable regulatory agency.

4-2.2 COMPLIANCE. The manufacturer, developer, and/or shipper are required to comply with the regulations prescribed for the preparation for shipment, certification, and packaging for shipment of any article which is regulated. This requirement does not exclude articles made by, or for the U. S. Government.

4-2.3 EDUCATION OF PERSONNEL. Education of the packaging and shipping personnel of the manufacturer, developer, and shipper is the legal and moral responsibility of the firm or U. S. Government agency concerned.

4-2.4 APPLICABILITY. Regulations governing the transportation of ammunition, explosives, and other dangerous articles cited herein are binding upon all shippers of regulated commodities.

4-2.4.1 Department of Defense Contractors. Manufacturers developing propellants and/or rocket motors together with their ancillary components under a Department of Defense (DOD) contract or DOD subcontract must obtain the appropriate hazard (Department of Transportation) shipping nomenclature from the commodity manager's transportation element.

4-2.4.2 Independent Producers. Manufacturers developing propellants and/or rocket motors together with their ancillary components as an independent "in-house" effort should follow the provisions of paragraph 4-2.4.1; however, independent action with the Bureau of Explosives (AAR) may be accomplished. Notwithstanding, the data obtained by this independent action will not govern the hazard classification should the item at a later date become a DOD contractual item. In this case, the provisions of paragraph 4-2.4.1 will apply.

4-3 REGULATIONS AND AUTHORITY

4-3.1 FEDERAL STATUTES. The shipment and preparation for shipment of explosives and other dangerous articles are regulated by the Code of Federal Regulations (CFR), Title 49–Transportation. The shippers are governed by Part 173, rail freight handling is governed by Part 174, motor carriers are governed by Part 177, the specifications for packages are contained in Part 178, and rail tank cars are covered by Part 179. Formal recognition of the agencies and associations to assist the Department of Transportation (DOT) in the development of regulations for the safe shipment of explosives and other dangerous articles is contained in paragraph 834(e) of Chapter 39, United States Code, Title 18, as amended by Public Law 86-710, dated 6 September 1960.

4-3.2 JURISDICTION. Transportation by motor vehicle common and contract carrier came under the jurisdiction of the Interstate Commerce Commission in 1935 (Part 177, CFR, Title 49), and in turn under DOT in 1967.

4-3.3 IMPORTANCE OF SPECIFYING QUANTITY. The study of DOT regulations is enlightening and shows the advance made in the various industries; however, one problem continues to prove troublesome to anyone who tries to evaluate the hazard classification of devices that are generically called by the manufacturer as a "squib" or a "detonator". The utilization in the regulations of either the phrase "a small quantity" or "a small amount" tends to further confuse the person making the analysis. Conversely, fireworks quantities are stipulated in precise amounts, e.g., "not more than 10 grains of explosive composition" or "not to exceed twenty grams each in weight." Specific definition of quantity is of the utmost importance in the safe and correct hazard analysis of any compound.

4-3.4 AVAILABILITY OF SHIPPING REGULATIONS.

4-3.4.1 Air. The interstate and intrastate shipment of Ammunition, Explosives, and Other Dangerous Articles (AE&ODA) by commercial air is regulated by the Civil Aeronautics Board under CFR-Title 14, Transportation of Dangerous Articles and Magnetized Materials, Part 103, Sections 103.1 through 103.33. Penalties for violation are contained in USC Title 49, Part 9, Sections 901-904. Both are available from the Superintendent of Documents, Government Printing Office, Washington, D.C. 20402. Packaging, labeling, and certification are published in Tariff No. 6D-Official Air Transport Restricted Articles (ATB No. 37, CAB No. 82), issued by Airline Tariff Publishers, C. C. Squire, President and General Manager, 1825 K Street, N. W., Washington, D.C. 20006. Shipments of AE&ODA by military aircraft are regulated by Packaging and Handling of Dangerous Materials by Military

Aircraft, AFM71-4, DSAM4145.3, TM 38-250, MCO P4030.19. This document is available from the Superintendent of Documents.

4-3.4.2 Intrastate. Intrastate shipments are regulated by the respective states in which the shipments are made. Note: 38 of the 50 states use the DOT Regulations as written or amended.

4-3.4.3 Motor. The interstate shipment of AE&ODA by motor vehicle is governed by the DOT regulations for the Transportation of Explosives and Other Dangerous Articles. These regulations include packaging specifications and are contained in CFR Title 49-Transportation, Parts 171 to 190. This document is available from the Superintendent of Documents. These regulations are also published by F. G. Freund, Issuing Officer, 1616 P Street, N. W., Washington, D.C. 20036.

4-3.4.4 Rail. The interstate and intrastate shipment of AE&ODA by rail is likewise governed by the Department of Transportation's regulations for the transportation of Explosives and Other Dangerous Articles. These regulations include the packaging specifications and are contained in: "CFR, Title 49-Transporation, Parts 171 to 190." This document is available from the Superintendent of Documents. These regulations are also published by Agent T. C. George, 63 Vesey Street, New York, New York 10007.

4-3.4.5 Water. The shipment of AE&ODA by water is regulated by the U. S. Coast Guard. These regulations are published in two documents. The CFR, Title 46-Shipping, Parts 146-149 and the Rules and Regulations for Military Explosives and Hazardous Munitions, CG-108. These documents are available from the Superintendent of Documents and HQ U. S. Coast Guard, respectively.

4-3.5 IDENTIFICATION OF MATERIAL. Potentially hazardous material must be identified prior to shipping. Refer to Sax, N.I., et al, Dangerous Properties of Industrial Materials, Library of Congress Card No. 63-20370, Reinhold Publishing Co., N.Y., 2nd Edition, 1963; The Condensed Chemical Dictionary, A. Rose and E. Rose, Reinhold Publishing Co., N.Y., 7th Edition, 1966; Fire Prevention Guide of Hazardous Materials, National Fire Prevention Association International, 60 Battery March Street, Boston, Mass. 02110.

a. Do not accept the manufacturer's and/or fabricator's hazard classification without confirmation by appropriate reference and/or the tests prescribed in TB 700-2 (NAVORDINST 8020.3, TO 11A-1-47, DASR 8220.1).

b. Military AE&ODA may be classified by the military (See Section 173.51(q) of the DOT Regulations).

c. Do not use or allow to be used "Trade Names" for any chemical compound or chemical, e.g., "Freon-12" (R) is not an accepted shipping name. This compound is dichlorodifluoromethane, a compressed nonflammable gas that requires specified containers that must bear the DOT "Green Label" when shipped (the shipping papers require a shipper's certificate.)

d. Manufactured devices are particular problem areas. The manufacturers often call a blasting cap a "squib". Again, it is required that the chemical ingredients be identified. For example, a squib could not contain diazodinitrophenol or mercury fulminate since both of these chemicals are initiating explosives. (See the DOT regulations for these and other initiating explosives.)

e. Sometimes a device may contain an explosive with a desensitizing compound; hence, testing of all new items should be performed according to TB 700-2. Equally, the converse is true and often exists, a normally insensitive, nonhazardous chemical may be mixed with another equally insensitive, nonhazardous chemical to create potential hazard, e.g., iron oxide (Fe_2O_3) and aluminum powder (Al) when mixed, produces a mixture "thermit", which, when ignited, cannot be extinguished, and produces temperatures from 2,000 - 5,000°F. (For shipping purposes, Thermit is a "Special Fireworks", and requires a Special Fireworks Label, a "Dangerous" placard, and appropriate shipping document notations.)

4-4 CLASSES OF HAZARDOUS MATERIALS

Classes of hazardous materials are defined for the various modes of transportation in the section or on the page cited in Table 4-1 (See Governing Regulation cited).

Table 4-1. Classes of Hazardous Materials

MATERIAL	COMMERCIAL AIR Tariff 6-D	MILITARY AIR AFM 71-5 (TM 38-250)	MOTOR & RAIL CFR Title 49	WATER CFR Title 46
Acids	L-55, P-28	8-23	173.240	146.23-1
Alkaline Caustic Liquids	L-190, P-28	8-8	173.240	146.23-1
Bacteriological Agents	L-95, P-32			
Biological Agents		1-1b		
Combustible Liquids				146.26-1
Corrosive Liquids	R-2	8-1	173.240	146.23-1
Etiological Agents	L-240, P-46	1-13		
Explosives, Class A	R-2	5-2a	173.53	146.20-7
Explosives, Class B	R-2	5-2b	173.88	146.20-9
Explosives, Class C	R-2	5-2b	173.100	146-20-11
Flammable Compressed Gases	R-2	9-z	173.300(b)	146.24-1
Flammable Liquids		I-1j	173.115	146.21-1
Flammable Solids		1-1k	173.150	146.22-1
FORBIDDEN ARTICLES		4-1b(8)	173.51	146.20-3
Hazardous Articles				146.27-1
Hypergolic Fuels		1-1(1)		
Magnetized Materials	P-57, P-142	11-19		
Not otherwise regulated		11-1	173.55	
NOT ACCEPTED (Acceptable)	R-3a	4-Ib(11)	173.51	146.20-3
Nonflammable Compressed Gases		9-1	173.300(a)	146.24-1
ORA, Group A	R-2			
ORA, Group B	R-2			
ORA, Group C	R-2			
Oxidizing Material		7-1b	173.151	146.22-1
Poison, Class A		10-3a	173.326(a)	146.25-5
Poison, Class B		10-3b	173.343(a)	146.25-10
Poison, Class C		10-3c	173.381(a)	146.25-15
Poison, Class D		10-51	173.391(a)	146.25-20
Polymerizable Materials	L-60, P-68	7-1c	173.21(b)	146.05-3
Pyroforic Materials		1-1z	173.115(c)	146.21-100
Radioactive Materials	R-2	1-1aa	173.391	146.25-20

L = Line number on the page	P = Page number
ORA = Other Restricted Articles	R = Rule number

4-5. TRANSPORTATION

4-5.1 GENERAL.

4-5.1.1 Additives. The evergrowing demand for high specific impulse rocket motors has initiated a need for close study of the transportation characteristics of the components of these devices. Many of the "additives" used in the formulations of these solid motors are in themselves dangerous and are high-explosive compounds. Since many additives, such as HMX, RDX, and nitroglycerine are used to enhance the specific impulse, the amount used determines the hazard classification for storage and shipping.

4-5.1.2 Test Methods. Each new formulation presents its own problems in hazard classification. Uniform testing methods have assisted and will continue to assist in standardization of hazard classification. Although the testing prescribed by the DOT Regulations were adequate in Pre-World War II days, the sophistication of new devices has caused the Department of Defense, through the Armed Forces Explosive Safety Board (AFESB), to formulate their own testing methods. These methods are promulgated in TB 700-2. Additionally, the DOT has recognized the applicability of TB 700-2, (without citing the document by number) in the provisions of Section 173.51(q) of the DOT Regulations.

4-5.1.3 Purpose of the Regulations.

a. To promote the uniform enforcement of law and to minimize the dangers to life and property coincident to the transportation of explosives and other dangerous articles by common carriers engaged in interstate or foreign commerce, regulations define these articles for transportation, and state the precautions that must be observed by the shipper in preparing them for shipment by air, rail freight, rail express, rail baggage, highway, or by water carrier. It is the responsibility of each shipper to effectively implement prescribed regulations and to instruct his employees appropriately.

b. Explosives and other dangerous articles may be offered to carriers for transportation provided the articles are in the proper condition for transportation, as defined, and are packed, marked, labeled, described, certified, etc., as provided for in the appropriate regulations. Articles must be loaded and stayed according to regulations applying to carriers by rail. Methods of manufacture, packing, and storage, insofar as they affect safety in transportation, must be open to inspection by a duly authorized representative of the initial carrier or of the Bureau of Explosives. Shipments that do not comply with the applicable regulations must not be offered for transportation. Dangerous articles, other than explosives having more than one hazardous characteristic, as defined by the regulations, must be classified according to the greatest hazard present, except those articles which are also poisons (class A, or class D) which must be classified according to both dangerous characteristics.

4-5.1.4 Consignments to the Services. Shipments of explosives or other dangerous articles offered by or consigned to the Departments of the Army, Navy, or Air Force, must be packed and must include the limitations of weight, in accordance with the appropriate regulations or must be packed in containers of equal or greater strength and efficiency.

4-5.2 PREPARATION FOR SHIPPING.

4-5.2.1 Prohibited Packing.

a. Packages of dangerous articles may not be shipped in the same compartment with packages that would cause a dangerous evolution of heat or gas or produce corrosive materials if mixed together, except as specified in the appropriate regulations. (See 4-5.2.4 c).

b. The offering for transporation of any package or container of any liquid, solid, or gaseous material which under conditions incident to transportation may polymerize (combine or react with itself) or decompose so as to cause dangerous evolution of heat or gas is prohibited. Such materials may be offered for transportation when properly stabilized or inhibited. Refrigeration may be used as a means of stabilization only when approved by the Department of Transportation after the recommendation of the Bureau of Explosives.

c. The offering for transportation of any package or container of any other material which will cause a dangerous evolution of heat or gas under conditions normally incident to transportation is forbidden.

d. The offering for transportation of any package containing or equipped with an ignition element, or any similar heating, lighting, or ignition device is forbidden unless the design and method of packaging insofar as they affect safety in transportation have been approved by DOT.

4-5.2.2 Shippers Responsibility. The shipper shall determine that shipments of explosives and other dangerous articles are made in containers which have been made, assembled with all parts or fittings in their proper place, and marked in compliance with applicable specifications prescribed for the container which were in effect at the date of manufacture of container. The shipper may accept the manufacturer's certification as evidence that the containers were manufactured in accordance with applicable specifications.

4-5.2.3 Quantity Limitations.

a. When quantity limitations are specified in the applicable regulations by United States liquid measure or by avoirdupois weight, it is authorized that quantities measured by the metric system may be substituted, up to but not exceeding 1 gallon for liquids and 10 pounds for solids, on the basis of 1 liter per quart specified and 500 grams per pound specified.

b. When quantity limitations do not appear in the packing requirements, the permitted gross weight or capacity authorized for a container to be offered for transportation by carrier by air, rail freight, rail express, highway, or water is shown in the container specification.

4-5.2-4 Railway Express Limitations.

a. Dangerous articles other than explosives, except such as are not accepted for transportation and except where special packing is prescribed in this part for rail express shipments, must, when offered for shipment by rail express be packed, marked and labeled in compliance with the applicable regulations.

b. The maximum quantity of any explosive or other dangerous article that may be offered for transportation by rail express in one outside container must not exceed that shown in the applicable commodity list.

c. When several dangerous articles are placed in one outside package without violating the regulations, the combined quantity of any one group must not exceed the lowest limit prescribed for any one of the articles of that group that is included.

4-5.2.5 Reuse of Containers. Containers used more than once (refilled and reshipped after having been previously emptied) must be in such condition, including closing devices and cushioning materials, that they will protect their contents during transit as efficiently as new containers. Repairs must be made in an efficient manner in accordance with the requirements for materials and construction prescribed in the packaging specifications for new containers, and parts that are weak, broken, or otherwise deteriorated must be replaced. The letters of the markings prescribed for containers should be as near together as is convenient.

4-5.3 MARKING AND LABELING.

4-5.3.1 Explosives.

a. Each package containing explosives must be marked with its proper shipping name as shown in the applicable commodity list and with such other markings as are prescribed for the explosives in the shipment. Abbreviations must not be used.

b. Packages containing explosives must show the marking "THIS SIDE UP" on the top when required by regulation.

c. Each package of explosives must show the name and address of the consignee. When offered for transportation by carrier by water, each package must show the name or mark of the consignor and/or consignee.

4-5.3.2 Dangerous Articles.

a. Packages containing flammable liquids, flammable solids, oxidizing materials, corrosive liquids, compressed gases, and poisons must be marked, unless exempted, with the proper shipping name as shown in the commodity list. For tank cars this marking appears either on the placards or commodity cards.

b. Additional shipping information not inconsistent with the regulations may be shown on the container if so desired, but no such label or marking shall be of a design, form, or size that may be confused with the required marking.

4-5.3.3 Labels for Air Shipment. Labels authorized for shipments of explosives and other dangerous articles are shown in 173.405(b), 173.406(b), 173.407(b), 173.408(b), 173.409(b), 173.410(b), 173.411(b), 173.412(b), and 173.414(c) of the DOT regulations. Shipments so labeled must be tendered with a signed certificate, in duplicate, that reads as stated in 4-5.4.3.1.

4-5.3.4 Labels for Subsequent Split Shipment. When it is known that subsequent shipments of these packages may be made by consignees in less-than-carload or less-than-truckload quantities, or in carload or truckload quantities to a point where they may be handled by other than the original consignee, the original shipper should attach labels to the packages as would be required for less-than-carload or less-than-truckload shipments.

4-5.3.5 Labels for Mixed Packing. Use red label only when red and other labels are prescribed except when poison gas label or radioactive materials label are prescribed, then both the red label and the poison gas label or red label and radioactive materials label must be used.

4-5.4 SHIPPING INSTRUCTIONS.

4-5.4.1 Shipping Order and Bill of Lading Description. A description of the class A, class B, or class C explosive, flammable liquid, flammable solid, oxidizing material, corrosive liquid, compressed gas, or poison, as defined by this part, must be included for transportation by carriers by rail, freight, rail express, highway, or water and must describe such article in the shipping order, bill of lading, or other shipping paper by the shipping name used in the commodity list and may add a further description not inconsistent therewith. Abbreviations must not be used. For shipments of blasting caps and/or electric blasting caps, the shipper must, in addition, show the number of caps in the shipment.

4-5.4.2 Label or Placard Notation. The shipping order, bill of lading, or other shipping paper must also show thereon, in connection with and following the entry of the article, the color or kind of label applied, and for cars containing such articles loaded by the shipper, requiring placards, the kind of placard applied to the car.

4-5.4.3 Certificate. The shipper offering for transportation by carriers by rail freight, highway, water, or air, any class A or class B explosive and blasting caps or electric blasting caps in any quantity, and any flammable liquid, flammable solid, oxidizing material, corrosive liquid, compressed gas, or poison, requiring labels, or carloads requiring placards, as prescribed by the applicable regulations must show on the shipping order, bill of lading, or other shipping paper, in the lower left-hand corner, the following certificate over the written or stamped facsimile signature of the shipper or his duly authorized agent:

"This is to certify that the above named articles are properly described, and are packed and marked and are in proper condition for transportation according to the regulations prescribed by the Department of Transportation."

4-5.4.3.1 Foreign and Passenger Air Shipment. Shipping papers for shipments made by air between the United States and other countries shall be certified in duplicate with certificate signed by the shipper reading as follows:

"This is to certify that the contents of this package are properly described by name and are packed and marked and are in proper condition for transportation according to the regulations prescribed by the Department of Transportation and the Administrator of the Federal Aviation Agency." (For shipment on passenger-carrying aircraft the following must be added to the certificate: "This shipment is within the limitations prescribed for passenger carrying aircraft.")

4-5.4.3.2 Switching Ticket. When the initial movement is a switching operation, the switching order, switching receipt, or switching ticket, and copies thereof, prepared by the shipper, must bear the placard indorsement and the shipper's certificate prescribed by the applicable regulations.

4-5.5 REGULATIONS APPLYING PARTICULARLY TO RAIL FREIGHT.

4-5.5.1 Purpose of the Regulations. To promote the uniform enforcement of law and to minimize the dangers to life and property incident to the transportation of explosives and other dangerous articles by carriers engaged in interstate, intrastate, or foreign commerce, regulations are prescribed to define these articles for transportation purposes and to state the precautions that must be observed by the carrier in handling them while in transit. It is the duty of each such carrier to make the prescribed regulations effective and to thoroughly instruct employes in relation thereto.

4.5.5.2 Acceptable Articles. Explosives, including samples of explosives and explosive articles not exceeding 5 pounds net weight, and other dangerous articles may be accepted and transported, provided they are in proper condition and are certified for transportation by rail, highway, or water. Articles must be loaded, stayed, and handled in transit according to regulations applying to service or services used. Methods of manufacture, packing, and storage, in so far as they affect safety in transportation, must be open to inspection by a duly authorized representative of the initial carrier or by the Bureau of Explosives. Shipments of explosives or other dangerous articles not in proper condition for transportation, or loaded or stayed as required, or certified as to proper packing, marking and description as required in the applicable regulations, must not be accepted for transportation or transported.

4.5.5.3 Forbidden Explosives. The acceptance of certain articles for transportation by common carrier is forbidden. The shipper must consult the appropriate commodity list prior to offering the item for transportation.

4-5.5.4 Improperly Packed or Damaged Shipments in Transportation. For the protection of the public against fire, explosion, or other, or further hazard with respect to shipments of explosives or other dangerous articles offered for transportation or in transit by any carrier by railroad, such carrier shall make immediate report to the DOT Office of Hazardous Materials, Washington, D. C. 20590 for handling any of the following emergency matters coming to their attention:

a. Instances of packages of explosives or other dangerous articles discovered in transit not properly prepared for transportation in accordance with applicable regulation.

b. Railroad wrecks or accidents involving damage to containers of explosives or other dangerous articles that necessitates repacking of the articles.

c. Other similar emergencies in which any carrier is or is likely to become involved or offer aid.

d. This section shall in no respect excuse either shipper or carrier for failure to exercise due care to prevent any departure from any regulation.

4-5.5.5 Loading Cars.

a. Packages containing any explosives or blasting caps or electric blasting caps in any quantity that requires a certified and placarded car must be stayed (blocked and braced) by the loader to prevent any change of position during transportation. Special care must be used to prevent such packages from falling or from having anything fall on them or slide against them during transit. (For recommended methods of blocking and bracing, see Bureau of Explosives Pamphlets 6 and 6A.)

b. Shipments of other dangerous articles must be properly loaded in closed cars, container cars, or in tightly closed truck bodies or trailers on flat cars and cars placarded as prescribed. Ends, side walls, or doors of truck bodies or trailers should not be relied upon to prevent the shifting of heavy loads. (See the Bureau of Explosives' Pamphlet No. 6 for recommended methods of blocking and bracing articles in cars. See the loading and storage chart before loading the labeled articles together or along with explosives. For recommended methods of blocking and bracing, in truck bodies or trailers on flat cars, see Bureau of Explosives' Pamphlet No. 6C.)

c. Shippers must furnish the material for staying packages of other dangerous articles loaded by them.

4-5.5.6 Inspection of Transportation Units Prior to Loading. When a transportation unit (vehicle, railcar) arrives at an installation or railhead for loading, it must be given a thorough exterior and interior inspection to determine its suitability to carry the types of chemical or explosive involved. The interior of the unit should be "broom clean" before the ammunition or chemicals are loaded. Protruding nails and bolt heads should either be removed or covered to prevent damage to packages. Railcars should be in good condition and all safety devices (brakes, doors, floors) should be in good condition. The carrier should certify that the railcar is in good condition and that it meets the standards required for transporting explosives. Vehicles should be inspected to ascertain that the mechanical condition and safety appliances of the vehicle are in good working order. All electrical wiring must be in good condition. Fire extinguishers must be available and in good working condition. Check lists must be provided to personnel responsible for inspecting all transportation units to insure that inspections are properly conducted.

4-6 DEFINITIONS

4-6.1 ACIDS, ALKALINE CAUSTIC LIQUIDS, OR CORROSIVE LIQUIDS. Acids, alkaline caustic liquids, or corrosive liquids will cause severe damage to living tissue, will damage or destroy other freight by chemical action, or may flame on contact with organic matter or certain chemicals.

4-6.2 AMMUNITION. A generic term "ammunition" includes missiles (bullets, projectiles, rockets, grenades, torpedoes, bombs, and guided missiles) and the propellants, primers, fuzes, detonators, and charges of conventional explosive, nuclear explosive, chemical, or other materials. In the broadest sense ammunition includes, all explosives, explosive devices, pyrotechnics, and pyrotechnic devices used for many purposes.

4-6.3 BOOSTER. A booster is an assembly of metal parts and an explosive charge used to augment the explosive component of a fuze for the purpose of detonating the main explosive charge. A booster may be an integral part of the fuze.

4-6.4 BLASTING CAPS. A blasting cap is a small tube, usually copper or aluminum, closed at one end and loaded with a charge (or charges) of high explosives, at least one of which is capable of detonating from the spit or sparks from the safety fuse. Electric blasting caps are blasting caps that can be fired by an electric current.

4-6.5 DETONATOR. A detonator is an explosive-train component which can be activated by either a nonexplosive impulse or a primer and in turn is capable of reliably initiating high-order detonation in a high-explosive. When activated by a nonexplosive impulse, a detonator acts as a primer. In general, detonators are classified with the method of initiation (precussion, stab, electric, flash, etc.). An explosive detonator is an explosive charge used to destroy equipment under certain conditions.

4-6.6 COMBUSTIBLE LIQUIDS. A combustible liquid is any liquid which gives off flammable vapors (as determined by flash point from Tagliabue's open-cup tester used for testing burning oils) at 80 - 150°F. This definition is binding on all shippers making shipments of combustible liquids by any passenger vessel, and shall apply to owners, charterers, agents, masters or other persons in charge of a vessel and to other persons transporting, carrying, conveying, storing, stowing, or using combustible liquids on board passenger vessels subject to R. S. 4472, as amended, and the regulations in this subchapter.

4-6.7 CORROSIVE LIQUIDS. (See 4-6.1)

4-6.8 DOT REGULATIONS. DOT Regulations are numbered by adding "100" to each section of the "ICC" Regulations, e.g., Section 72.5 (Commodity List) of the ICC Regulations becomes Section 172.5 of the DOT Regulations.

4-6.9 EXPLOSIVES. An explosive is any chemical compound, mixture, or device, the primary or common purpose of which is to function by, or which is capable of, explosion, i.e., with substantially instantaneous release of gas and heat.

4-6.9.1 Class A Explosives. A Class A explosive is capable of detonating or is otherwise of maximum hazard as defined in DOT Regulations, Part 173.53.

4-6.9.2 Class B Explosives. A Class B explosive is one which in general functions by rapid combustion rather than detonation. The class includes some devices which contain smokeless powders, such as special fireworks, flash powders, pyrotechnic signal devices, and liquid or solid propellant explosives. Explosives, Class B, are highly combustible or deflagrating, as defined in DOT Regulations, Section 173.88. Explosives, Propellant, Class B, are solid or liquid chemicals or chemical mixtures which function by combustion. The combustion is controlled by composition, size, the form of the grain, or other chemical or mechanical means. Any propellant is included in Class B if it fails to detonate in five trials when tested in the package in which it is offered for shipment. Class B explosive propellants include smokeless powder for small arms and cannon, liquid monopropellant fuels, smokeless powder or solid propellants for rockets, jet thrust units, or other devices. Black powder is not included in this classification and is defined specifically in Section 173.53 of the DOT Regulations.

4-6.9.3 Class C Explosives. A Class C explosive is an explosive that has a minimal hazard as defined in DOT Regulations, Section 173.100. Class C explosives are defined as certain types of manufactured articles which contain Class A or Class B explosives, or both, as components but in restricted quantities, and certain types of fireworks, e.g., explosive rivets containing less than 375 milligrams of explosives, blank small arms cartridges containing less than 500 grains of propellant, and Cordeau Detonant containing less than 400 grains per linear foot.

4-6.10 FLAMMABLE COMPRESSED GAS. A flammable compressed gas is any liquid flammable material having a vapor pressure exceeding 40 psi absolute at 100°F as determined by the Reid method covered by the American Society for Testing Materials Method of Test for Vapor Pressure of Petroleum Products. (See Section 173.300 of the DOT Regulations).

4-6.11 FLAMMABLE LIQUID. A flammable liquid is any liquid having a flash point of 80°F or less in the Tangliabue open-cup tester (73°F or less in the Abel-Pensky closed-cup tester), except when its vapor pressure brings it within the definition of a flammable compressed gas.

4-6.12 FLAMMABLE SOLIDS. A flammable solid is defined as a material, other than one classified as an explosive, which, under conditions incident to transportation, is liable to cause fires through friction, absorption of moisture, spontaneous chemical changes, retained heat from manufacturing or processing, or which can be ignited readily and when ignited burns so vigorously and persistently as to create a serious transportation hazard.

4-6.13 FORBIDDEN EXPLOSIVES. The offering of explosives listed in Section 173.51 of CFR Title 49, for transportation by carriers by rail freight, rail express, highway, or water is forbidden.

4-6.14 HAZARDOUS ARTICLE (COMMERCIAL AIR). A hazardous article is defined as any article or substance (other than an explosive, flammable liquid, flammable solid, oxidizing material, corrosive liquid, compressed gas, poisonous article or combustible liquid, *as defined in this subchapter*) which is liable to spontaneous heating in excess of 10°F when subjected to a test of three continuous hours in a Mackey apparatus at or below 212°F or liberates vapor susceptible to ignition by spark or open flame at or below 300°F.

4-6.15 ICC (DOT) REGULATIONS. Parts 171 through *190 of CFR Title 49 contain* the United States Department of Transportation Regulations for the Transportation of Explosives and other Dangerous Articles.

4-6.16 DOT SPECIFICATIONS. Specifications for shipping containers are set forth in part 178 of the DOT Regulations (49 CFR Part 178 and 179.)

4-6.17 IGNITER. An igniter is any device used to ignite a specifically arranged charge of a ready burning composition which in turn initiates the main propelling charge. It may also be used to amplify the initiation of a fuze primer. Igniters may be of the chemical, electrical, or mechanical type. The ready burning composition is usually black powder.

4-6.18 JATO UNIT. A JATO is a rocket motor consisting of one or more continuous type combustion units closed at one end with a nozzle(s) at the other end. It is normally used to assist in vehicle acceleration. The terms "JATO" and "JATO UNIT" have been discontinued in favor of "ROCKET MOTOR" or "ROCKET ENGINE."

4-6.19 LIQUEFIED COMPRESSED GAS. A liquefied compressed gas is a gas which, under the charged pressure, is partially liquid at 70°F.

4-6.20 NO LIMIT (COMMERCIAL & MILITARY AIR). This term means maximum quantity per package is not limited.

4-6.21 NON-FLAMMABLE COMPRESSED GAS. This gas is any material, not otherwise classified, which will not burn when mixed with air in any proportion and which has an absolute pressure in the container of greater than 40 lb/sq in. at 70°F or exceeding 104 lb/sq in. at 130°F, or both.

4-6.22 NON-RESTRICTED. The term means not subject to or not restricted by regulations.

4-6.23 NOT ACCEPTED (COMMERCIAL & MILITARY AIR). Any article listed in the commodity list as "not accepted" cannot be carried on any aircraft governed by the commodity list concerned.

4-6.24 OTHER RESTRICTED ARTICLES (COMMERCIAL AND MILITARY AIR). These articles are those items which are not otherwise defined by regulation and which possess characteristics as described in one of the following groups.

4-6.24-1 Group A. Noxious, toxic, irritating, or other characteristics which can cause extreme annoyance or discomfort to passengers and crew in the event of leakage in flight.

4-6.24.2 Group B. Capable of causing damage to aircraft structures, such as solid materials which are strongly corrosive when wet.

4-6.24.3 Group C. Other inherent characteristics which make it unsuitable for air carriage, unless properly prepared for shipment, e.g., etiologic agents, polymerizable materials, safety matches, etc.

4-6.25 OXIDIZING MATERIAL. An oxidizing material is a substance that yields oxygen readily and includes materials such as bromates, chlorates, nitrates, perchlorates, permanganates, peroxides, or nitro carbonitrate.

4-6.26 POISONS, CLASS A - POISON GAS LABEL. Extremely dangerous Class A poisons are poisonous gases or liquids of such a nature that a very small amount of the gas, or vapor of the liquid, mixed with air is dangerous to life.

4-6.27 POISONS, CLASS B. The materials are liquids or solids substances (including pastes and semisolids), other than classes A, C, or D poisons which are hazardous during transportation, or which, in the absence of adequate data on human toxicity, are presumed to be toxic to man. (See Section 173.343 of the DOT Regulations.)

4-6.28 POISONS, CLASS C. These materials are liquid or solid substances (brombenzylcyanide, chloracetophenone, dipnenylaminechlorarsine, or diphenylchlorarsine), which upon contact with fire or when exposed to air give off dangerous or intensely irritating fumes but they do not include any Class A poisonsous articles.

4-6.29 POISONS, CLASS D (RADIOACTIVE MATERIALS). These substances are any material, or combination of materials, that spontaneously emit ionizing radiation with a specific activity of greater than 0.002 microcuries per gram. Radioactive materials are divided into four groups as follows:

4-6.29.1 Group I. Radioactive materials that emit gamma rays only or both gamma and electrically charged corpuscular rays.

4-6.29.2 Group II. Radioactive materials that emit neutrons and either or both the types of radiation characteristic of Group I materials.

4-6.29.3 Group III. Radioactive materials that emit electrically charged corpuscular rays only, i.e., alpha or beta, or any other material that is so shielded that the gamma radiation at the surface of the package does not exceed 10 milliroentgens for 24 hours during transportation.

4-6.29.4 Group IV. Group IV fissile radioactive materials include uranium-233, uranium-235, plutonium-238, plutonium-239, and plutonium-241. They are classified according to the controls needed to provide nuclear criticality safety during transport as follows:

a. Fissile Class I. Packages which may be transported in unlimited numbers and in any arrangement, and which require no nuclear criticality safety controls during transport.

b. Fissile Class II. Packages which may be transported together in any arrangement but in numbers which do not exceed an aggregate of 40 radiation units. Such shipments require no nuclear criticality safety control by the shipper during transport.

c. Fissile Class III. Shipments of packages which do not meet the requirements of Fissile Classes I or II and which are controlled transport by special arrangements by the shipper.

4-6.30 PYROFORIC. A pyroforic liquid is a flammable liquid (see section 4-6.11 of this section) that becomes self-igniting when exposed to normal atmospheric conditions.

4-6.31 PRIMARY HIGH EXPLOSIVE. This material is an explosive which is extremely sensitive to heat and shock and is normally used to initiate a secondary high explosive. A primary explosive is capable of building up from a deflagration to a detonation in an extremely small diameter column. The term is generally used in referring to a pure compound and not to an explosive mixture. Mercury fulminate, lead azide, lead styphnate, and tetracene are included in this class.

4-6.32 PRIMER. A primer is a relatively small and sensitive initial explosive-train component which on being actuated initiates the explosive train. A primer will not reliably initiate high-explosive charges directly. In general, primers are classified according to the method of initiation, e.g., percussion, stab, electric, friction, etc. "Primer" is also used as a term referring to the assembly which ignites propellant charges.

4-6.33 PROHIBITED EXPLOSIVES. Explosives prohibited by Section 146.20-3, CFR Title 46, fulminates or other detonating compounds in bulk in dry conditions, or explosive compositions that ignite spontaneously or undergo marked decomposition when subjected for 48 consecutive hours to a temperature of 167°F, or compositions containing an ammonium salt and a chlorate, or other like explosives, shall not be accepted by any vessel. A passenger vessel shall not accept any Class A or Class B military explosives for transportation as cargo.

4-6.34 PROPELLANT. A propellant is a low-explosive substance or mixture of substances which, through burning, can be made to produce gases at controlled rates and to provide the energy necessary to propel a projectile or missile. Propellants may be classified as liquid propellants or solid propellants according to their physical state.

4-6.35 RADIOACTIVE MATERIALS. See POISONS, CLASS D (4-6.28).

4-6.36 RESTRICTED ARTICLES. A restricted article is any item defined as a corrosive liquid, explosive, flammable compressed gas, flammable, poisonous substance, radioactive material, etc., or which is listed in Section II of the Tariff cited in paragraph 4-3.4.1 and is not marked "Not Restricted."

4-6.37 ROCKET FUEL. This material is a fuel, either in liquid or solid form developed for, or used by, a rocket. This may include an oxidizing agent in some cases.

4-6.38 ROCKET MOTOR. This device is a chemical reaction propulsion device that consists of a thrust chamber(s) and exhaust nozzle(s) and that carries its own solid oxidizer-fuel combination.

4-6.39 SQUIB. This term is used in a general sense to mean any of various small pyrotechnic or low-explosive devices that are usually initiated electrically or mechanically.

4-7 STORAGE. (Missiles, their components, rocket motors and their associated chemical).

4-7.1 SAFETY RULES.

4-7.1.1 Storage Buildings. Bulk chemical ingredients, propellants (both in process and in completed form), explosives, and ammunition will be stored in buildings designed, designated, and isolated for the specific purpose.

4-7.1.2 Storage Arrangement. Propellants and their ingredients in bulk storage, and other types of ammunition, will be stacked by lot (batch) number and arranged so that free circulation of air beneath and throughout the stack is possible. When more than one lot is stored in the same vicinity, all items or containers of a lot will be stored together and the line of separation between the lots will be clearly indicated.

4-7.1.3 Containers. Boxes, cases, and other containers will be clean and dry before storage. Containers will not be opened in the storage area. They should not be stored after having been opened unless they are securely closed, except that ammunition and explosives in damaged containers in process of being repaired may be stored temporarily overnight in isolated magazines.

4-7.1.4 Prohibited Equipment. Components should not be kept loose in a magazine. Empty containers, excess dunnage, or tools will be permitted to remain in a magazine only during the time required for the particular job. Oily rags, paint, and other flammable materials will not be left in a magazine.

4-7.1.5 Mixed Storage. Incompatible hazardous materials will not be stored together.

4-7.1.6 Pyrotechnic Items. Black powder, tracer composition, pyrotechnic mixtures, or similar items which are subject to rapid deterioration in damp atmospheres or at continued high temperatures, should be stored in adequately ventilated buildings that provide protection against dampness.

4-7.2 OUTDOOR STORAGE.

Outdoor storage is neither desirable nor recommended and should be utilized only in an extreme emergency as a temporary expedient. Solid propellants, pyrotechnics, critical items, and bulk high-explosives should not be stored outdoors.

4-8 SURVEILLANCE AND INSPECTION

4-8.1 GENERAL. The term surveillance includes the observation, inspection, test, study, and classification of chemicals, ammunition, ammunition components, rocket motors, propellants, other explosives, and other dangerous articles in movement, storage, and use. The term is related to the degree of serviceability and rate of deterioration, the inspection of the containers and buildings in which dangerous articles are stored, the inspection of facilities and methods used in handling, storing, shipping, maintaining, reconditioning, renovating, salvaging, and destruction of ammunition, and the preparation and maintenance of all technical data, reports, and records, required by current safety and operating regulations.

4-8.2 SURVEILLANCE PROGRAM. A surveillance program should be in effect at any facility that handles chemicals or ammunition. It is necessary to insure that all items are in a serviceable condition and ready for use and that items which are not serviceable will be repaired, salvaged, destroyed, or evacuated as required. The program must outline procedures and practices concerned with the prevention of chemical or ammunition deterioration and the promotion of safe chemical and ammunition handling practices.

4-8.3 STANDARDS OF SERVICEABILITY.

4-8.3.1 Inspection. The objective of inspection is to determine the state and nature of any deterioration present and the degree of serviceability of the item inspected; that is, to ascertain whether the item inspected is serviceable as it stands, requires reconditioning or renovation, or must be rejected as unserviceable.

4-8.3.2 Safety. A discussion of serviceability standards must start with an emphasis on safety. No item of ammunition may be considered serviceable if it is not safe to handle or use, even though proper instructions for the item are followed.

4-8.3.3 Procedures. Inspection procedures include visual inspection, physical tests, such as gaging or strength tests, and functional tests in which sample rounds are fired and the results observed.

4-8.3.4 Deterioration. Moisture is the prime enemy of many chemicals and ammunition. When moisture is combined with a rise in temperature, deterioration of the chemicals is accelerated. An inspector should be especially watchful for rust, corrosion on projectiles and fuzes, corrosion and incipient cracks on cartridge cases, deterioration of propellants, loose closing caps, and moisture or dampness on or in containers.

4-8.3.5 Classification. Based on the results of inspection, chemicals and ammunition are classified into categories. The category "for immediate destruction" is applicable to hazardous chemicals and ammunition. An item having a fuze or igniter in an armed position, which cannot be rendered safe, items with damaged fuzes, or any item that cannot be definitely identified as safe must be considered unsafe to handle. These items, and any others that appear to be in a condition so critical that they may be set off by shock, should be carefully isolated for destruction as quickly as practicable. Such materiel should not be allowed to accumulate in excessive quantities. In some cases it may be found advantageous to move other materiel away from the unsafe items.

4-8.4 FIELD SURVEILLANCE. This includes inspection of containers and buildings in which chemicals, ammunition, and explosives are stored, and of operations involved in the handling, storing, and shipping of chemicals, ammunition, and explosives. It may also include inspection of destruction procedures.

4-8.5 INSPECTION OF MAGAZINES AND STORAGE AREAS. Magazines and storage areas are usually inspected monthly, or more often based on local conditions. Unsafe practices, methods, or conditions which are noted during the inspections must be reported and immediate corrective action taken. Points which should be carefully checked during inspection are listed as follows:

a. Compliance with storage chart.

b. Segregation of lots.

c. Adequacy of aisles.

d. Stability of stacks.

e. Separation of stacks by safety distances where such are specified.

f. Compliance with quantity limits in stacks and magazines.

g. Satisfactory "housekeeping" in the magazine.

h. Maintenance of adequate firebreaks around above-ground type.

i. Magazines and outdoor storage sites.

j. Condition of doors and ventilators.

k. Suitability of the magazine for the type of material in storage.

l. Compliance with quantity-distance limitations for the nearest magazine, public railway, public highway, inhabited buildings, or operations buildings.

4-8.6 INSPECTION OF INCOMING SHIPMENTS. The inspection of incoming ammunition, chemicals, and other dangerous articles is accomplished to assure that damage in transit has not occurred. Normally, visual inspection of random selected containers will be indicative of the condition of the material.

4-8.7 INSPECTION OF MISSILES AND HEAVY ROCKETS.

4-8.7.1 Instorage Inspection. Instorage inspection of missiles and heavy rockets will normally consist of a visual examination of the dessicants or humidity indicators within the missile or rocket containers. Missile and rocket containers will also be inspected for damage or indication of tampering or rough handling. These inspections will be conducted at all installations or activities handling missiles or heavy rockets.

4-8.7.2 Serviceability Questionable. When the serviceability of a missile is questioned, due to the condition of the containers or due to abnormal changes in dessicants or humidity indicators, assistance will be requested from the customers.

4-8.7.3 Damaged Containers. If containers containing solid propellants for missiles or heavy rockets have been dropped or appear to have been dropped or damaged to the extent that the grains may be cracked, the containers will be restricted from issue until a thorough inspection of the grain has been made.

4-8.8 SURVEILLANCE RECORDS. Surveillance records contain information gathered during the performance of surveillance and inspection activities. Surveillance records not only are the basis for the preparation of reports but they are vital in determining the rate of deterioration of chemicals and ammunition since the last inspection. This file is a valuable reference and source of information in connection with both routine and special inspections and investigations of materials. The results of all investigations, inspections, and tests and a record of any unusual or changing conditions affecting the chemicals or ammunition will be entered on the card and will become a part of the technical history of the material.

CHAPTER 5

ASSEMBLY, LAUNCH AND STATIC TEST

5-1 INTRODUCTION

The high potential energy of the launch vehicles or test motors is hazardous to people and property in that inadvertent ignition or other undesired event may cause death, injuries and property losses. Accordingly, elimination, or at least containment of the dangers, is a facet of the assembly, test, and launch of the vehicles. This section of the manual touches upon certain of the most fundamental hazards and general precautions. Safety requirements in accordance with appropriate regulations are mandatory for transportation, receiving, assembly, checkout, conditioning and firing. Every effort is made to create the safest environment possible and eliminate the recognized hazardous conditions.

The hazards potential is lessened by isolating the people who are working with explosives from others who are not essential to the work being done, and by isolating the ordnance item itself. Basically, the retention of only essential people and limiting explosive and propellant quantities to the absolute minimum is the fundamental safety precaution.

5-2 GENERAL PRECAUTIONS

5-2.1 QUANTITY DISTANCE (QD) REQUIREMENTS. The QD requirements for assembling missiles are an expression of the actual distance required to isolate a specific quantity of explosive. Military explosives and propellants are classified upon the basis of their characters and the predominance of the hazards presented if they should explode or ignite. The various classes are listed in Department of Defense (DoD) Instructions. Usually, in assembling and launching vehicles we are concerned with Class 7 materials such as an explosive warhead or a detonable propellant. Class 7 by weight is equivalent to or greater than the same amount of TNT while Class 2 is mainly a fire and fragmentation hazard such as a nondetonable propellant.

5-2.2 DISTANCE REQUIREMENTS FOR SOLIDS. Quantity distance requirements for solids are determined as follows:

a. Class 7, use QD tables in DOD Instruction 4145.17 or applicable service manual.

b. Class 2, use QD tables in DOD Instruction 4145.17 or applicable service manual. Note: Because of the fragmentation, the Class 2 distances are not normally enough to protect personnel and property; therefore, the distance is usually increased by using the results of fragmentation tests of Class 2 propellants. Some of the military services use Class 7 distance when item is on a static test stand or a launcher.

c. Combinations of Class 7 and Class 2 are rated in terms of % TNT equivalence. Distances are then determined using Class 7 tables of DOD Instruction 4145.17 or service manuals.

d. A TNT equivalency rating system sometimes used by the National Ranges is:

(1) Class 7. 100% TNT

(2) Class 2 adjacent to Class 7. 100% TNT for total weight of both Class 7 and Class 2. All other Class 2 solid rocket motors in the assembly are 25% TNT. Actual TNT equivalencies depend on the propellant formulations, and may be greater than the numbers expressed.

5-2.3 DISTANCE REQUIREMENTS FOR LIQUIDS. Quantity Distance for large liquids are based on the quantity and type of propellants used as listed in DOD Instruction 4145.21 and applicable service documents.

5-2.4 DISTANCE REQUIREMENTS FOR LIQUID/SOLID SYSTEMS. There are no set rules for determing the quantity distance for missiles having a liquid core combined with solids. Destructive tests of these combined systems are inadequate for a clear establishment of QD relationships, however, a policy used at some Ranges to rate such combined systems is:

a. The TNT equivalence specified for the liquids, plus 100% TNT equivalency for Class 7 solids.

b. The TNT equivalence specified in appropriate service manuals for the liquids plus 50% TNT for Class 2 solids.

5-3 ELECTRICAL HAZARDS

Electrical hazards are significant because of the possible inadvertent electrical ignition of Electro-Explosive Devices (EED), flammable or explosive atmosphere, solid rocket motors, or similar devices. Because of the potentially catastrophic results of stray electrical currents, fundamental precautions must be taken.

5-3.1 STATIC ELECTRICITY. Static electricity is a potential ignition source when it accumulates to a voltage differential high enough to result in insulation failure. When the insulation fails or becomes inadequate, the resultant sudden discharge of energy creates an arc, which may ignite an explosive or inflammable atmosphere. The discharge path might be across the bridgewire of an EED causing inadvertent ignition. The best way to eliminate the hazard of static build-up is through grounding. With all locations of static build-up maintained at the same potential by low resistance grounds, it is impossible for static to accumulate and discharge. Removal of the static charge producers is helpful but identification of all the producers is difficult. Certain materials such as plastics and synthetics should be avoided whenever it has been determined that static electricity is a potential hazard. Personnel working in the vicinity of a hazardous atmosphere or ordnance items should also be grounded. Wrist stats, leg stats, and conductive shoes are typical methods of personnel grounding. The National Electric Code, "Grounding in Hazardous Locations", and applicable service manuals are good references on the subject.

5-3.2 ELECTRICAL EQUIPMENT. Arcing and Sparking of electrical equipment are potential ignition sources in hazardous locations. The National Electric Code, "Equipment in Hazardous Locations", is one of the best sources of information on this hazard and its elimination.

5-3.3 RADIO FREQUENCY (RF) ENERGY. Radio Frequency energy may be induced in the leads of an EED and cause ignition under some circumstances. Induction occurs whenever a receiving antenna is placed in an RF field. There is always a possibility that EEDs, igniters, squibs, blasting caps and electrically initiated devices, when exposed in an RF field, such as that created by radar, radio transmitters and other RF sources, may be ignited by induced RF. Many RF sources are used on the ranges and in missiles, therefore, surveys of launch and assembly positions should be made. Consequently, if a hazard exists, RF sources must be controlled when ordnance devices are exposed. This control applies during checkout, transport, assembly, installation, and hookup. Basic control of this hazard consists of shielding the ordnance leads and firing circuit and using shielded shipping containers. If there is any doubt about the effects of the shielding or susceptibility of the ordnance item, the suspect RF sources should be shutdown when the ordnance item is handled or its leads exposed.

5-3.4 STRAY CURRENTS. Additional sources of stray currents capable of igniting ordnance must be considered and controlled. Ground support equipment and missile flight equipment contain complicated electrical-electronic systems capable of causing inadvertent ignition. Careful consideration should be given to the physical layout of all circuitry associated with electrically initiated ordnance items, to reduce the chances of stray voltages being introduced into the firing system. Some of the features that should be considered are:

a. Physical separation of firing circuitry from instrumentation and other current carrying conductors.

b. Connectors in firing circuits separate from all other connectors and non-interchangeable.

c. Firing circuitry shielded through 360°.

d. Ordnance leads and firing system configured to eliminate their antenna effectiveness.

e. Firing circuits completely isolated from control and monitor circuits.

f. Test current from a Resistance Test Set is dangerous in that the ordnance may fire during the test. Consequently, careful consideration must be given to the safety features of the meter including:

(1) current limiting to safe no-fire current level with ample safety factors;

(2) current limiting circuitry and resistors protected against shorting;

(3) power supply from a low voltage, self-contained battery.

5-3.5 LIGHTNING. Lightning protection information is found in applicable service manuals. The area supervisor may suspend ballistic tests and clear the area when an electrical storm is immiment. Operations should not commence until the storm has passed.

5-3.6 ELECTRICAL HAZARDS TO PERSONNEL. Hazards are increased because of the requirement for personnel grounding during certain operations. Personnel required to wear static grounding devices should be very careful when working in the vicinity of power sources.

5-3.7 GROUNDING ELECTRICAL EQUIPMENT. All electrical equipment within the test area shall be grounded in accordance with the National Electrical Code. Electrical equipment includes, but is not limited to, the following permanently installed equipment: data acquisition systems, control equipment, warning systems, lighting systems, switch gear, heating and ventilating equipment, power supplies, lighting protection systems, photographic control systems, closed circuit TV systems, emergency lightning systems, electric stoves, electric coffee makers, motor generators, environmental test chambers, static test stands, and all metal fixtures in the vicinity of a hazardous test.

5-3.7.1 Method. Grounding systems may be made by connecting to existing water systems or by driving electrodes into the earth. In cases where earth conductance is very low, a good ground may be obtained by percolating salt solution into the earth surrounding the electrode.

5-3.7.2 Conductance Measurement. Earth conductance measurements shall be made to determine the optimum method of grounding. It is important that the study include the maximum and minimum seasonal fluctuations in water table and earth moisture content.

5-3.7.3 Testing. It is also important to maintain an active program for ground testing of all electrical equipment. The maximum acceptable value for electrical resistance of electrical equipment to ground shall be two ohms.

5-4 ENVIRONMENTAL HEALTH HAZARDS

Environmental health hazards to personnel include the hazards of noise, ionizing and nonionizing radiation, and chemical exposures. (Refer to Vol. I.)

5-4.1 NOISE. Noise presents a danger of damage to hearing organs due to high sound pressure levels from various sources. Excessive noise can cause temporary and/or permanent hearing loss to inadequately protected personnel. Such factors as intensity, duration, frequency of repetition, and the individual susceptibility of persons exposed must be considered in establishing standards for noise exposure.

A summary checklist for hazardous noise exposure follows:

a. Locate all noise sources generating noise fields that may result in hazardous noise exposures for personnel.

b. Determine the operating schedules of these noise sources.

c. Identify areas where hazardous noise exposure may occur.

d. Record the duration of specific levels as changes occur during the operating schedule.

e. Personnel whose duties require exposure to noise hazards will be given preassignment audiometric examinations.

f. Follow-up periodic examinations should be given at intervals depending on the degree of exposure.

5-4.2 RADIATION. Control of radiation exposure may be accomplished by the following.

a. Maintain controls (shielding, direction, etc.) to the minimum area required to perform the desired task.

b. Restrict access of personnel to those required for the performance of the desired task by means of signs, barricades, etc.

c. Monitor and record exposures of personnel assigned to the controlled area.

5-4.2.1 Ionization Radiation. Ionizing radiation sources used on launch facilities fall into three distinct areas; calibration sources in the Microcurie-Millicurie range, auxiliary power units (APUs) in the Megacurie range, and X-ray or radiographic examination of components. Control of the first is more an administrative than a health requirement. The latter two, however, present the greater hazard. Requirements for larger auxiliary power units, extended satellite lifetimes, and more complex launch vehicles and payloads, will increase the radiological health hazard.

5-4.2.2 Microwave Radiation. Microwave radiation is a hazard to personnel. It differs from ionizing radiation in that it does not possess sufficient energy to cause ionization. It is similar to infrared in that it causes localized heating, but its penetration may be deeper. The hazard associated with microwave is a result of this localized heating, therefore, those body areas that are less efficient at dissipating heat are the most affected, namely, the lens of the eye. Permanent corneal damage has been documented. Sources of microwave radiation are radar beams directed toward the launch vehicle, and telemetry sources on the missile, or within the payload.

5-4.3 CHEMICAL AND TOXIC HAZARDS. Explosion, fire, and toxic hazards are associated with the chemicals and also with their decomposition products. Personnel injury may vary from external irritation to internal poisoning. Inadequate ventilation, spills, leaks, and disposal are the main sources of health hazards.

General precautions for chemical and toxic hazards include the following.

a. Provide adequate ventilation.

b. Provide respiratory protective equipment for concentrations in excess of the threshold limit value (for temporary use only, not as a substitute for proper controls).

c. Provides facilities for removal or neutralization.

d. Develop operational procedures and training programs.

e. Consider compatibility or substitution of less hazardous materials.

f. More detailed information is available in Volume I.

5-5 CONTROL OF ACTIVITIES

Essential Safety Precautions include a definition of hazardous activities during the flow from receipt to launch, in order that these activities may be controlled. The following controls are to be included.

a. Approval of buildings and facilities for specific operations.

b. Posted explosive weight and personnel limits.

c. Proper fire fighting symbols displayed on buildings and/or areas where explosive material is located.

d. Detailed, approved procedures for hazardous operations and all possible emergencies.

e. Designation of qualified safety supervisor for hazardous operations.

f. Use of trained qualified personnel with adequate equipment.

g. Application of National Electric Code requirements to hazardous atmospheres and environments including ordnance areas and buildings.

h. Exposure of only minimum essential personnel to hazards.

i. Propulsion restraints such as tie-downs, spikes, I beams and other devices to eliminate the threat of inadvertent propulsion.

j. Initiators and nozzles installed as late as possible in the countdown.

5-6 MOTOR ASSEMBLY

5-6.1 SOURCES OF HAZARD. Some sources of hazard during assembly of motor components are the possibility of accidental ignition of the motor by initiating the igniter or by squeezing entrapped propellant in threads, crevices, and cracks either to cause a localized explosion or to generate enough heat to cause autoignition of the propellant. Accidental ignition may be caused electrostatically, through RF energy, application of power through test equipment, and shorting.

5-6.2 ENVIRONMENT AND GENERAL PRACTICE. No parts should be forced during assembly or disassembly, as propellant may be entrapped between the mating parts or else enough heat may be generated to ignite the propellant. The motor shall be properly restrained against flight if accidentally ignited during the assembly sequence. Initiators should be left out of the motor until final installation for flight or testing, if possible. Any safe/arm device shall be in the safe position.

5-6.2.1 Presses and Tools. Assembly presses shall be provided with blast deflectors to protect the operator and surrounding units. Assembly tooling should not obstruct either the nozzle or the igniter boss during assembly. These precautions apply equally to assembly and disassembly of motors.

5-6.2.2 Grounding Checks. Grounding precautions shall involve procedures that frequently check the effectiveness of the grounding.

5-6.3 NOZZLE AND HEAD PLATE ASSEMBLY. Where the nozzle or head plate is directly screwed onto the motor case, threads should be double checked for propellant contamination. Where the nozzle or head plate is bolted onto the case, bolts and bolt holes shall also be checked for propellant entrapment. For small test motors, flanged designs are recommended.

5-6.4 ASSEMBLY OF THE IGNITION SYSTEM. By nature, the ignition system is more sensitive than the motor that it ignites. As many as possible of the operations that are not hazardous should be done in a nonhazardous area. Preparation of initiating mixtures, such as the electric squib, is extremely hazardous. All equipment shall be properly grounded. The operator must be grounded, and an observer must be present who is also grounded. Flame-retardant clothing and goggles shall be worn. Spark or friction ignition of certain pyrotechnic mixtures is an extreme hazard, and operations with such mixtures should be conducted in a "glove box" under an inert atmosphere, using approved tools. Aged ignition materials may become more sensitive and must be used with caution.

5-6.4.1 Igniter Assembly. The assembly of the igniter should allow the live squib to be installed immediately prior to test or at the last possible moment during flight assembly. Igniter chambers should be restrained during the installation of igniter pellets or grains, in a manner similar to that for loaded motors. Assembly operations involving torquing thread igniter components shall be accomplished remotely or behind operational shields.

5-6.4.2 Quantity in the Assembly Area. Minimum quantities of igniter assemblies shall be maintained in the assembly area. Completed assemblies should be stored in metal containers.

5-6.4.3 Installation of the Ignition Assembly. The ignition installation procedures must be established into a Standard Operating Procedure (SOP). It shall specify the personnel qualified for each operation, the maximum number of personnel allowed in the working area, and the instrumentation to be used for squib checkout. The sequence of operations shall be called out in detail and shall be precisely followed.

5-6.4.4 Shielding. Because of electromagnetic hazards, connector-type squibs should be fitted with RF opaque "dust" caps that are nonshorting (electrically isolated from firing leads). Pigtail squibs should have twisted, shielded leads which terminate in a connector, have no exposed contacts, and have the connectors covered by an RF opaque connector cap, preferably nonshorting and electrically isolated from the connector pins. The overall length of the pigtail should be as short as practicable. No firing leads shall utilize connectors or shielding in common with any other circuit whatsoever. Broad-band RF detectors should be used where RF hazard is high.

5-6.4.5 Static-Generating Materials. Because of electrostatic hazards, no static-generating materials (such as polystyrene, polyethylene, or other synthetics, unless it is one of the new conductive ones) shall be utilized for packaging or dust covers of either ignition systems or rocket motors. Moreover, static-generating materials should not be allowed in the area where motor assembly is occurring. Anti-static, conductive sprays may be used on items such as nozzles and motor cases. Cover materials, if used, should have a resistance of no more than 4000 ohms per foot. Large conductive components of an assembly should be grounded with a grounding strap (1/2 inch braid) of minimum length. Maximum resistance should be about 25 ohms. If leg stats are used, conductive flooring is required. The conductivity of such precautionary devices must be checked and frequently verified.

5-6.4.6 Igniter Installation. Igniters are to be removed from metal boxes just prior to installation. Installation shall be done as late as practicable. Firing leads should be shorted and connected to ground via a bleed resistor of one megohm. A no-voltage check should be made of the firing leads from the power source immediately prior to joining these leads to the squib connector. The RF cap should be removed from the igniter connector as late as practicable, and the power leads should then be immediately connected to the igniter. Any final resistance check-out should be accomplished remotely, with the area cleared, using approved instrumentation or circuitry. Circuit testers should be checked to see that only negligible currents are capable of being passed.

5-6.5 BURST DIAPHRAGMS. For heavy-walled test motors, burst diaphragms may be incorporated to relieve unexpectedly high pressures. Where they are mounted by a threaded piece to the motor chamber, care is required to check threads for propellant contamination.

5-6.6 MOTOR REWORK. The repair of loaded motors shall be done with extreme care. Hand trimming of propellant to allow liner or insulation repair must follow all the precautions for manual trimming and cutting. The motor must be restrained during rework.

Spot welding on loaded motor chambers may be done only with extreme precautions. Capacitance spot welding can be done to install thermocouples or strain gauges on metal no thinner than 0.010 inch. All transformers, switches, and storage condensers that must be in the vicinity should be enclosed in an explosion-proof container. Surface spots on the case to be welded should be cleaned to bare metal, to minimize contact sparks.

5-7 HAZARD OF STATIC TESTING AND LAUNCHING

5-7.1 INTRODUCTION. The hazards of static testing and launching have been studied very thoroughly. The physical hazards involved in handling propellants and controlling high pressure combustion processes impose safety precautions which govern the method used. Almost all tests or launch with live propellants are conducted by remote control from a protected barricade or blockhouse. Precautions are usually taken to minimize the damage in case of accident or explosion. Following are the more common safety features for testing or launching a solid propellant rocket engine.

a. Protection provided operators by concrete blockhouses or barricades some distance away from the test setup.

b. Closed circuit television for remote viewing of the test.

c. The separation of individual test stations by walls so that an accident in one test station will not affect the remainder of the test stations.

d. Warning signals to notify personnel to clear a hazardous area prior to tests.

e. The limitation of the amount of propellant stored in any one station.

f. Instruments of the remote recording type.

g. The prohibiting of smoking or flame producing material where propellants are present. Sparkproof shoes and tools utilized where needed.

h. Personnel limits posted in test stations.

i. The establishment and rigid enforcement of safety rules to minimize carelessness.

5-7.2 TEST FACILITY LAYOUT CRITERIA. The following criteria are the basic considerations for layout and design of a solid rocket motor test or launch facility. Once the facility becomes operational, additional data should be gathered, whenever possible, to augment these primary requirements and aid in future designs.

a. Natural terrain versus earth revetments for protection against ballistic projectiles.

b. Proximity to inhabited buildings, railroads, and highways.

c. Quantity-distance requirements.

d. Effects of noise on surrounding areas and neighbors.

e. Perimeter control to keep out personnel, animals, etc. from the neighboring area.

f. Predominate wind direction in relation to the surrounding area.

g. Air pollution control as dictated by governmental agencies.

h. Underground water pollution control as dictated by governmental agencies.

i. Disposal or dispersion of contaminated items of products.

j. Climatic conditions that would preclude unsafe operations, e.g., ice, electrical storms, excessive rains, temperature variations, etc.

k. Aircraft patterns, both commercial and military, that would involve aircraft overhead at low altitudes during vertical nozzle-up firings, first order detonations, or launch.

5-7.2.1 Static Test Bays. Static test bays that will sustain the least amount of damage during a major malfunction are listed below in order of preference.

a. One-hundred percent open test bay, concrete base with a steel plate or rails.

b. Test bay, open on two sides and top.

c. Double open ended horizontal or one side and top open vertical test bay.

d. Closed test bay (usually underground), with only the firing end open.

5-7.2.1.1 Sites. Launch sites are always open with protective personnel shelter provided at adequate distance.

5-7.2.1.2 Solid Rocket Motor Restraining (ANTI-FLIGHT) Gear. Test restraint should be provided for all active tests (static firing, vibration, structural, etc.) The design loads should be:

a. Four times the nominal thrust applied axially forward or aft.

b. Two times the nominal thrust applied in any direction in the transverse plane at any retainer. Resulting stressed should not exceed ultimate material allowables.

5-7.3 PERSONNEL CONTROL.

5-7.3.1 Control Lights. Control lights may be provided at all normal entrances to each test or launch area to designate the progress of testing. Green light or no light indicates the normal status of the test area with no restriction on traffic.

5-7.3.2 Barrier Gates. Gates can be used to control personnel traffic into restricted test and launch areas.

5-7.3.3 Signs. Suitable signs should be displayed at the entrance to the test area indicating the following.

a. Use of radio transmitters prohibited beyond this point.

b. Flame or spark producing devices are prohibited beyond this point.

c. Test area is closed to all unauthorized vehicles and personnel.

5-7.3.4 Audible Warning. A suitable audible warning system should be activated at least five minutes before the start of a hazardous test or launch to clear personnel from the area.

5-7.3.5 Personnel Limits. In any operation dealing with potentially hazardous materials or situations, only the personnel necessary to perform the work should be allowed in the area. Therefore, a determination must be made to establish personnel limits in the various operating areas, consistent with safe and efficient operation of the area. Personnel limit signs should be prominently displayed at the entrances to any hazardous building or area. The number of people in the area should be strictly controlled by the test supervisor. Personnel limit signs should be periodically updated as required by changes in operations.

5-7.3.6 Observation Control. Visual observation of tests by means of TV or indirect viewing devices is desirable for the following reasons.

a. The observer is allowed to assess property damage, recommend type of fire fighting, and direct control of post firing operations in the event of a motor malfunction.

b. The observer is allowed to note any unusual features of a test.

c. Insurance that the test area is clear is provided.

d. Visitors are allowed to observe tests.

5-7.3.7 Smoking regulations. Smoking is permitted in the test area, but only in designated areas where electric lighters are provided. Signs bearing the inscription "Smoking Is Not Permitted Beyond This Point" should be prominently displayed near all exits from the smoking area.

5-7.4 ACOUSTIC NOISE.

5-7.4.1 Importance of Controlling. Acoustic noise should be an important factor in the operation of ballistic test and launch facilities. Noise should be controlled, when practical, because of its effect on the surrounding community and the health of the employee.

5-7.4.2 Reflection and Accentuation. Ballistic testing and launching offer unique noise problems that are not easily solved. For example: temperature inversion of the area surrounding the site serves as a sounding board to reflect and accentuate sounds that are not normally bothersome.

5-7.4.3 Alerting the Public. When a particularly noisy test or launch is imminent, the local news media and radio stations should be asked to alert the public. This procedure will assist in reducing the number of inquiry calls usually made after a test of this nature.

5-7.4.4 Measurements. Additionally, sound level measurements should be made within the test or launch area and selected locations in the community during a firing. These measurements will assist in determing the effect of acoustic noise on the test itself, and they will also assist in negotiating any legal disputes with the community.

5-8 UNPACKING AND HANDLING AT FIRING POSITIONS

5-8.1 UNPACKING. Receiving and unpacking precautions include:

a. Inspect handling equipment before operation to insure proper working order.

b. With the use of suitable hoisting equipment move motor container to unloading position. Life container at only those points designated as approved for lifting.

c. Personnel will remain clear of the container at all times while container is being raised, lowered, or moved.

d. The container will be opened and the motor given a visual inspection to assure no damage has occurred during transit.

e. In event of damage the motor will be rejected for flight or static test.

f. The motor will be moved to the firing site.

g. The explosive components will be bonded and grounded at the first opportunity.

5-8.2 LIFTING EQUIPMENT. Lifting equipment precautions are included because of the many accidents involved in their use. The following are recommended:

a. All lifting equipment such as slings, chains, spreader bars, etc., shall be checked after any alternation or redesign and at least every six months to 111 percent of rated maximum capacity.

b. Spreader bars shall be loaded during check in the same manner that they are used, and they are to be loaded to 111 percent of usable load.

c. Commercial straps and chains shall be loaded along their full length rather than folded or doubled.

d. Test personnel should not fabricate slings and general lifting equipment that are commercially available.

e. Commercial lifting straps and chains shall not be altered or repaired by test personnel.

f. All lifting equipment should be properly identified to indicate capacity and should carry the date that it was checked.

5-8.3 IGNITER HANDLING PRECAUTIONS. Precautions are necessary for handling motor igniters because the igniter is sensitive to shock, heat and static electricity.

5-8.3.1 Igniter Launch Site Precautions. The precautions at launch site include:

a. The igniter will normally be taken to the igniter barricaded test area where the test leads at the test stand will be shorted. The igniter will be carried to the other side of the barricade and installed in the igniter test mount. The shorting plug will be removed from the igniter and the test leads will be installed.

b. The operator stationed at the test stand will insure that the test lead wires remain shorted and grounded until the other operator returns and reports the igniter is installed.

c. The operator will then remove the short from the test leads and install the wires to the igniter tester and perform the continuity test.

d. When the continuity test is completed the test lead wires will be removed from the igniter tester and shorted and grounded.

e. The other operator will then return to test site and remove the test lead wires from the igniter and install the shorting plug to the igniter. Transport igniter to launch position and place in ready storage.

f. Shorting plugs will be removed only during electrical check outs and will be reinstalled immediately upon completion of such check outs.

g. Some igniters may consist of initiator and booster in which case the initiator alone is tested.

5-8.3.2 Igniter Static Firing Site Precautions. Igniter handling precautions may differ somewhat at static firing position in that:

a. Igniters are pretested prior to delivery to test site.

b. Continuity of firing circuit is tested prior to installation of igniters.

c. Final continuity check is made after igniter is installed in test item after area is cleared and just before actual firing.

5-9 STATIC TEST AND LAUNCH FIRING CIRCUITS

5-9.1 IMPORTANCE. Safety pertaining to firing circuits cannot be overemphasized because human lives are at stake if a test unit should fire prematurely.

5-9.2 DEFINITION. Firing circuits are defined as those independent electrical systems that are required to safely deliver electrical power to igniters, destruct packages, thrust termination devices, safety and arming devices, and other initiating devices.

5-9.3 SEGREGATION OF CIRCUITS. Cabinets, racks, etc., housing components of the firing circuits, should be segregated from other circuitry and should be easily recognized.

5-9.4 OPERATING PROCEDURES. Test area operating procedures should be explicit in describing the manner by which firing systems are operated, maintained, installed, and altered.

5-9.5 SHORTING. The ends of the firing line should be shorted to ground and insulated from all stray voltage sources prior to connecting the firing line to the unit and should remain so until immediately prior to the firing command.

5-9.6 RESISTANCE AND CONTINUITY MEASUREMENTS. Before the test unit is armed, the firing line resistance and continuity measurements should be recorded. Firing relays should be checked for frozen contacts. To eliminate the danger of the presence of stray voltage on the firing lines, a voltage measurement for deleterious voltages should be made.

5-9.7 INTERLOCKS. The firing lines should have two or more interlocks to break the circuit. One of these interlocks should be a key switch, the key being in the possession of the test foreman. Other interlocks may be gap switches, warning system interlocks, and others as required.

5-10 TEST AND LAUNCH PREPARATION AND SETUP.

5-10.1 CLEAR THE SITE. Before the test unit is taken to the test bay or launch site the area should be cleaned and all equipment removed that is not necessary for the given test. Only the people required to prepare for the test will be allowed in the area when the test unit is present.

5-10.2 ARMING. The test unit should be secured as soon as practical in a hold-down fixture at the thrust stand or launch stand.

5-10.2.1 Restraining Fixtures. All restraining fixtures should be in place before arming preparations start.

5-10.2.2 Personnel Evacuation. All personnel except arming crew must be evacuated from the test or launch area and the area will be cleared before arming procedures are started.

5-10.3 COUNTDOWN.

Countdown will be continued after all personnel are in approved safe areas.

a. The countdown should consist of voice communication over the public address system in the test area.

b. Regular and frequent announcements should be made to indicate the remaining time before the start of the test and the progress of the test.

c. The test unit will be fired on the command, "Fire."

5-10.4 ABNORMAL TEST CONDITIONS.

5-10.4.1 Misfire. A misfire is defined as any failure to ignite the test unit. In the event of misfire, a wait will be required as determined by the test conductor, usually 30 minutes. (Hang fires have lasted 25 minutes).

a. After the approved waiting period, and after removing the firing jumper and installing the shorting plug, and removing the firing key, an inspection team consisting of two or not more than three persons will proceed to the test bay.

b. Disconnect any internal power source.

c. Remove the igniter, inspect and determine disposition.

d. The area security will be maintained while the inspection team examines the motor.

e. Dispostion of the motor will be determined by the test site supervision.

f. The "all clear" will not be sounded until after one of the following conditions has been complied with:

   (1) The inspection team has isolated and corrected the discrepancy.

   (2) The motor has been placed in a "safe" condition to permit additional personnel to enter the bay and correct the trouble.

   (3) The misfire has been corrected by firing the motor with a new igniter.

   (4) The motor has been removed from the test bay.

5-10.4.2 A Hang Fire. A hang fire is defined as a firing with undue ignition delay. It requires no special treatment after firing occurs.

5-10.4.3 Malfunction. A malfunction may involve a case separation, nozzle failure, head-end failure, bond failure, case rupture, fall back, etc. In the event of a malfunction the following steps will be taken.

a. The area will be checked for fires by the test personnel, and appropriate action will be taken to alert the fire crew if required.

b. A wait will be required as determined by the test conductor. After the approved waiting period, and after removing the firing jumper, a two man inspection team will proceed to the test bay or launch site to investigate the hazardous conditions.

c. Hazardous debris will be cleared before the "all clear" is sounded.

d. The test area should be policed for unburned propellant.

5-10.5 PROCEDURES FOR POST TEST OPERATIONS.

5-10.5.1 Waiting Period. After a successful motor firing, a waiting period will be determined at the discretion of the test conductor after which one man will enter and check the test or launch area.

5-10.5.2 All Clear. If it is safe to enter, the "all clear" signal will be given.

5-10.6 PRECAUTIONS FOR PROBE LAUNCHES.

Some specific precautions for probe type launches are:

a. The launcher will be inspected and if any bolts are loose or damaged it is mandatory they be replaced before operation is initiated.

b. The assembled missile will then be transported to the launcher from the assembly building and mounted on the launcher.

c. Establish telephone communication between launch pad and firing point.

d. The key will be removed from firing panel prior to initiation of prefiring tests.

e. A prefiring continuity test will be made of the igniter squib circuit and the firing line.

f. A procedure should be established stating at what time and which personnel are to be evacuated to designated areas.

g. The igniter will be installed in the missile by the igniter crew only. No other personnel should be in the area.

h. The firing line will be checked for stray voltage.

i. Remove the igniter squib line shorting plug and connect firing line to the squib line. This step constitutes final arming.

j. Clear igniter crew from launching area.

k. Obtain final range and launch area safety clearance.

l. Arm firing panel by replacing the key.

m. At command "FIRE" close circuit.

5-10.7 IMPACT LIMIT LINES. Impact Limit Lines are located geographically as identification of areas to be protected from missile impact. Destruct or thrust termination criteria is applied to keep the possible impact of significant pieces within these lines.

a. Destruct limits should accomodate missile turning rates and dynamic lag in a missile destrution.

b. Wind effects should be analyzed.

c. Penetration capability of fragments should be considered in determining lines and shelters.

d. Instantaneous Impact Predictions should be used in real time control, to indicate points where the missile would impact if terminated.

e. In general, about 1 mile stand-off is used between impact limit lines and the area to be protected.

f. Effects of overpressure, fragmentation, and toxic cloud dispersion should be considered in establishing impact limit and evacuation criteria.

CHAPTER 6

FACILITIES

6-1 INTRODUCTION

Propellant manufacturing, rocket, and missile assembly plants are all highly controlled. The responsible industry or government agency must assure that they meet safety standards. These standards are set by an agency of the federal government or in a few cases the state in which the plant is located. The safety standards are readily available and are usually designated in the facility contract. Because of the availability of specific standards this chapter will be used to review some safety philosophy and a few of the major problem areas. This chapter should enable the user to quickly review facility plans to assure that the most significant items have been included.

6-2 SITING

6-2.1 LOCATION. Several factors have to be considered in siting propellant facilities. One of these is the availability of land; another is the layout of the facilities in such a way as to provide a safe and efficient flow of the various raw materials through the manufacturing processes, leading to the finished motor. The proper separation of these facilities from other plant areas and the plant boundary must be considered.

6-2.2 PLANT LAYOUT. Each process involved in the manufacturing of solid propellant must be analyzed to determine the degree of the hazards associated with the process. A basic rule in explosive operations is to limit the exposure to a minimum number of personnel, for a minimum time, to the minimum amount of the hazard material consistent with safe and efficient operations. The most hazardous operations should be located in separate buildings and should, when feasible, be conducted by remote control with operators provided adequate protection from any potential explosion. Several of the less hazardous operations may be performed in the same building, if required for the efficiency of the operation. However, even these less hazardous operations should be segregated from each other by concrete dividing walls or adequate operational shields to prevent a chain reaction involving the entire building in case of an incident in one of the operations. Finally, operations involving inert items, such as preparation of motor cases, which do not have to be performed in an explosive building, should be conducted in an "inert" location, located at a minimum of intraline distance from all explosive locations.

6-2.3 QUANTITY-DISTANCE REQUIREMENTS. Quantity-Distance is used to define the relationship between quantity of explosives versus distance separation. In determining the quantity-distance requirements, explosives and ammunition are divided into seven basic classes, according to the damage expected if they are involved in an accident. Each class is further subdivided for "inhabited building distance", public highway and railway distance, "intraline distance" and "magazine distance". These various distances furnish different degrees of protection with the "inhabited building distance" offering the greatest protection. The basic requirements for the quantity-distance tables are developed and published by the "Armed Services Explosives Safety Board" (ASESB). Each service implements the quantity-distance tables with its own publication: AMCR 385-224 (Army), TO 11A-1-40 (Air Force) and NAVWEPS - ORD Pamphlets (Navy).

6-2.4 MANUFACTURE AND LOADING. Propellant manufacturing and motor loading operations shall be performed in an area that is separated from all other areas and the boundary of the establishment by inhabited building distance. Intraline distances from the appropriate QD tables shall be used for determining the separation distances required for explosives buildings within a propellant operating line. For specific guidance in determining the explosives hazard classification of propellants during the stages of the manufacturing process, DoD Manual 4145.26M should be utilized unless there is a more restrictive requirement placed by the procurement agency or the state. In the location of the propellant manufacturing area and in the spacing of the individual buildings within the area, allowances should be made for the future increase of explosives limits at each explosives operation.

6-2.5 MAGAZINES. Service magazines, provided for storage of explosive material, needed in the assembly or manufacturing process, shall be located at a minimum of intraline distance, based on quantity of explosive in the service magazine, from the nearest operating building. Service magazines for inert items should be separated 100 ft. from the operating building.

6-3 GENERAL DESIGN CRITERIA

6-3.1 GUIDANCE. Safety guidance for building design and construction requirements is contained in DoD Manual 4145.26M and in the specific armed services safety manuals. To insure that the safety guidance is properly utilized in planning and designing new propellant facilities, a qualified safety engineer should be a member of the planning and design review groups. The safety engineer with his past experience, while not directly responsible for facility design will advise the design engineers of potential hazard areas and suggest corrective measures.

6-3.2 SAFETY DESIGN. Safety design of propellant facilities should include the following.

a. Incorporation of necessary special handling and equipment features to minimize the possibility of accidentally providing a means of ignition.

b. Incorporation of special safety features (such as substantial dividing walls, blow out panels and etc.) to minimize amount of damage and injuries in case of an accidental explosion or fire.

c. Adequate distances between various facilities, based on quantities of explosives in each facility.

6-3.3 CONSTRUCTION DESIGN. The following construction design features will tend to minimize injuries and property damage in case of an accidental explosion or fire.

a. Use of substantial dividing walls (twelve inch reinforced concrete) to prevent propagation of an explosion within a building where more than one explosives operation is being conducted.

b. Use of operational shields for protection against explosion or fire. For example, a twelve inch reinforced concrete wall provides protection against a maximum of 15 pounds of explosives involved in a detonation if the explosives is 3 feet from the wall and 3 feet above the floor. For greater quantities, when there is a possibility of a detonation, a protected remote control facility should be used.

c. Incorporation of diaphragms in duct work, used to convey explosive materials, to act as a vent in case of accidental ignition or explosion.

d. Use of shatter proof materials in windows in lieu of glass.

e. Use of earth barricades to protect against fragment damage when adequate distance is not available.

f. Restriction of explosives buildings to one story, unless operational requirements dictate use of more than one story.

g. Utilization of light and frangible material for at least one wall and the roof of explosives buildings. This will provide venting to relieve or limit the pressure resulting from a fire or explosion and cut down on the weight of missiles. Roofs should be independently suspended above each explosives bay.

h. Restriction of the equipment allowed within explosives bay to the bare minimum. All equipment, not required to be in a bay, should be located outside the building. In some cases heavy walls or barricades can be used to protect the equipment from the process hazard.

i. Proper engineering of fire protection and alarm systems.

j. Adequate exits to include safety chutes for all work levels above the ground floor. Emergency doors should be the quick opening type, either free swinging or equipped with panic hardware.

6-3.4 PROCESSING SAFETY. Safety in propellant manufacturing processes depends to a large degree on equipment design and personnel action. However, proper design of facilities and utilities can aid considerably in the control of hazards. Accidental ignition of the propellant or its ingredients is a most difficult hazard to guard against. This hazard should be minimized by control of propellant dust, flammable vapors, and ignition control.

6-3.4.1 Dust Control.

a. Proper facility design will help to minimize the accumulation of explosive dust particles. High gloss paint on walls and ceilings will encourage employees to keep the walls and ceilings dust free. Adequate space between equipment and walls will enable employees to keep the area free of both dust and other trash. Cracks and crevices in floor, walls, or ceiling should be repaired. Dead spaces between ceilings and roofs should be avoided.

b. Vacuum collector systems, installed at propellant cutting or other dust producing operations, will aid in the removal of the dust. However, precautions should be taken in the design of the vacuum collector system to assure that the system is not the cause of an explosion and that the system will not propagate an explosion or fire. Local exhaust ventilation should be provided for processes that may produce flammable vapors or gases.

6-3.4.2 Ignition Source Control. Since the elimination of all potential flammable or explosive material from propellant manufacturing processes is not possible, the ignition sources must be controlled.

a. Floors and other work surfaces in areas where exposed explosives or hazardous concentrations of flammable vapor or gases are located should be nonsparking to avoid sparks that may be generated by falling metal components. In some cases the propellant ingredients may be so sensitive to static sparks that they can be set off by the static electricity that may accumulate on a person. In this case, conductive floors should be required. However, conductive floors can cause undue electrical hazards around electrical equipment; therefore, if practicable electrical controls, switches, circuit breakers, and similar electrical devices should be located away from areas requiring conductive floors.

b. Static grounding conductors should be provided at each location where the process may generate static electricity. Common sources of static electricity generation include the following:

(1) Metallic powders or pulverized materials passing through chutes or pneumatic conveyors.

(2) Nonconductive power or conveyor belts in motion

c. Control of the relative humidity in the air by means of humidification can minimize the accumulation of static electricity. However, some metallic powders and pyrotechnic materials may react with the moisture and undergo spontaneous ignition. Humidification should not be utilized for such materials. NFPA Manual No. 77M-1961 contains further guidance on the control of static electricity.

d. Lightning protection systems should be provided at all locations where explosive materials are processed or stored.

e. The design of the electrical equipment used in propellant manufacturing processes must preclude accidental ignition of flammable liquids, vapors, and dusts that may be released to the atmosphere. Insofar as practicable, the electrical equipment, utilized in a hazardous process, should be located outside of the hazardous area. Electrical equipment, required to be located in the hazardous area, should meet the requirements of the National Electrical Code (Article 500) for the type hazard that may be encountered. In some cases, dual rated systems are required since there may be both gas (Class I) and dust (Class II) hazards involved in the process.

f. Where air conditioning is specified for a hazardous location, the air should not be recirculated if there are flammable or toxic gases and dust generated. Motors for exhaust fans in a hazardous location should be located outside the air stream with fan blades constructed of nonsparking material.

g. Steam heat is preferred for both heating and process heat requirements. For heating, radiators with exposed radiating surfaces in the form of S-shaped smooth pipe or fin-type radiators should be used to facilitate cleaning. The steam pressure normally should not exceed 5 psi (228°F) thus preventing pipe temperatures hot enough to provide an ignition source. Reducing stations should be located outside the buildings.

6-4 CONTROL BUILDING

For hazardous operations involving explosives materials, such as the mixing of composite propellants, personnel protection at least intraline distance from the hazardous operation is required. The control building should be of reinforced concrete construction (minimum of 12 inches recommended) and should be protected by a minimum of two feet of earth cover. Entrance to the control building should be in the opposite direction from the hazardous facility. If the control building services more than one hazardous facility, so that the entrance must face one of the hazardous operations, a metal door, sufficiently strong to withstand any expected blast, should be used to protect the entrance. Additional protection can be provided by a labyrinth leading to the door.

6-5 STORAGE OF EXPLOSIVES OR PROPELLANTS

The military services have classified all the items they commonly use and have designated the storage requirements. Most items will be covered in DoD Manual 4145.26M previously referenced. Detailed information will be found in the appropriate TM or regulation concerning the items in question.

6-6 AIR AND WATER POLLUTION

6-6.1 DRAINS. In propellant manufacturing operations there are always waste materials which can become troublesome. Many waste materials are either purposely or accidentally discharged to floor drains making it imperative that the drains be provided with cleanable sumps or holding ponds. Many ingredients which may be harmless, as long as they are covered with water, are just as treacherous as ever when removed from this environment. Designs of sumps must provide easy removal of waste and be free of obscure and hidden spaces that might confine so tightly that additional applied energy would initiate the material. This problem area is worthy of more attention so consult an explosives safety engineer about construction.

6-6.2 DISCHARGES TO THE ATMOSPHERE. Every process that discharges waste materials to the atmosphere should immediately become suspect and be investigated. Today's anti-pollution laws are being enforced.

6-7 ROADWAYS AND WARNING SYSTEM

6-7.1 ROAD SYSTEMS. The road system serving the propellant manufacturing area should be so designed that there are always "two ways out" from any location so that motor vehicles carrying explosives will not be isolated in case of an incident in the area.

6-7.2 WARNING LIGHTS. A system of warning lights or other signals should be utilized on all approaches to a hazardous facility with fences or other appropriate barricades to prevent unnecessary exposure of transients to the hazardous operations.

6-8 TECHNICAL ADVICE

Most propellant manufacturers have safety staffs, anti-pollution personnel, industrial hygienist, health physicist, and other specialists. Check the processes often and carefully and have the appropriate specialist assist. While plans for new facilities, or processes, are being made, these experts can save you dollars and future troubles. If the contract involves a governmental agency, the agency is usually glad to have its own team of specialists review plans so that there is assurance that the plans comply with specifications and regulations.

CHAPTER 7

WASTE DISPOSAL

7-1 GENERAL

7-1.1 WASTE DISPOSAL PERSONNEL. Disposal of waste should be regarded as an integral part of solid propellant rocket operations. A common mistake is to treat waste disposal as a nuisance job, to be assigned to the lowest labor grade in the plant, and to give it supervisory attention only when waste materials accumulate to a point where there is interference with normal work production. Such an attitude can result in serious hazard situations. While it is not essential that the entire waste disposal crew be manned by professionals, it is important that the crew receives direct professional supervision. The crew members should have sufficient on the job experience or training to recognize the different types of hazardous waste material, and to respect their potential for explosive or fire destruction. The crew should be specialized to the point that their operations do not indiscriminately mix propellant wastes with inert trash or garbage. The foreman of the crew should be thoroughly versed in the proper techniques for using primary explosives, igniters and blasting galvanometers, as recommended for commercial blasting (reference 1).

7-1.2 WASTE GENERATING PERSONNEL. In addition to adequate training of the waste disposal crew, a complete safety program for proper handling of hazardous waste materials must include instruction to the personnel at the waste-generating facility in the matter of properly preparing the material for pickup by the disposal crew. If the hazardous material at the waste-generating station is inadequately segregated, packaged, or labeled, it constitutes a booby trap for the pickup crew in spite of the best techniques that the latter can employ.

7-2 GUIDELINES

Safe disposal of solid propellant waste according to the above general principle involves the following.

a. Keep waste separated into like composition and like hazard characteristics, to the maximum extent possible, with attention to compatibility. This separation should be maintained from point of origin to point of final disposal, including all transportation steps.

b. Research and development propellant waste should always be kept separated from production waste. R&D propellant waste disposal should always be performed by trained technical personnel, not unskilled labor.

c. The safest means of disposing of propellant wastes is by burning, in an open area, in combustible containers. Poorly ignitable or poorly combustible materials should be interdispersed with scrap propellant, to aid in complete combustion; this is the one recommended exception to the separation rule given above.

d. Propellant wastes should be regarded as having sensitivity characteristics as least as hazardous as those of the propellants from which obtained. Waste collection and holding areas should conform to the propellant quantity-distance standards.

e. Experience has shown that propellant wastes which have been contaminated with foreign material - for example, floor sweepings, sand or dust from ground spillage, or residual materials from uncleaned containers are likely to be more sensitive than the propellants from which the waste is derived. From this, it follows that:

(1) transfer of waste from one container to another should be avoided as much as possible; it is preferable to destroy the material in the container in which it was collected,

(2) every effort should be made to avoid spillage or leakage which will result in contamination of the material with ground or floor dirt,

(3) when such spillage or leakage does occur, it should be quantitatively retrieved, and the mixture of waste and foreign material is to be handled as a primary explosive, not as propellant.

7-3 DISPOSAL OF SPECIFIC TYPES OF PROPELLANT WASTE

Several categories of solid propellant waste materials, and the recommended techniques for disposing of each, are summarized in Table 7-1. Detailed directions for handling the various categories are given in the paragraphs following.

7-3.1 UNCONTAMINATED OXIDIZER. Uncontaminated oxidizer for disposal consists of reject material (ammonium perchlorate, potassium perchlorate, or ammonium nitrate), process fines, or overrun from grinding or blending facilities or mix stations. It is collected and transported to the burn site in open-top 30-gallon drums, with lid attached. The oxidizer is spread thinly over a layer of excelsior or other flammable dry materials. The flammable material is ignited by a train of the material laid in such a manner that it will burn against the wind.

7-3.2 CONTAMINATED OXIDIZER OR IGNITER MATERIAL. Containers of contaminated oxidizer, igniter materials, or pyrotechnic materials from the igniter line may be destroyed individually or as a mixed group, but are separated from the other categories of waste material. Contaminated oxidizer is that which contains floor sweepings, partial addition of fuel, aluminum powder, or any other foreign material. Igniter and pyrotechnic materials are phlegmatized at the generating source with a non-reactive liquid suspending agent. The containers for all of these materials are fibreboard cartons, lined with conductive polyethylene bags. The bags are taped shut to preserve the phlegmatizing liquid and/or to exclude moisture. Destruction of these materials is performed in the containers in which

TABLE 7-1

MATERIAL	NOTE	CONTAINER	METHOD OF DISPOSAL
Waste oxidizer, out of specification but uncontaminated; ammonium perchlorate, ammonium nitrate, potassium perchlorate.	(a)	Opentop drums.	Drums are dumped into shallow, polyethylene-lined trench at burning grounds. Oxidizer is burned with excelsior or waste propellant as combustion aids.
Waste oxidizer, contaminated; igniter and pyrotechnic materials from igniter line (desensitized).	(a)	Conductive polyethylene bags in fibreboard containers.	Burned in containers with waste propellant as combustion aid.
Mixer cleanings, suspended in cleaning diluent (trichloroethylene).	(a)	20-gal fibreboard drum, with leakproof (Capran) liner.	Burned in containers with waste propellant as combustion aid.
Explosive liquid nitro compounds and nitroplasticizers.	(a)	Phlegmatized with diluting solvent; in drums or carboys.	Container top removed; burned in container with propellant as combustion aid.
Propellant, free-standing grains.	(a)	Wrapped in Kraft paper for transporting.	Ignited with other waste propellant at burning grounds.
Propellant, uncured.	(b)	Fibreboard containers, polyethylene lined.	Ignited in container with other waste propellant at burning grounds.
Propellant, machinings and cuttings (Class 2)	(c)	Fibreboard containers, polyethylene lined; water damp.	Ignited in container with other waste propellant at burning grounds.
Propellant, machinings and cuttings (Class 7).	(a)	Fiber drums or cardboard cartons.	Remove from container, spread in thin layers on ground and burn.
Propellant-contaminated paper, tissues and cleaning rags.	(c)	Polyethylene bags.	Ignited in container with other waste propellant at burning grounds.
Waste fuel and liner preparations.	(a)	Drums, cartons.	Container top removed; burned in container with propellant as combustion aid.
Casting (or base grain) powder.	(a)	Poly bags in metal cans, or fiber containers.	Metal cans removed; burn.
Defective motors, case-bonded, or motor from which grain cannot be removed.	(a)	Motor case.	Fire on static test stand, or split lengthwise with lineal charge and allow to burn.
Contaminated nitroglycerin.	(a)	Fiber container, with NG absorbed in sawdust.	Burn with powder train.
RDX and HMX (wet or out of specification).	(a)	Normal shipping container.	Explode dry material with plastic explosive; dry wet material in sun, and burn with scrap lumber.

NOTES:
(a) Disposed of individually and separate from all other materials.
(b) Disposed individually or as aid to insure burning of (a).
(c) Disposed with propellant only.

collected. The cartons are grouped at the burn site, interdispersed with cartons of waste propellant. A black powder squib is placed in the propellant, and the mass is remotely ignited.

7-3.3 TRICHLOROETHYLENE. Waste trichloroethylene, contaminated with propellant residues either in solution or suspension, is generated at mix stations, degreasers, mold cleaning stations, or any facility where propellant is cleaned from metal parts. Accident history has shown that spillage and evaporation of these residues can result in an extremely sensitive material, more so than the parent propellant. Accordingly, all transfer and handling steps should be minimized. Destruction should be accomplished in the collection container, preferably a non-metallic one. A 20-gallon fiberboard drum with a heavy-gauge leakproof liner has been found suitable. Prolonged holding of these wastes is to be avoided, as the liner may be embrittled and develop leaks. The drums are handled and transported by means of a forklift and wooden pallet, four drums to a pallet. At the destruction site the drums are burned, pallet and all, by means of added waste propellant. Ignition of the propellant is accomplished by means of a black powder squib. Repeat burns are performed with addition of more waste propellant, when necessary, after a 24-hour cooloff period.

7-3.4 NITRO COMPOUNDS. Explosive liquid nitro compounds and nitroplasticizers are sometimes left from propellant processes. These miscellaneous hazardous liquids are destroyed in the containers as collected, drums, cans, or carboys. Prior to transporting to the burn site, the liquids are desensitized by addition of a phlegmatizing solvent, where possible. At the burn site, container tops or drum bungs are removed, waste propellant is interdispersed with the containers, and the propelant is ignited by a black powder squib.

7-3.5 PROPELLANT. Waste propellant is burned separately or is grouped with hazardous waste materials as a combustion aid to ensure complete consumption of the latter. It is important to properly package the various forms of waste propellant for transporting and handling. Free-standing grains are wrapped in Kraft paper. Cuttings and sawdust from machining are placed in containers lined with polyethylene and filled with enough water to keep the scrap thoroughly wetted. Uncured propellant is also collected in the cartons with polyethylene liner, but without any water addition. Propellant-contaminated paper and cleaning rags are collected in polyethylene bags, and are burned with other waste propellant.

7-3.6 CASE BONDED MOTORS. Case bonded motors are always treated as special disposal problems, for which the disposal method depends upon the degree of confinement by the case. If sufficient and safe removal of the nozzle and end closures can be accomplished to make the motor non-propulsive, the grain may be burned with other scrap propellant. An alternative technique is to split the case lengthwise with a lineal charge and allow the propellant to burn.

7-3.7 FUEL. Waste fuel and liner preparations are collected and burned in the same manner as propellant liquid wastes. Collection is made in non-leaking drums or cartons. Container tops or bungs are removed at the burn site. Propellant is dispersed throughout the containers, and ignition is accomplished with a black powder squib.

7-4 DEEP SEA DISPOSAL

Preparation for deep sea disposal shall be coordinated with the appropriate Military Traffic Management Terminal Services office through procurement channels utilizing the Offering, Acceptance, and Release procedure outlined in Military Traffic Management Regulation DSAR 4500.3, AR 55-355, NAVSUP PUB 444, AFM 75-2, MCO P 4600.14, Chapter 202, section V. Such requests shall include the type of material by nomenclature and hazard class, packaging data, and available shipping dates.

7-5 BURN SITES

7-5.1 SITE SELECTION. The selection of a burn site is important to safe waste disposal practice. Choice of the area and procedures governing it will be subject to local and plant regulations regarding quantities stored, fire protection needed, and air and ground water pollution limits; but in any case, it will be necessary to isolate the area in conformance with quantity-distance standards.

7-5.2 PROCEDURES. Operations to be conducted in the burning grounds should be completely detailed in standard operating procedures. The procedures should be consistent with the general principles given above, with plant safety policy, and with accepted good safety practice in commercial blasting. The following details should be covered in the procedures.

a. Personnel access to the area, personnel limits, clothing for operating personnel, and safety shelters.

b. Posting the area including non-smoking areas.

c. Restrictions on radio transmitting in the vicinity of electrically actuated explosive devices and restrictions on operations during electrical storms.

d. Regulations on allowable wind, humidity, and weather conditions for conducting burning.

e. Restrictions on flammable grass or vegetation in the area.

f. Maximum quantity of material in one burn site, number of burn sites to be ignited simultaneously and spacing of burn sites.

g. Restrictions on vehicles in the burn area.

h. Limits on amount of material to be held in the area for future destruction, maximum time it may be held and distance requirements on material in holdover state.

7-6 TRANSPORTATION OF WASTE FOR DISPOSAL

In transporting hazardous waste from the point of generation to the burn site, all safety practices normally employed for transportation of propellant or explosives should be used. These should be in conformance with local and plant regulations with respect to speed limits, placards on the vehicle, routes and allowable passengers.

The vehicle should be inspected daily for cleanliness, and any waste residues should be removed before the vehicle is returned to service. Floors and side walls of vehicle bed should be covered with nonsparking materials (such as wood). They must be kept free of cracks or crevices that would provide catch and pinch places for waste materials.

REFERENCE

1. "Blaster's Handbook," duPont deNemours, Latest Edition.

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13. ABSTRACT

(U) This publication is intended as a source of information and as a set of basic guidelines for the processing, handling, storage, and transportation of chemical solid rocket propellants and propellant ingredients. It contains chapters on Hazard Analysis; Propellant Manufacture; Packaging, Storage Surveillance, and Transportation; Assembly, Launch and Static Test; Facilities; and Waste Disposal.

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